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ABSTRACTS

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Table of Contents

PLENARY SESSION 01 – PERSPECTIVES ON FUSION ENERGY

01.1: Vision for a Sustainable Energy Future <i>David E. Baldwin</i>	2
01.2: Material science and technology in the roadmap towards a fusion reactor <i>Minh Quang TRAN</i>	2

PARALLEL ORAL SESSION 02A – FERRITIC STEELS – I

02A.1: Status of Reduced Activation Ferritic/Martensitic Steel Development <i>N. Baluc, D.S. Gelles, S. Jitsukawa, A. Kimura, R.L. Klueh, G.R. Odette, B. van der Schaaf</i>	6
02A.2: Irradiation Effects on Precipitation and its Impact on the Mechanical Properties of Reduced-Activation Ferritic/Martensitic Steels <i>Hiroyasu Tanigawa, Hideo Sakasegawa, Naoyuki Hashimoto, Ronald L. Klueh, Masami Ando, Mikhail A. Sokolov</i>	6
02A.3: New Nano-Particle-Strengthened Ferritic/Martensitic Steels by Conventional Thermomechanical Processing <i>R.L. Klueh, N. Hashimoto</i>	7
02A.4: Mechanical Properties of 9Cr Martensitic Steels and ODS-FeCr Alloys After Neutron Irradiation at 325°C up to About 40 dpa <i>A. Alamo, J.L. Bertin, V. Shamardin, P. Wident</i>	8
02A.5: Friction Stir Welding of Oxide Dispersion Strengthened Steels <i>G.J. Grant, R. Lindau, S. Packer, R. Steel, D.S. Gelles</i>	9

PARALLEL ORAL SESSION 02B – HIGH HEAT FLUX PLASMA FACING MATERIALS - I

02B.1: High Heat Flux Testing of Plasma Facing Materials and Components - Status and Perspectives for ITER Related Activities <i>J. Linke, I.V. Mazul, R. Nygren, J. Schlosser, S. Suzuki</i>	12
02B.2: Overview of Codeposition and Fuel Inventory in Castellated Divertor Structures at JET <i>M.J. Rubel, J.P. Coad, Contributors to the JET-EFDA workprogramme</i>	13
02B.3: Development of Brazing Foils to Join Monocrystalline Tungsten Alloys with Eurofer Steel <i>B.A. Kalin, V.T. Fedotov, O.N. Sevrjukov, A.N. Kalashnikov, A. Moeslang, M. Rohde</i>	14
02B.4: Thermal Stability of Tungsten - Clad Low Activation Steel for Fusion Energy <i>Glenn Romanoski, Lance Snead, Muralidharan Govindarajan</i>	15
02B.5: Effects of Solid Transmutation Elements on Defect Structure Development of W using W-Re and W Re Os Model Alloys <i>K. Abe, J.C. He, A. Hasegawa, M. Fujiwara, M. Satou, T. Shisido</i>	16

PARALLEL ORAL SESSION 03A – FUNDAMENTAL RADIATION EFFECTS

03A.1: Ab initio Study of Helium in α-Fe: Dissolution, Migration and Clustering <i>François Willaime, Chu-Chun Fu</i>	20
03A.2: Direct Observation of Dislocation-Stacking Fault Tetrahedra Interaction <i>Yoshitaka Matsukawa, Yuri N. Osetsky, Roger E. Stoller, Steven J. Zinkle</i>	20

03A.3: A Magnetic Interatomic Potential for Molecular-Dynamics Simulations of Body-Centered Cubic Iron <i>P.M. Derlet, S.L. Dudarev</i>	21
03A.4: Molecular Dynamics Simulation of Screw and Mixed Dislocation Interaction with Stacking Fault Tetrahedron in FCC Cu <i>Hyon-Jee Lee, Yoshitaka Matsukawa, Ian M. Robertson, Brian D. Wirth</i>	21
03A.5: Feature of Damage Accumulation in Be12Ti Intermetallic Compound <i>Naoaki Yoshida, Hiroto Iwakiri, Yoshiyuki Watanabe</i>	22
PARALLEL ORAL SESSION 03B – BLANKET ENGINEERING - I	
03B.1: Material System Integration and Irradiation Test for Fusion Blanket <i>Akira Hasegawa, T. Nuroga, A. Kimura, S.J. Zinkle</i>	26
03B.2: On Qualification, Codes and Standards, Quality Assurance and Licensing <i>David Petti</i>	26
03B.3: The Feasibility of Recycling and Clearance of Active Materials from a Fusion Power Plant <i>M. Zucchetti, L.A. El-Guebaly, R.A. Forrest, T.D. Marshall, N.P. Taylor, K. Tobita</i>	27
03B.4: Estimation of Tritium Release Behavior from Solid Breeder Materials Under the Condition of ITER Test Blanket Module <i>T. Kinjyo, M. Nishikawa, M. Enoeda</i>	28
03B.5: Material Compatibility Issues in Fusion Fuel Cycle R&D and Design <i>D.K. Murdoch, I. Cristescu, C. Day, M. Glugla, R. Lässer, A. Mack</i>	29
POSTER 4 SESSION	
04-1: Fracture Toughness Vis-a-Vis the Master Curve for Some Advanced Reactor Pressure Vessel and Structural Steels <i>Randy K. Nanstad, Mikhail A. Sokolo Metals, Ceramics Division Oak Ridge National Laboratory</i>	32
04-2: In Situ Fatigue of the Eurofer 97 steel <i>Pierre Marmy</i>	32
04-3: Interaction of $1/3\langle 1120 \rangle\{0001\}$ and $1/3\langle 1120 \rangle\{1100\}$ Edge Dislocations with Self-interstitial-atom Loops in alpha-Zirconium <i>Zhouwen Rong, David J. Bacon, Roman E. Voskoboynikov, Yuri N. Osetsky</i>	33
04-4: Analysis of Recovery Process of Neutron- Irradiation-Induced Defects in SiC by Isothermal Annealing up to 1400°C <i>Saishun Yamazaki, Kousuke Yamaya, Masamitsu Imai, Toyohiko Yano</i>	34
04-5: Neural Network Analysis of Charpy Toughness of Neutron Irradiated Low-activation Martensitic steels <i>G.A.Cottrell, R.Kemp, H.K.D.H. Bhadesia, G.R.Odette, T.Yamamoto, H.Kishimoto</i>	35
04-6: Atomistic Insight into Thermal Conductivity Degradation and Swelling of Irradiated SiC <i>Tjacka Bus, Brian D. Wirth, Y. Katoh, L.L. Snead</i>	35
04-7: Proton Irradiation Modifications in Ultraviolet Transmission on KU1 Quartz Glass <i>B. Constantinescu</i>	36
04-8: Plastic Deformation of SUS304 under In-situ and Post-Irradiation Fatigue Loadings <i>Y. Murase, Johsei Nagakawa, N. Yamamoto</i>	36

04-9: Embrittlement Behavior of Neutron Irradiated RAFM Steels <i>E. Gaganidze, H.-C. Schneider, B. Dafferner, J. Aktaa</i>	37
04-10: Strain Hardening and Long Range Internal Stress in the Localized Deformation of Irradiated Polycrystalline Metals <i>Thak Sang Byun, Naoyuki Hashimoto</i>	37
04-11: Inner structure of dislocation channels in neutron-irradiated V-Cr-Ti alloys <i>Ken-ichi Fukumoto, Masanari Sugiyama, Hideki Matsui</i>	38
04-13: Small Punch Creep Properties of Reduced Activation Ferritic Steel <i>T. Nakata, S. Komazaki, M. Fujiwara, Y. Kohno, K. Shiba, A. Kohyama, T. Hashida</i>	38
04-13: Helium Effect of Microstructural Evolution in Ion irradiated Reduced Activation Ferritic/ Martensitic Steel to High Fluences <i>H. Ogiwara, A. Kohyama, H. Tanigawa, H. Sakasegawa</i>	39
04-14: Stress Corrosion Cracking Susceptibility of Ferritic/ Martensitic Steel in Super Critical Pressurized Water <i>T. Hirose, K. Shiba, M. Enoda, M. Akiba</i>	39
04-15: Optimization of the EUROFER Uniaxial Diffusion Weld Process <i>Axel von der Weth, Helmut Kempe, B. Dafferner, J. Aktaa</i>	40
04-16: Activation and Radiation Damage Behaviour of Russian Structural Materials for Fusion Reactors in the Fission and Fusion Reactors <i>A.I.Blokhin, V.M.Chernov, N.A.Demin, M.V.Leont'eva-Smirnova, M.M.Potapenko</i>	41
04-17: A Closer Look at the Fracture Toughness of Ferritic/Martensitic Steels <i>Enrico Lucon, Abderrahim Al Mazouzi, Marc Scibetta</i>	42
04-18: Mechanical Properties and Microstructure of Three Russian Ferritic/Martensitic Steels Irradiated in BN-350 to 50 dpa at 490°C <i>A.M. Dvoriashin, S.I. Porollo, Yu.V. Konobeev, N.I. Budylnkin, E.G. Mironova, F.A. Garner</i>	42
04-19: Research and Development on the Chinese Low Activation Martensitic steel (CLAM) <i>Jinnan YU, Qunying HUANG, Farong WAN, Gang YU</i>	43
04-20: Microstructural Inhomogeneity of Reduced-Activation Ferritic/Martensitic Steels <i>Hiroyasu Tanigawa, Mikhail A. Sokolov, Ronald L. Klueh</i>	43
04-21: Mechanical Properties of Irradiated 9Cr-2WVTa Steel With and Without Nickel <i>R.L. Klueh, M.A. Sokolov</i>	44
04-22: Effects of Heat Treatment and Irradiation on Mechanical Properties in F82H Steel doped with Boron and Nitrogen <i>N. Okubo, E. Wakai, S. Matsukawa, T. Sawai, S. Jitsukawa</i>	45
04-23: Fracture toughness properties in the transition of the EUROFER97 tempered martensitic steel <i>R. Bonadé, P. Spätig, N. Baluc</i>	45
04-24: Effect of Heat Treatments on Tensile Properties and Microstructures of F82H Steel Irradiated by Neutrons <i>E. Wakai, M. Ando, T. Sawai, H. Tanigawa, N. Okubo, N. Hashimoto, R.E. Stroller, T. Tomita, A. Ishikawa, T. Yamamoto, Y. Kato, F. Takada, S. Jitsukawa</i>	46
04-25: Comparison of radiation damage of the CLAM steel under irradiation in IFMIF and a fusion power reactor <i>Y. Chen, Q. Huang, Y. Wu, U. Fischer</i>	46
04-26: Ultra-high strength in nanocrystalline materials under shock loading <i>E.M. Bringa, A. Caro, Y.M. Wang, M. Victoria, A. Hodge, J. McNaney, B.A. Remington, R. Smith, B. Torralva, H. Van Swygenhoven</i>	47

04-28: Implantation of D+ and He+ in Candidate Fusion First Wall Materials <i>R.F. Radel, G.L. Kulcinski</i>	47
04-29: Infrared Thermal Fatigue Test for IFE First Wall Materials <i>Glenn Romanoski, Lance Snead, Joseph Kelly, Adrian Sabau</i>	48
04-30: Diffuse X-ray Scattering Measurements of Point Defects and Clusters in Iron <i>R.E. Stoller, F.J. Walker, P. Zschack, G.E. Ice</i>	48
04-31: The 1-D Reciprocating Motion of Vacancy-type Dislocation Loops in FCC Metals <i>Yoshitaka Matsukawa, Yuri N. Osetsky, Anna Serra, Roger E. Stoller, Steven J. Zinkle</i>	49
04-32: Void swelling behavior in electron irradiated Fe-Cr-Ni model alloys under temperature variation <i>Y. Satoh, S. Abe, H. Matsui, I. Yamagata</i>	50
04-33: Microstructures in F82H steel irradiated under alternating temperature <i>N. Okubo, E. Wakai, T. Tomita, S. Jitsukawa</i>	50
04-34: Microstructure of Helium-implanted and Proton- irradiated T91 Ferritic/Martensitic Steel <i>Z. Jiao, N. Ham, G.S. Was</i>	51
04-35: Hydrogen Retention Properties of Co-Deposition Layers Under High-Density Plasmas in TRIAM-1M <i>M. Tokitani, M. Miyamoto, K. Tokunaga, T. Fujiwara, N. Yoshida, M. Sakamoto, H. Zushi, K. Hanada, TRIAM group, S. Nagata, B. Tsuchiya</i>	51
04-36: The effect of Helium production on void growth in metals, during irradiation with 14MeV fusion neutrons <i>A.J. Webster</i>	52
04-37: Anisotropy Migration of Self-point Defects in Dislocation Stress Fields in BCC Fe and FCC Cu <i>A.B. Sivak, N.A. Chernavskaya, V.M. Chernov, V.A. Romanov</i>	52
04-38: Temperature Dependence of One Dimensional Motion of Interstitial Clusters in Fe and Ni <i>T. Yoshiie, M. Horiki, Q. Xu, K. Sato</i>	53
04-40: Thermally Activated Transport of a Dislocation Loop within an Elastic Model <i>Kazuhito Ohsawa, Eiichi Kuramoto</i>	53
04-41: Defect Evolution in Silicon Carbide Irradiated with Ne and Xe Ions with energy of 2.3 MeV/u <i>Chonghong Zhang, Youmei Sun, T. Shibayama, Yunfan Jin, Yin Song, Jinglai Duan</i>	54
04-42: Effect of Impurities on the Reaction Kinetics of SIA Clusters and Damage Accumulation in Metals under Cascade Irradiation <i>H. Trinkaus, B.N. Singh, S.I. Golubov</i>	54
04-43: Computer Simulation of Cascade Damage in Alpha-Iron <i>Andrew F. Calder, David J. Bacon, Alexander V. Barashev, Yuri N. Osetsky</i>	55
04-44: The Effects of Cascade Damages on the Dynamical Behavior of Helium Bubbles in Cu <i>M. Miyamoto, K. Ono, K. Arakawa, R.C. Birtcher</i>	55
04-45: Energetic and Crystallographic Characteristics of Self-point Defects and Their Clusters of Different Geometry in Iron <i>V.M. Chernov, V.A. Romanov, A.B. Sivak</i>	56

04-46: Kinetic Monte Carlo Studies of the Reaction Kinetics of Crystal Defects that Diffuse One-dimensionally with Occasional Transverse Migration <i>H.L. Heinisch, B.N. Singh, H. Trinkaus</i>	56
04-47: Irradiation Temperature Dependence of Self-Organized Nanostructure Generated on Au Surface Under Electron Irradiation <i>K.Niwase, F.Phillipp, A.Seeger</i>	57
04-48: Radiation damage in Fe-Cr alloys: computer modelling and modelling-oriented experiments <i>A. Almazouzi, L. Malerba</i>	57
04-49: Monte Carlo simulations of radiation damage in hcp metals <i>C. Arévalo, M.J. Caturla, J.M. Perlado</i>	58
04-51: Accumulation damage in Fe using kinetic MonteCarlo implemented with the last ab-initio parameters <i>E. Martínez, J.Manuel Perlado, M.Victoria, M.J.Caturla, P.Cepas, J.Marian</i>	58
04-52: Displacement cascades due to energetic recoils in amorphous silica using molecular dynamics simulations <i>F. Mota, M.-J. Caturla, J.M. Perlado, A. Ibarra, M. León, A. Kubota</i>	59
04-53: Molecular Dynamics Simulation of the Hardening of Fe by Irradiation Induced Defects <i>R. Schäublin, Y.L. Chiu, M.J. Caturla</i>	59
04-54: A Dislocation Dynamics Model for the Effects of Irradiation in the Brittle - Ductile Transition of F82H <i>S.J. Noronha, N.M. Ghoniem</i>	60
04-55: Mechanisms of mobility of single-interstitial and interstitial clusters in Fe-Cr alloys: a computer simulation study <i>D. Terentyev, L. Malerba, M. Hou</i>	60
04-56: Atomistic View of Dislocation – Obstacle Interactions in Ni via MD Simulations <i>Peeravuth Boonsuwan, Hyon-Jee Lee, Brian D. Wirth, Ian M. Robertson</i>	61
04-57: Simulations of Elastic Electron Diffuse Scattering from Small Dislocation Loops <i>Zhongfu Zhou, S L Dudarev, M L Jenkins, A P Sutton, M A Kirk</i>	61
04-58: Lattice kinetic Monte-Carlo modelling of helium cluster formation in ferritic steel <i>V. Borodin, P. Vladimirov, A. Möslang</i>	62
04-59: Point Defct Interactions in Fe-Cr Alloys <i>K.L. Wong, J.H. Shim, B.D. Wirth</i>	62
04-60: PKA Energy Spectra and Primary Damage identification in Amorphous Silica under different neutron energy spectra <i>M.L. Gámez , M. Velarde, F. Mota, J. Manuel Perlado, M. León, A. Ibarra</i>	63
04-62: Cracking at elevated temperature in CuCrZr alloys during Electron Beam Welding <i>A.Durocher, D.Ayrault, Ch.Chagnot, M.Lipa, W.Saikaly,</i>	63
04-63: The New Electron Beam Test Facility JUDITH-2 for Investigations on Plasma Facing Components – Design and First Operational Experience <i>M. Rödíg, W. Kühnlein, J. Linke, P. Majerus, T. Hirai, M. Neumann</i>	64
04-64: Application of lock-in technique to CFC armoured plasma facing components inspection <i>F. Escourbiaca, S.Constans, X. Courtois, A. Durochera, V.Casalegnob</i>	64

04-65: Operational Conditions in a W-Clad Tokamak <i>R. Neu, R.Dux, O. Gruber, H. Greuner, A. Kallenbach, K. Krieger, H. Maier, T. Pütterich,</i>	65
04-66: Accumulation of Helium in Tungsten Irradiated by Helium and Neutron <i>Q. Xu, N. Yoshida and T. Yoshiie</i>	65
04-67: Behavior of Deuterium in Boron Films Covered by Oxygen-containing Layer <i>M.X. Wang, H. Miyauchi, T. Nakahata, Y. Nishikawa, Y. Oya, N. Noda, K. Okuno</i>	66
04-68: Silicon Doped Carbon/Cu Joints Based on Amorphous Alloy Brazing for First Wall Application <i>Zhang-jian Zhou, Zhi-hong Zhong, Chang-chun Ge</i>	66
04-69: Properties of co-deposits on graphite high heat flux components <i>E. Fortuna, M.J. Rubel, M. Pisarek, W. Zielinski, M. Miskiewicz, V. Philipps, K.J. Kurzydowski</i>	67
04-70: Comparison of Erosion Processes of RAF and Pure Fe by Hydrogen and Carbon Mixed Ion Beam Irradiation <i>Yoshio Ueda, Masakatsu Fukumoto, Daisuke Sakizono, Isao Sawamura, Masahiro Nishikawa</i>	67
04-72: In-reactor Creep-Fatigue Tests of a CuCrZr Alloy at About 333K <i>P. Moilanen, S. Tähtinen, B.N. Singh, P. Jacquet, J. Dekeyser</i>	68
04-73: Production and Characterization of Titanium Beryllides for HIDOBE Irradiation <i>P. Kurinskiy, M. Klimiankou, A. Moeslang, A.A. Goraieb</i>	68
04-74: Progress of Research on Plasma Facing Materials in USTB <i>Chang-Chun GE, Zhang-Jian Zhou, Wei-Ping Shen, Zhi-Hong Zhong</i>	69
04-75: Effects of microstructure on exfoliation and blistering behavior of various W by He implantation at about 550C <i>T.Ogawa, A.Hasegawa, K.Tanaka, K.Abe</i>	70
04-76: Distribution of hydrogen isotope retained in the divertor tiles used in JT-60U <i>Y. Hirohata, T. Tanabe, T. Shibahara, M. Oyaidzu, K. Sugiyama, Y. Oya, A.Yoshikawa, Y.Onishi, T. Arai, K. Masaki, Y. Ishimoto, K. Okuno, N. Miya</i>	71
04-77: Analysis on Damage to TF Coils of A Compact Reversed Shear Tokamak CREST <i>Q. Huang, S. Zheng, L. Lu, R. Hiwatari, Y. Asaoka, K. Okano, Y. Ogawa</i>	71
04-78: Experimental and numerical analyses on LiSO4 Pebble beds used in a ITER Test Module Blanket <i>Donato AQUARO, Nicola ZACCARI</i>	72
04-79: Characteristics of surface water on Li4SiO4 <i>Kazuo Nakashima, Masabumi Nishikawa, Takayuki Terai, Tomohiro Kinjyo, Tomotaka Ishizaka</i>	72
04-80: Pre-irradiation characterization of beryllium for High Fluence Irradiation <i>J.B.J. Hegeman, M.S. Stijkel, P. ten Pierick, J.G. van der Laan</i>	73
04-81: Use of Refractory Metal Alloys in Permeator and Heat Exchanger Applications in Dual-Coolant PbLi Breeding Blankets <i>R.J. Kurtz</i>	73
04-82: Experimental Determination of Creep Properties of Beryllium Irradiated to Relevant Fusion Power Reactor Doses <i>M. Scibetta, E. Rabaglino, A. Pellettieri, L. Sannen,</i>	74
04-83: Investigation of Phase Transition in Li2TiO3 by High Temperature X-ray Diffraction <i>Junichi Makita, Takuya Hashimoto, Tsuyoshi Hoshino</i>	74

04-84: Experiments on Eurofer steel corrosion by Pb-17Li <i>A. Gessi, A. Aiello, G. Benamati</i>	75
04-86: Beryllium Interactions in Molten Flibe <i>G.R. Smolik, M.F. Simpson, P.J. Pinhero, et al.</i>	75
04-87: Study on reaction of titanium Beryllide with water vapor <i>K. Munakata, H. Kawamura, M. Uchida</i>	76
04-88: Effect Of Neutron Irradiation On Radiation Hardening And Electrical Resistivity Of A Number Of Refractory Metals And Alloys <i>A.S. Pokrovsky1 S.A. Fabritsiev</i>	77
04-89: Molybdenum-Rhenium Alloys for High Heat Flux and High Neutron Flux Applications <i>J.T. Busby, L.L. Snead, F.W. Wiffen, S.J. Zinkle</i>	78
04-90: Evaluation of the mechanical properties of W and W 1%La₂O₃ in view of divertor applications <i>Michael Rieth</i>	78
04-91: Effect of In-Cascade Defect Cluster Formation on Radiation Hardening of Molybdenum <i>Meimei Li, T.S. Byun, N. Hashimoto, B. Cockeram, S.J. Zinkle</i>	79
04-92: Anodic polarization properties of V-Cr-Ti type alloys for fusion applications <i>T. Kudo, M. Fujiwara, M. Satou, A. Hasegawa, K. Abe</i>	79
04-93: Deformation Properties of V-4Cr-4Ti in Compression <i>M.B. Toloczko, R.J. Kurtz</i>	80
04-94: Evaluation of Interfacial Strength between Yttrium Oxides and Vanadium <i>M. Satou, M.Kakie, M.Fujiwara, T.Sawada, T.Komatsu, K.Abe</i>	80
04-95: Impurity behavior in V-4Cr-4Ti-Y alloys produced by levitation melting <i>T. Nagasaka, M. Satou, T. Hino, T. Muroga, K. Abe, T. Chuto, T. Iikubo</i>	81
04-96: Effects of Si, Al and Y Additions on Neutron Irradiation Behavior of V-Cr-Ti type Alloys <i>T.Hino, M.Satou, M.Fujiwara, T.Nagasaka, K.Abe</i>	81
04-97: Helium Gas Permeability of SiC/SiC Composite After Heat Cycles <i>T. Hino, E. Hayashishita, A. Kohyama, Y. Yamauchi, Y. Hirohata</i>	82
04-98: Evaluation of Ion Irradiation Effect on Mechanical Properties of High Purity SiC by Flexural Test <i>S. Ikedaa, T. Hinokib, K.H. Park b, A. Kohyamab</i>	83
04-100: Electrical Conductivity of SiC/SiC <i>G.E. Youngblood, E. Thomsen, G. Coffey</i>	84
04-101: Bubbles in Neutron Irradiated SiC Fibers <i>D.S. Gelles, G.E. Youngblood</i>	84
04-102: Effect of Al and Be as Transmutation Products on Formation and Growth of Helium Bubbles in SiC/SiC Composites <i>T. Taguchi, N. Igawa, E. Wakai, S. Jitsukawa, L.L. Snead, A. Hasegawa</i>	85
04-103: Swelling and Time-Dependent Crack Growth in SiC/SiC Composites <i>C.H. Henager, Jr.</i>	86

04-104: Microstructural Evolution Analysis on NITE SiC/SiC Composite Using TEM Examination and Dual-Ion Experiments <i>Hirotsu Kishimoto, Kazumi Ozawa, Akira Kohyama, Okinobu.Hashitomi</i>	86
04-105: The Microstructural Evolution of SiC/SiC composites under Multiple-beam Ion Irradiation at Elevated Temperature <i>Ji-Jung Kai, Hsu-Tsu Keng, Zi-Huai Zeng, Fu-Rong Chen</i>	87
04-107: Slip-Infiltration of SiC-Fiber Preforms for Production of SiC/SiC Composites <i>S. Novak, G. Drazic</i>	88
04-108: Irradiation Creep of Chemically Vapor Deposited Silicon Carbide as Estimated by Bend Stress Relaxation Method <i>Yutai Katoh, Lance L. Snead</i>	88
PLENARY SESSION 05 – MATERIALS CHALLENGES FOR NEXT STEP FUSION DEVICES	
05.1: Materials Challenges for ITER – Current Status and Future Activities <i>V. Barabash, the ITER International Team, A. Peacock, S. Fabritsiev, G. Kalinin, S. Zinkle, A. Rowcliffe, J.-W. Rensman, A.A. Tavassoli, P.J. Karditsas, F. Gillemot, M. Akiba</i> 1.....	92
05.2: The role of ITER and associated facilities on the pathway towards fusion energy <i>M.Seki</i>	93
PARALLEL ORAL SESSION 06A – TEST BLANKET MODULES FOR ITER	
06A.1: Test Blanket Modules in ITER: an overview on proposed designs and required DEMO relevant materials <i>L. Giancarli, V. Chuyanov, M. Abdou, M. Akiba, B.G. Hong, R. Lässer, C. Pan, Y. Strebkov, the TBWG Team</i>	96
06A.2: Structural Materials for TBMs in ITER <i>V.M.Chernov, M.V.Leonteva-Smirnova, M.M.Potapenko, V.R.Barabash, D.T.Hoelzer, R.L.Klueh, R.Kurtz, S. Zinkle, E.Diegele, R.Laesser, R. Lindau, E. Lucon, A.Moeslang, M. Rieth, B. van der Schaaf, T. Muroga, S. Jitsukawa, Chuanhong Pan, Yican Wu</i>	96
06A.3: The Irradiation Performance of TBM Welds in Relation to Their Heat Treatment <i>J. Rensman, E. Rigal, R. Meyder, A. Li Puma</i>	97
06A.4: Status and Perspective of the R&D on Solid Breeder Materials for Testing in ITER TBMs <i>A. Ying, M. Akiba, L.V. Boccaccini, M. Enoeda, K. Hayashi, R. Knitter, J.D. Lulewicz, S. Sharafat, J. van der Laan, Z.Y. Wen</i>	98
06A.5: ITER Test Blanket Module Functional Materials <i>C.P.C. Wong, V. Chernov, A. Kimura, Y. Katoh, N. Morley, T. Muroga, K.W. Song, Y.C. Wu, M. Zmitko</i>	99
PARALLEL ORAL SESSION 06B – FERRITIC STEELS - II	
06B.1: Recent Progress in US-Japan Collaborative Research on Ferritic Steels R&D <i>Akihiko Kimura, Ryuta Kasada, Akira Kohyama, Hiroyasu Tanigawa, Takanori Hirose, Kiyoyuki Shiba, Shiro Jitsukawa, Satoshi Ohtsuka, Shigeharu Ukai, Mikhail A. Sokolov, Ronald L. Klueh, Takuya Yamamoto, G. Robert Odette</i>	102
06B.2: Fracture Toughness and Charpy Impact Properties of Several RAFS Before and After Irradiation in HFIR <i>M.A. Sokolov, H. Tanigawa, G.R.Odette, K. Shiba2, R.L.Klueh</i>	103
06B.3: Implications of the Temperature Dependence of the Arrest Toughness of Ferrite to an Invariant Master Curve Shape <i>M. Hribernik, G.R. Odette</i>	103
06B.4: Thermal Creep Behavior of Japanese Reduced Activation Martensitic Steels <i>K. Shiba, T. Nakata, Y. Kohno, M. Ando, H. Tanigawa</i>	104

06B.5: Structure Features of the Heat Resistant RAFMS RUSFER-EK-181.

M.V. Leonteva-Smirnova, V.M. Chernov, A.G. Ioltukhovskiy, Yu.R. Kolobov, E.N. Kozlov, T.M. Bulanova, Z.E. Ostrovskiy, A.I. Blokhin, B.K. Kardashev..... 105

PARALLEL ORAL SESSION 07A – IFMIF & SPECIALIZED TEST TECHNIQUES

07A.1: The Role of IFMIF in the Roadmap Toward Fusion Power System

Hideyuki TAKATSU, Masayoshi SUGIMOTO, Shiro JITSUKAWA2, Hideki MATSUI..... 108

07A.2: Evaluation and Validation of D-Li Cross-Section Data for the IFMIF Neutron Source Term Simulation

U. Fischer, M. Avrigeanu, P. Pereslavitsev, S.P. Simakov, I. Schmuck..... 109

07A.3: Preliminary Assessment of the Safety of IFMIF

N.P. Taylor, B. Brañas, E. Eriksson, T. Pinna, L. Rodriguez-Rodrigo, S. Ciattaglia, R. Laesser..... 110

07A.4: Thermo-Structural Design of the Replaceable Backwall in IFMIF Liquid Lithium Target

H. Nakamura, M. Ida, K. Shiba, K. Shimizu, M. Sugimoto..... 110

07A.5: The Role of Small Specimen Test Technology in Fusion Materials Development

G.E. Lucas, G.R. Odette, H. Matsui, A. Möslang, P. Spätig, J. Rensman..... 111

PARALLEL ORAL SESSION 07B – DEVELOPMENT OF ODS STEELS

07B.1: Current Status and Future Prospects of ODS Steel Development for Fusion

R. Lindau, D.T. Hoelzer, G.R. Odette, A. Kimura, S. Ukai..... 114

07B.2: Kinetic Monte Carlo Simulations of Nanocluster Formation and Structure in Nanostructured Ferritic Alloys

M.J. Alinger, B.D. Wirth, G.R. Odette..... 115

07B.3: Nano-Mesosopic Structural Control of 9Cr ODS Martensitic Steel for Improving Creep Strength

S. Ohtsuka, S. Ukai, M. Fujiwara, H. Sakasegawa, T. Kaito, T. Narita..... 115

07B.4: Influence of Particle Dispersions on the High-Temperature Strength of Ferritic Alloys

D.T. Hoelzer, J. Bentley, M.A. Sokolov, M.K. Miller, G.R. Odette, M.J. Alinger..... 116

07B.5: Model Experiment for Minor Alloying Element Effect of Dispersing Nano-Particles in ODS Steel

S. Ohnuki, M. Murata, K. Oka, S. Yamashita, N. Akasaka, S. Ukai, H. Tanigawa..... 117

POSTER 8 SESSION

08-2: Clustering Behavior of Ni, Mn and Si in Irradiated and Thermally Aged RPV Steels

Naoki Soneda, Kenji Dohi, Toshiharu Ohnuma..... 120

08-3: Wigner Energy Predicted by Dislocation Accumulation Model

K.Niwase..... 120

08-4: Effects of Cold Work on Degree of Sensitization of Type 316L Stainless Steel

Tomohiro NODA, Hideaki OHKUBO, Hang-Sik CHO, Akihiko KIMURA..... 121

08-6: Creep mechanism of V-4Cr-4Ti alloys after thermal creep in a vacuum

Ken-ichi Fukumoto, Takuya Nagasaka, Takeo Muroga, Nobuyasu Nita, Hideki Matsui..... 122

08-7: Oxidation Behavior of a V-4Cr-4Ti Alloy During the Commercial Processing of Thin-Wall Tubing

A.F.Rowcliffe, D.T.Hoelzer, C.M.Young, R.J.Kurtz..... 123

08-8: Dynamic and static hydrogen effects on mechanical properties in Vanadium alloys <i>T. Yasuda, S. Ohnuki, K. Yashiki, T. Suda, S. Watanabe, T. Nagasaka, T. Muroga</i>	123
08-9: Studies of Reactor Irradiation Effect on Hydrogen Isotopes Release from Vanadium Alloy V4Cr4Ti <i>T. Kulsartov, V.Shestakov, Y. Chikhay, Y.Kenzhin, A.Kolbayenkov, I.Tazhibayeva</i>	124
08-10: Effects of 2.1%Ti Addition on Tensile Properties and Microstructures of an Ultra-Fine Grained V-1.7%Y Alloy with Nano-Sized Y2O3 and YN <i>H. Kurishitaa, S. Odab, K. Nakaib S. Kobayashib, T. Kuwabarac, M Hasegawaa, H. Matuia</i>	124
08-11: Effect of Ti(CON) Plates on Dynamic Strain Aging in V-Cr-Ti Alloys <i>D.T. Hoelzer, J. Bentley, A.F. Rowcliffe</i>	125
08-12: Effect of Internal Oxidation on Microstructure and Mechanical Properties of Vanadium Alloys <i>A.N. Tyumentsev, V.M. Chernov, A.D. Korotaev, S.V. Ovchinnikov, Yu.P. Pinzhin, M.M. Potapenko, A.K. Shikov</i>	125
08-13: Irradiation induced precipitates in vanadium alloys studied by atom probe microanalysis <i>N. Nita, Y. Anma, H. Matsui, T. Ohkubo, K. Hono</i>	126
08-14: Retention and Desorption Behavior of Helium in Oxidized V-4Cr-4Ti Alloy <i>D. Oku, T. Yamada, Y. Hirohata, Y. Yamauchi, T. Hino</i>	127
08-15: Creep of V-4Cr-4Ti Pressurized Tube Specimens <i>D.S. Gelles, R.J. Kurtz</i>	127
08-16: Diffusional behavior of tritium in V-4Ti-4Cr alloy <i>K. Hashizume, J. Masuda, T. Otsuka, T. Tanabe, Y. Hatano, Y. Nakamura, T. Nagasaka, T. Muroga</i>	128
08-17: The Diffusion Behaviors of Interstitial Impurities in V 4Cr-4Ti Alloys under Ion Irradiation. <i>M. Hatakeyama, S. Tamura, T. Muroga, N. Yoshida, M. Hasegawa, H. Matsui</i>	129
08-18: Dissolution of Hydrogen Isotopes into V-4Cr-4Ti Alloy <i>Y. Hatano, R. Hayakawa, L. Wan, M. Matsuyama, T. Nagasaka, T. Muroga, Y. Nakamura, K. Watanabe</i>	130
08-19: Helium in Irradiated Iron: a Multi-Scale Study <i>T. Seletskaya, Yu .N. Osetsky, R.E. Stoller, G.M. Stocks</i>	130
08-20: Thermal Activated Helium Induced Swelling of Beryllium Irradiated up to Fusion Reactor Relevant Doses <i>L. Sannen, M. Scibetta, S. van den Berghe, A. Leenaers, G. Verpoucke, E. Rabaglino</i>	131
08-21: Radiation Enhanced Diffusion of Hydrogen in Insulating Materials Under Reactor Irradiation <i>B. Tsuchiya, T. Shikama, S. Nagata, K. Toh, M. Narui, M. Yamazaki</i>	131
08-22: Diffusion of He Interstitials and Small He Clusters at Grain Boundaries in α-Fe <i>F. Gao, R.J. Kurtz, H.L. Heinisch</i>	132
08-23: Helium desorption behavior in tungsten irradiated with low energy helium ions at high temperature <i>T. Baba, H. Iwakiri, N. Yoshida</i>	132
08-24: The Effects of Helium on Irradiation Damage in Single Crystal Iron <i>Maria A. Okuniewski, Chaitanya S. Deo, Srinivasan G. Srivilliputhur, Stuart A. Maloy, Mike I. Baskes, Michael R. James, James F. Stubbins</i>	133
08-25: Microstructure change and helium release due to tensile stress on austenitic stainless steel implanted low energy helium ion <i>T. Kawakami, K. Tokunaga, N. Yoshida</i>	134

08-26: Evaluation of helium effects on ODS ferritic steels under ion irradiation <i>K. Yutani¹, H. Kishimoto, R. Kasada, A. Kimura,</i>	135
08-27: Cavity formation in Iron and Eurofer-97 due to He Implantation and Neutron Irradiation <i>M. Eldrup, B.N. Singh, P. Jung</i>	135
08-28: Synergistic Effect of PKA and Helium in Fe-0.1 % He Matrix <i>Jinnan YU, Robin Schaeublin</i>	136
08-29: Behavior of Helium in Steel Cr12W2VTaB under Various Implantation Temperatures <i>I.I. Chernov, S.Yu. Binyukova, B.A. Kalin, Myo Htet Win, Than Swe, S.V. Chubarov, A.N. Kalashnikov, A.G. Yoltukhovsky, M.V. Leontyeva-Smirnova</i>	136
08-30: Dynamic Monte--Carlo modeling of hydrogen isotope reactive-diffusive transport in porous graphite <i>R. Schneider, A. Rai, A. Mutzke, M. Warrier, E. Salonen, K. Nordlund</i>	137
08-31: Computer Simulation of Defect Accumulation Processes in Tungsten under Low Energy Helium Ion Irradiation <i>Yoshiyuki Watanabe, Hiroto Iwakiri, Naoaki Yoshida</i>	138
08-32: Mechanisms of Retention and Blistering in Near-Surface Region of Tungsten Exposed to High Flux Deuterium Plasmas of Tens of eV <i>W.M. Shu, G.-N. Luo, M.F. Nishi</i>	139
08-33: Atomistic Modeling of the Interaction of He With Interfaces in Fe <i>R.J. Kurtz, F. Gao, H.L. Heinisch, B.D. Wirth, G.R. Odette, T. Yamamoto</i>	139
08-34: 08-34APPLICATION OF THE MASTER CURVE TO INHOMOGENEOUS FERRITIC/MARTENSITIC STEEL <i>M.A. Sokolov, H. Tanigawa</i>	140
08-35: In-reactor Creep Rupture Properties of Ferritic martensitic Stainless Steel <i>S.Mizuta, S.Ukai</i>	140
08-36: Mechanical Properties and Microstructure of China Low Activation Martensitic Steel Compared with JLF-1 JOYO-II HEAT <i>Y. Li, Q.Huang, Y.Wu, T.Nagasaka, T.Muroga</i>	141
08-37: Creep Behavior of Reduced Activation Ferritic/Martensitic steels Irradiated at 573 and 773 K up to 5 dpa <i>Masami Ando, Meimei Li, Hiroyasu Tanigawa, Martin L. Grossbeck, Sa-Woong Kim, Tomotsugu Sawai, Kiyoyuki Shiba, Yutaka Kohno, Akira Kohyama</i>	141
08-38: Numerical investigation by finite element simulation of the ball punch test: application to tempered martensitic steels <i>E. Campitelli, P. Spätig, J. Bertsch</i>	142
08-39: Interface reactions and control of diffusion at the interface between SiC fibres and EUROFER 97 <i>S. Levchuk, S. Lindig, A. Brendel, H. Bolt</i>	142
08-40: Dynamical Interaction of helium bubbles with Phase Boundaries or Grain Boundaries in Fe-Cr Ferritic Alloys <i>Kotaro Ono, Kazuto Arakawa,2 Mitsutaka Miyamoto</i>	143
08-41: Hydrogen Transport and Trapping in EUROFER'97 <i>G.A. Esteban, A. Peña, I. Urra, F. Legarda, B. Riccardi</i>	143
08-42: A Critical Stress-Critical Area Statistical Model of the KJc(T) Curve for MA957 in the Cleavage Transition <i>W.J. Yanga,b, G.R. Odette, T. Yamamoto, P. Miao, M.J. Alingera, M. Hribernika, J.H. Leeb</i>	144
08-43: Ductile Brittle Transition Behavior of F82H After High Concentration He Implantation at 550C <i>M.Ishiga, A.Hasegawa, Y.Wakabayashi, K.Abe, S.Jitsukawa</i>	144

08-44: The Influence of Thermal Parameters on Thermal-Fatigue Resistance of Reduced Activation Martensitic Steels <i>L. Pilloni, Gianni Filacchioni</i>	145
08-45: The Heat Resistant RAFMS RUSFER-EK-181 for Fusion and Fast Breeder Power Reactors Applications <i>A.G. Ioltukhovskiy, V.M. Chernov, M.V. Leonteva-Smirnova, V.V. Novikov, L.I. Reviznikov, M.I. Solonin, V.V. Tsvelev, A.V. Vatulin1, T.M. Bulanova, V.N. Golovanov, V.E. Shamardin</i>	145
08-46: Preliminary Experimental Investigation on Hot Isostatic Pressing Diffusion Bonding for CLAM <i>C. Li, Q. Huang, Y. Li, Y. Feng, M. Zhang, M.Chen, L. Peng, Y. Wu</i>	146
08-47: Evaluation of Fracture Toughness Master Curve Shift of JMTR Irradiated F82H Using Small Specimens <i>T. Yamamoto, G.R. Odette, D. Gragg, H. Kurishita, H. Matsui, W.J. Yang, M. Narui, M. Yamazaki</i>	146
08-48: A Critical Stress-Critical Area Statistical Model of the KJc(T) Curve for MA957 in the Cleavage Transition <i>W.J. Yang, G.R. Odette, T. Yamamoto, P. Miao, M.J. Alinger, M. Hribernik</i>	147
08-49: Tensile and transient burst properties of advanced ferritic/martensitic steel (PNC-FMS) claddings after neutron irradiation <i>Y. Yano, T. Yoshitake, S. Yamashita, N. Akasaka, H. Takahashi</i>	147
08-50: Deuterium Retention of Boronized Ferritic Steel Wall in JFT-2M Tokamak <i>Y. Yamauchi, T. Hino, K. Takemoto, K. Tsuzuki, Y. Kusama, Y. Hirohata</i>	148
08-51: Assessment of Different Welding Techniques for Joining EUROFER Blanket Components <i>Michael Rieth</i>	148
08-52: Grain size effects of irradiated nanocrystalline metals using atomistic simulations <i>M. Samaras, P.M. Derlet, H. Van Swygenhoven, W. Hoffelner, M. Victoria</i> ,	149
08-53: Atomistic Modeling of Self-Interstitial Dislocation Loops in Fe-Cr Alloys <i>Jae-Hyeok Shim, Brian D. Wirth</i>	149
08-54: The role of stacking fault energy in the MD simulations of irradiation induced defects in Ni <i>Z. Yao, M.J. Caturla, R. Schäublin</i>	150
08-55: Image Simulations of Small Dislocation Loops Based on the Howie-Basinski Equations <i>Zhongfu Zhou, S L Dudarev, M L Jenkins, A P Sutton, M A Kirk</i>	150
08-56: Atomic-scale dynamics of dislocation interaction with vacancy agglomerates in neutron irradiated bcc iron <i>Yu.N. Osetsky, D.J. Bacon, B.N.Singh</i>	151
08-57: The Effects of Irradiation on the Deformation of Cu: A Comparison between Dislocation Dynamics Modeling and Experiments <i>Ming Wen, Zhiqiang Wang, Nasr M. Ghoniem, Bachu Singh</i>	151
08-58: Molecular Dynamics Study of Influence of Helium Bombardment on Carbon Nanocluster Structure Evolution <i>A.M. Ilyin</i>	152
08-59: First-principles calculation of vacancy- solute element interaction in bcc iron <i>Toshiharu Ohnuma, Naoki Soneda, Misako Iwasawa</i>	152
08-60: On the capture of small glissile dislocation loops by large sessile loops <i>Taira Okita, Wilhelm Wolfer, Naoto Sekimura</i> ,	153

08-62: Molecular Dynamics Simulations study of hydrogen isotopic effects in amorphous Silica using an empirical potential <i>F. Mota, M. León, A. Ibarra, M.-J. Caturla, J. Mollá, J.M. Perlado</i>	153
08-63: Modeling of Sub-cascade Formation in Irradiated Materials by Fast Neutrons with Fusion Energy Spectrum <i>A.I.Ryazanov, E.V.Metelkin, E.A.Semenov</i>	154
08-64: Dislocation Loop Formation in Irradiated Binary Vanadium Alloys <i>A.I.Ryazanov, V.A.Egorov, H.Matsui</i>	154
08-65: Effects of Over Sized Element Sn on Diffusion of Interstitial Clusters in Ni Irradiated by Ions and Neutrons <i>Q. Xu, T. Yoshiie, H. Watanabe N. Yoshida</i>	155
08-66: Small Angle Neutron Scattering Study of Radiation Damage in a Proton-Irradiated Tempered Martensitic Steel <i>G. Yu, P. Spätig, R. Schäublin, N. Baluc, S. Van Petegem</i>	155
08-67: Application of Positron Beam Doppler Broadening Technique to Ion Beam Irradiation in Iron and Nickel <i>Takeo Iwai, Hidetsugu Tsuchida, Misa Awano</i>	156
08-68: Microstructural Investigation, Using Small Angle Neutron Scattering (SANS), of OPTIFER Steel Under Low Dose Neutron Irradiation and Subsequent High Temperature Tempering <i>R. Coppola, R. Lindau, M. Magnani, A. Möslang, M. Valli</i>	156
08-69: The Evolution of Stacking Fault Tetrahedra and Damage Accumulation in FCC Metals under Cascade Damage Conditions <i>S.I. Golubov, B.N. Singh, H. Trinkaus, S.J. Zinkle, R.E. Stoller</i>	157
08-71: The influence of stepwise change of irradiation temperature on microstructural evolution of precipitate strengthened copper alloys <i>Y. Sumino, H. Watanabe, N. Yoshida</i>	158
08-72: One dimensional Motion of Interstitial Clusters in Ni-Au Alloy <i>K. Sato, T. Yoshiie, Q. Xu</i>	159
08-73: MEAM Potentials of Fe, Fe-Cu, Fe-Cr and Fe-C Systems for Cascade Simulations <i>Je-Wook Jang, Byeong-Joo Lee, Jun-Hwa Hong</i>	159
08-74: New Mechanism of the Formation of <100> Dislocation Loops in Iron <i>K. Arakawa, M. Hatanaka, T. Taniwaki, K. Ono, H. Mori</i>	160
08-75: A Comparison of Radiation Damage in Ion Irradiated Monolithic CVD- and NITE-SiC <i>Sosuke Kondo, Tatsuya Hinoki, Kazuya Shimoda, Akira Kohyama</i>	161
08-76: Effect of Solute Elements of Ni Alloys on Swelling and Blistering under He+ and D+ Ion Irradiation <i>E. Wakai, T. Ezawa, T. Takenaka, J. Imamura, T. Tanabe, R. Oshima</i>	161
08-77: Modeling Cascade Aging and Dose Rate Effects in Ferritic Alloys <i>B.D. Wirth, G.R. Odette</i>	162
08-78: Using High Entropy Alloys as the First Wall Structural Materials for Fusion Reactors <i>Ji-Jung Kai, Juen-Wei Yeh, Fu-Rong Chen</i>	162
08-79: Overview of the Recent Russian Materials and Technologies R&D Activities Related to ITER and DEMO Constructions <i>A.Shikov, V.Beliakov</i>	163

08-80: ITER Vacuum Vessel In-Wall Shielding Block Material Requirements <i>C.S. Kim, S.J. Son, S. Cho</i>	163
08-81: Verification of Design Rules for EUROFER under TBM Operating Conditions <i>R. Sunyk, J. Aktaa</i>	164
08-82: The effect of low dose neutron irradiation on mechanical properties, electrical resistivity and fracture character of NiAl bronze for ITER <i>V. Barabash, A. Pokrovsky, A. Fabritsiev</i>	164
08-83: Narrow-gap, Thick section, Hybrid Laser Conduction Welding Developments for ITER Vacuum Vessel <i>G. de Dinechin, F. Janin, P. Aubert, S. Morgan, S. Williams, L. Jones</i>	165
08-84: Irradiation stress relaxation of ITER candidate bolting materials <i>F. Schmalz, J. Rensman, J.G. van der Laan, D.S. d'Hulst, J. Boskeljon, P. ten Pierick</i>	165
08-85: Thermal Analysis of the US Solid Breeder TBM (Quarter port Submodule) <i>A. Abou-Sena, A. Ying, M. Abdou</i>	166
08-86: Carbon transport, deposition and fuel accumulation in castellated structures exposed in TEXTOR <i>A. Litnovsky, V. Philipps, A. Kirschner, P. Wienhold, G. Sergienko, O. Schmitz, A. Kreter, P. Karduck, M. Blöme, B. Emmoth, M. Rubel</i>	166
08-87: On the effects of the supporting frame on the radiation-induced damage of HCLL-TBM structural material <i>P. Chiovaro, P.A. Di Maio, E. Oliveri, G. Vella</i>	167
08-88: Experimental Research of Pb(83)Li(17) for Blanket Cooling <i>Semenov A.V., Beznosov A.V., Pinaev S.S., Konstantinov V.L., Baranova O.V.</i>	167
08-89: Numerical Characterization of Thermo-mechanical Performance of Breeder Pebble Beds <i>Zhiyong An, Alice Ying, Mohamed Abdou</i>	168
08-90: High Energy Heavy Ion Induced Structural Disorder in Li₂TiO₃ <i>T. Nakazawa, A. Naito, T. Aruga, V. Grismanovs, Y. Chimi, A. Iwase, S. Jitsukawa</i>	168
08-91: Oxidation and stability studies of Beryllium Titanate <i>E. Alves, L.C. Alves, N. Franco, M.R. da Silva, A. Paúl</i>	169
08-92: Compatibility between Be-Ti alloys and F82H <i>B. Tsuchiya, T. Ishida, H. Kawamura</i>	169
08-94: Post-irradiation examination of the Ceramic Breeder materials from the Pebble Bed Assemblies Irradiation for the HCPB Blanket concept <i>J.B.J. Hegeman, A.J. Magielsen, M.P. Stijkel, J.H. Fokkens, J.G. van der Laan</i>	170
08-95: Validation of Potential Models for Li₂O in Classical Molecular Dynamics Simulation <i>Takuji Oda, Yoshihisa Oya, Satoru Tanaka</i>	170
08-96: Saturation in Degradation of Thermal Diffusivity of Neutron-Irradiated Ceramics at 3×10²⁶ n/m² <i>Masafumi Akiyoshi, Toyohiko Yano, Yoshiaki Tachi, Hiromi Nakano</i>	171
08-97: Some Features of Lithium Penetration into Beryllium Under corrosion Tests in Be-liquid Li-V4 Ti 4 Cr alloy system <i>I.B. Kupriyanov, E.N. Bazaleev, L.A. Kurbatova, I.E. Lyublinski, A.V. Vertkov, V.A. Evtikhin</i>	171
08-98: Electrical Insulating Property of Ceramic Coating Materials in Radiation and High Temperature Environment <i>Teruya Tanaka, Ryo Ngayasu, Akihiro Suzuki, Akihiko Sawada, Takeo Muroga, Fuminobu Sato, Toshiji Ikeda, Toshiyuki Iida</i>	172

08-99: Measuring Creep Strain of Individual Li₂TiO₃ Spheroids <i>Daniel Papp, Patrick Calderoni, Alice Ying</i>	173
PLENARY SESSION 09 – MATERIALS FOR PRESENT AND FUTURE NUCLEAR POWER SYSTEMS - LESSONS LEARNED FOR FUSION	
09.1: Critical Questions in Materials Science and Engineering for Successful Development of Fusion Power <i>E.E. Bloom, J.T. Busby, C.E. Duty, P.J. Maziasz, T.E. McGreevy, B.E. Nelson, B.A. Pint, P.F. Tortorelli, S.J. Zinkle</i>	176
09.2: Materials Degradation in Commercial Nuclear Power Reactors: Lessons Learned and Implications for Future Fusion Power Systems <i>Gary S. Was</i>	176
PARALLEL ORAL SESSION 10A – MATERIALS ISSUES FOR CURRENT AND FUTURE FISSION ENERGY SYSTEMS	
10A.1: Generation IV Reactor Integrated Materials Program <i>William R. Corwin</i>	180
10A.2: Overview of PERFECT Project for Prediction of Irradiation Damage in Fission Reactor Components <i>J.-L. Boutard, D. Lidbury, O. Diard, B. Marini, S. van Dick, S. Bugat</i>	180
10A.3: Cross-Cutting Materials Issues for the Fusion and Spallation Neutron Environments <i>Stuart A. Maloy, Louis K. Mansur</i>	181
10A.4: Materials Design of NITE-SiC/SiC for VHTR/GFR and Fusion <i>Akira Kohyama, T. Hinoki, J.S. Park, M. Sato</i>	181
PARALLEL ORAL SESSION 10B – FUNCTIONAL MATERIALS	
10B.1: Effects of Neutron Irradiation on the Properties of Functional Materials for Fusion Applications <i>Tatsuo Shikama, E.R. Hodgson</i>	184
10B.2: Radiation Effects on the Deuterium Diffusion in Oxides <i>A. Ibarra , A. Muñoz-Martín , P. Martín , A. Climent-Font , E.R. Hodgson</i>	185
10B.3: H and OH Effects on Ion Beam Induced Luminescence in SiO₂ <i>S. Nagata, S. Yamamoto, A. Inoue, K. Toh, B. Tsuchiya, T. Shikama</i>	185
10B.4: Surface Electrical Degradation of Helium Implanted SiO₂ <i>S.M. González, A. Morono, E.R. Hodgson</i>	186
10B.5: Crystallization and Microstructure of Lithium Orthosilicate Pebbles <i>Regina Knitter, Birgit Alm, Georg Roth</i>	186
PARALLEL ORAL SESSION 11A – H & HE EFFECTS IN IRRADIATED MATERIALS	
11A.1: The Transport and Fate of Helium in Nanostructured Ferritic Alloys at Fusion Relevant He/DPA Ratios and DPA Rates <i>T. Yamamotoa, G.R. Odettea, N. Hashimoto, D.T. Hoelzerb, H. Tanigawac</i>	190
11A.2: Retention and Desorption of Deuterium in Model Alloys of Ferritic Steel <i>H. Iwakiri, C. Ebina² Y. Takao, M. Miyamoto, K. Okuno, N. Yoshida</i>	190
11A.3: Effect of Implanted Helium on Flow and Fracture Properties of 9Cr Martensitic Steels <i>J. Henry, L. Vincent¹ X. Averty¹ B. Marini¹ P. Jung</i>	191

11A.4: The Transport and Fate of Helium in Martensitic Steels at Fusion Relevant He/DPA Ratios and DPA Rates <i>R.J. Kurtza, G.R. Odetteb, T. Yamamoto, D.S. Gellesa, P. Miaob, B.M. Olivera</i>	191
11A.5: The Behavior of Martensitic Steels at High Irradiation Dose and Helium Concentration – an Overview of STIP Results <i>Y. Dai, X. Jia, M. Grosse, S.A. Maloy, B.M. Oliver, P. Marmy, F. Groeschel</i>	192
PARALLEL ORAL SESSION 11B – SiC COMPOSITES	
11B.1: Current Status and Critical Issues for Development of SiC Composites for Fusion Applications <i>Y. Katoh, L.L. Snead, C.H. Henager, Jr., A. Hasegawa, A. Kohyama, B. Riccardi, H. Hegeman</i>	196
11B.2: Mechanical Characterisation of Commercial Grade Tyranno SA/CVI SiC Composites <i>B. Riccardi, M. Labanti, E. Trentini, S. Roccella, E. Visca</i>	197
11B.3: Elevated Temperature Swelling of Neutron Irradiated SiC <i>L.L. Snead, Y. Katoh, S.D. Connery</i>	197
11B.4: Irradiation Effect on NITE-SiC/SiC Composites <i>Tatsuya Hinoki, Akira Kohyama, Yutai Katoh, Kazumi Ozawa, Takashi Nozawa</i>	198
11B.5: Strength of Neutron Irradiated SiC/SiC Composite with Multilayer SiC/PyC Interface <i>Takashi Nozawa, Yutai Katoh, Kazumi Ozawa, Lance L. Snead, Akira Kohyama</i>	198
POSTER 12 SESSION	
12-2: Investigations of Helium Effects on Cavity Evolution in Metals under Cascade Damage Conditions <i>S.I. Golubov, B.N. Singh, H. Trinkaus, A.M. Ovcharenko, R.E. Stoller, S.J. Zinkle</i>	202
12-3: Thermal Helium Desorption of Helium-Implanted Iron <i>Stephen C. Glade, Lisa Ventelon, Brian D. Wirth</i>	203
12-4: Characteristics of Traps for Hydrogen in Helium irradiated Copper <i>I. Takagi, M. Akiyoshi, N. Matsubara, T. Nishiuchi, K. Moritani, T. Sasaki, H. Moriyama</i>	203
12-5: Kinetic Monte Carlo Simulations of Substitutional Helium Diffusion <i>L. Ventelon, B.D. Wirth, F. Gao, R.J. Kurtz</i>	204
12-6: Effect of Helium on Mechanical Properties in HIP-Bonded Reduced-Activation Ferritic/Martensitic Steel <i>K. Furuya, E. Wakai, M. Sato, K. Oka, F. Takada, Y. Kato</i>	204
12-7: Effect of Irradiation Damage up to 50 dpa on Swelling Behavior and Microstructural Development in SiC/SiC Composites <i>T. Taguchi, N. Igawa, S. Miwa, E. Wakai, S. Jitsukawa, L.L. Snead, A. Hasegawa</i>	205
12-8: Atomistic Modeling of Helium Interacting with Dislocations in Alpha-Iron <i>H.L. Heinisch, F. Gao, R.J. Kurtz</i>	205
12-9: Effectiveness of Helium Bubbles as Traps for Hydrogen <i>S.Yu. Binyukova, I.I. Chernov, B.A. Kalin, Than Swe</i>	206
12-10: Effects of Precipitation Microstructures on Bubble Distribution in Helium-Embrittled Fe-Ni-Cr Alloys <i>Norikazu Yamamoto, Yoshiharu Murase, Johsei Nagakawa</i>	206

12-11: Irradiation Embrittlement of Martensitic Steels: Hardening and Non-Hardening Mechanisms and the Effect of Helium <i>T. Yamamoto, G.R. Odette, H. Kishimoto</i>	208
12-12: The study of interaction between dislocation and helium atom <i>R. Sugano, K. Morishita, N. Yoshida</i>	208
12-13: Effects of interstitial impurity on behavior of helium-defect complexes in vanadium studied by THDS <i>N. Nita, K. Miyawaki, Y. Fujiwara, H. Matsui</i>	209
12-14: Desorption of Tritium and Helium from High Dose Neutron Irradiated Beryllium <i>I.B. Kupriyanov, G.N. Nikolaev, V.V. Vlasov, A.M. Kovalev, V.P. Chakin</i>	209
12-15: The Influence of Hydrogen on the Fatigue Behaviour of Base and GTA Welded Eurofer <i>Marie-Françoise Maday, Luciano Pilloni</i>	210
12-16: Analytical Estimation of Accessibility to Activated Lithium Loop in IFMIF <i>M. Ida, H. Nakamura, T. Nishitani, M. Yamauchi, M. Sugimoto</i>	210
12-17: Irradiation facility LiSoR <i>H. Glasbrenner, F. Gröschel</i>	211
12-18: Designing Experiments for the International Fusion Materials Irradiation Facility <i>R. Kemp1 G.A. Cottrell, H.K.D.H. Bhadesia</i>	212
12-19: Transmutation Analysis of Realistic low-Activation Steels for Magnetic Fusion Reactors and IFMIF <i>O. Cabellos, J. Sanz, N. García-Herranz, S. Díaz, S. Reyes</i>	212
12-20: Reduction of radioactive inventories in the IFMIF test cell <i>S.P. Simakov, U. Fischer, V. Heinzel, F. Wasastjerna, P.P.H. Wilson</i>	213
12-21: Thermo-hydraulics and Technology of Neutron Lithium Target for IFMIF <i>N. Loginov, Yu. Aksyonov, M. Arnoldov, L. Berensky, V. Chernov, V. Fedotovskiy, A. Mikheyev, V. Morozov, H. Nakamura, V. Shishulin</i>	214
12-22: Neutron Induced Activation of the IFMIF Lithium Loop Corrosion Products <i>S. Boeriu, G. Cambi, D.G. Ceperaga, M. Frisoni, T. Pinna</i>	215
12-23: Neutron and deuteron activation calculations for IFMIF <i>R.A. Forrest, M.J. Loughlin</i>	216
12-25: Cavitation detection to avoid material erosion in Liquid Lithium Target Facility for IFMIF <i>G. Dell'Orco, H.Horiike, Hiroo Nakamura</i>	217
12-26: Material Irradiation Conditions for IFMIF Medium Flux Test Module <i>P. Vladimirov, A. Möslang, U. Fischer, S. Simakov</i>	218
12-27: Shielding analyses of the IFMIF Test Cell <i>Y. Chen, U. Fischer, S.P. Simakov, F. Wasastjerna</i>	218
12-28: Corrosion Resistance and Thermal Aging Behavior of High-Cr Oxide Dispersion Strengthened Ferritic Steels in Super-Critical Pressurized Water <i>H.S. Cho, A. Kimura, S. Ukai, M. Fujiwara</i>	219

12-29: Small Angle Neutron Scattering Study of ODS martensitic/ferritic materials <i>M.H. Mathona, Y. de Carlanb, L. Chaffronc, S. Ukaîd, C. Cayrone, A.Alamob</i>	219
12-30: Fracture toughness of the newly designed EU ODS EUROFER steel <i>P. Fernández, M. Serrano, A.M. Lancha, J. Lapeña</i>	220
12-31: Potential Microstructural Features Affecting Creep Property of ODS Steel <i>H. Sakasegawa, S. Ohtsuka, S. Ukai, H. Tanigawa, M. Fujiwara, H. Ogiwara, A. Kohayama</i>	220
12-32: Effect of Milling on Morphological and Microstructural Properties of Raw-powder Particles for High-Cr Oxide Dispersion Strengthened Ferritic Steels Production <i>N.Y. Iwata, A. Kimura, M. Fujiwara, N. Kawashima</i>	221
12-33: Mechanical and Microstructural Behavior of Y2O3 ODS EUROFER 97 <i>T. Leguey, A. Muñoz, M.A. Monge, V. de Castro, P. Fernández, A.M. Lancha, R. Pareja</i>	221
12-34: Microstructural characterisation of reduced activation EUROFER ODS alloy after long-term annealing <i>A. Paúl, L.C. Alves, E. Alves, J.A. Odrizola</i>	222
12-35: Improvement of 9%Cr ODS Steel by a Microstructural Modification during the Post Consolidation Process <i>Jinsung Jang, Soon Hyung Hong, Jun Hwa Hong</i>	222
12-37: Properties of Friction Welding between 9Cr-ODS martensitic and Ferritic-martensitic steels <i>T.Uwaba, S.Ukai, T.Nakai, M.Fujiwara</i>	223
12-38: Direct Correlation Between Chromium Phases and Impact Behavior Of ODS Steels <i>M. Klimiankou, R.Lindau, A. Möslang</i>	223
12-39: Microstructural Evaluation of Hot Rolled Plates of Oxide Dispersion Strengthened 8Cr-2W and 8Cr-1W Steels <i>Kei Shinozuka, Kiyoyuki Shiba, Kazuyuki Nakamura, Manabu Tamura, Hisao Esaka</i>	224
12-40: Structure and stability of nano-size oxide in ODS steels <i>K. Oka, M. Murata, S. Yamashita, N. Akasaka, H. Tanigawa, T. Sawai, T. Suda, S. Watanabe, S. Ohnuki</i>	224
12-41: Heavy-ion Irradiation Effects on the Morphology of Complex Oxide Particles in Oxide Dispersion Strengthening Ferritic Steels <i>H. Kishimoto, K. Yutani, R. Kasada, A. Kimura, O. Hashitomi</i>	225
12-42: The Microstructure and of MA957 Nanostructured Ferritic Alloy Joints Produced by Friction Stir and Electrospark Deposition Welding <i>P. Miao, G.R. Odette, M. Alingera, J. Bernathc, J. Gouldc, R. Millerd</i>	225
12-44: Ductile-to-Brittle Transition Temperature Shift in Ferritic Steels under High Fluence Neutron Irradiation <i>R.G. Faulkner, Z. Lu, J.F. Knott, M. Novovic, H. Hurchand, S.D. Kenny, R. Smith, P.E.J. Flewitt</i>	226
12-45: Effect of Neutron Dose and Temperature on Tensile and Fracture Toughness Properties of Titanium Alloys <i>S. Tähtinen, P. Moilanen, B.N. Singh</i>	226
12-46: Influence of radiation induced defects on fracture behavior in highly pure SiC <i>K.H. Park, T. Hinoki, A. Kohyama</i>	227
12-47: Modeling of austenitic alloy hardening under irradiation <i>Oleksandr Shepelyev, Naoto Sekimura, Hiroaki Abe</i>	227

12-48: Effects of Reductions in Strain Hardening on Irradiation Induced Transition Temperature Shifts <i>M.Y. He, G.R. Odette</i>	228
12-49: Brittle-Ductile Transitions in Vanadium and Iron-Chromium <i>T.D. Joseph, M. Tanaka, A.J. Wilkinson, S.G. Roberts</i>	228
12-50: Comparison of irradiation creep of γ-TiAl alloy under He and H- implantation <i>J. Chen, P. Jung, W. Hoffelner</i>	229
12-51: Effect of Cold Work on the Radiation-Induced Deformation of Austenitic Stainless Steels <i>Johsei Nagakawa, Keiko Ueno, Yoshiharu Murase¹, Norikazu Yamamoto</i>	229
12-52: The Dependence of Obstacle Strength on Copper Precipitate Diameter in Fe-Cu Alloys Studied by in-situ TEM Observation <i>K. Nogiwa, N. Nita, H. Matsui</i>	230
12-53: Fracture toughness characterization of JLF-1 steel after irradiation in HFIR up to 5 dpa <i>M.A. Sokolov, A. Kimura, H.Tanigawa, S. Jitsukawa</i>	231
12-54: Irradiation Behavior of Ti – 4Al – 2V (PT – 3V) Alloy for ITER Shield Blanket Modules Flexible Attachment <i>B.S. Rodchenkov, A.V. Kozlov, G.M. Kalinin, Yu.G.Kuznetsov, Yu.S.Strebkov</i>	232
12-55: Surface Roughening Mechanisms for Tungsten Exposed to Laser, Ion, and X-ray Pulses <i>Michael Andersen, Nasr M. Ghoniem</i>	233
12-56: Examination of Crack Tip Microstructures in F82H on the Lower Shelf <i>D.S. Gelles, G.R. Odette, P. Spätig, J.W. Rensman</i>	233
12-57: Effect Of Irradiation Temperature On Radiation Hardening Of Pure Copper And Copper-Based Alloy <i>S.A. Fabritsiev, A.S. Pokrovsky</i>	234
12-58: Effect Of Bake-Out Regime On Recovery Of Properties And Microstructure Of Neutron-Irradiated Pure Copper And Copper-Based Alloys <i>S.A. Fabritsiev, A.S. Pokrovsky</i>	235
12-59: Transport of Carbon Impurity Using ¹³CH₄ Gas Puffing in JT-60U <i>Y. Ishimoto, Y. Gotoh, T. Arai, K. Masaki, N. Miya, K. Tsuzuki, N. Asakura, T. Tanabe</i>	236
12-61: Neutronics Calculations for Waste Characterisation in IFMIF <i>M.J. Loughlin, R.A.Forrest</i>	236
12-62: Activation properties of Nb₃Sn, NbTi and GFRP irradiated with D-T neutrons <i>K. Ochiai, A. Nishimura, T. Nishitani, S.Nishijima</i>	237
12-63: Evaluation Of Tritium Behavior In Concrete Materials <i>Kazuya Furuichi, Hiroki Takata, Kazunari Katayama, Toshiharu Takeishi, Masabumi Nishikawa, Takumi Hayashi, Kazuhiro Kobayashi, Haruyuki Namba</i>	237
12-64: Recycling of fusion reactor materials <i>L. Ooms, V. Massaut</i>	238
12-65: Some Oxidation and Inflammation Characteristics of Compact Beryllium and Its Fine Particles <i>D.A. Davydov, O.V. Kholopova, B.N. Kolbasov</i>	238
12-66: Carbon and hydrogen behavior with formation of carbon deposition layer by hydrogen RF plasma <i>Kazunari Katayama, Hiroyasu Nagase, Masabumi Nishikawa</i>	239

12-67: Coolant Pipe Activation due to Sequential Reactions by Charged Particles <i>Michinori Yamauchi, Jun-ichi Hori, Takeo Nishitani, Kentaro Ochiai, Satoshi Sato, Hiromitsu Kawasaki</i>	239
12-68: Desorption Mechanism of Water Adsorbed on Metal Oxide Films <i>Ryushi Jinzenji, Takuji Oda, Shintaro Miyazaki, Yasuhisa Oya, Satoru Tanaka</i>	240
12-69: Recycling and Clearance of Vanadium Alloys in Fusion Reactors <i>S.A. Bartenev, A. Ciampichetti, B.N. Kolbasov, I.B. Kvasnitskij, P.V. Romanov, V.N. Romanovskij, M. Zucchetti</i>	241
12-70: Activation characteristics of the materials in the fusion driven subcritical system FDS-I <i>M. Chen, Q.Huang, Y.Chen, Y. Wu</i>	242
12-71: Development of China Liquid Lithium-Lead Blanket and Its Material Technology <i>Y. Wu, Q. Huang, C. Li, W. Wang, S. Liu, H. Chen, S. Zheng, Y. Chen, D. Huang, the FDS Team</i>	242
12-72: Irradiation Effect in Zr-Based Metallic Glasses <i>S. Higashi, K. Murooka, S. Nagata, B. Tsuchiya, T. Shikama</i>	243
12-73: An Interactive Web-based Fusion Materials Properties Database <i>S. Sharafat, N. Ghoniem, R. Odette, T. Yamamoto, S. Zinkle</i>	244
12-74: Development of 300°C heat resistant boron-loaded resin for neutron shielding <i>A. Morioka, S. Sakurai, K. Okuno, H. Yamada, S. Sato, Y. Verzilov, A. Kaminaga, T. Nishitani, H. Tamai, Y. Kudo, S. Yoshida, M. Matsukawa</i>	245
12-75: Materials selection and requirements for DEMO in a fast track development of fusion power <i>D Ward, N Taylor, I Cook</i>	245
12-76: Deformation and Damage of RAFM Steels under Thermomechanical Loading: A Challenge for Constitutive Equations <i>J. Aktaa, M. Klotz, C. Petersen</i>	246
12-77: Multiaxial Fatigue Behavior of EUROFER 97: Experiments and Modeling <i>M. Weick, J. Aktaa</i>	246
12-78: Statistical Assessment of Representativeness of Mechanical Testing Results To Substantiate The Guaranteed Level of Strength Properties of ITER Structural Materials <i>K.S. Gartvig, S.A. Fabritsiev</i>	247
12-79: Extending ITER Materials Design to Welded Joints <i>A-A.F. Tavassoli</i>	248
12-80: High Heat Flux Performance of He-cooled Divertor Module for DEMO Reactor <i>A. Gervash, R. Giniyatulin, T. Ihli, V. Kuznetsov, A. Makhankov, I. Mazul, P. Norajitra, N. Yablokov</i>	248
12-84: Strengthen CVI-SiC Matrix in SiC/SiC Composites by SiC Nanowires <i>Wen Yang, Hiroshi Araki, Akira Kohyama, Somsri Thaveethavorn, Hiroshi Suzuki, Tetsuji Noda</i>	249
12-85: Microstructure and Mechanical Property of Fiber/Matrix Interphase in SiC/SiC Composites after Irradiation <i>Kazumi Ozawa, Tatsuya Hinoki, Takashi Nozawa, Sosuke Kondo, Akira Kohyama</i>	250
12-86: Efforts on Large Scale Production of NITE-SiC/SiC for Test Blanket Module of ITER <i>Joon-Soo Park, Akira Kohyama, Tatsuya Hinoki</i>	251

12-87: Mechanical Properties of Tyranno-SA/SiC Composite Prepared by the Whisker Growing Assisted CVI Process <i>J.Y.Park#, K.H.Park, S.M.Kang, W.-J.Kim, W.S.Ryu</i>	251
12-88: Mechanical Properties of SiC/SiC Composite with Magnesium-Silicon Oxide Interphase <i>N. Igawa, T. Taguchi, R. Yamada, Y. Ishii, S. Jitsukawa</i>	252
12-89: Reaction Sintering of Two-Dimensional Silicon Carbide Fiber-Reinforced Silicon Carbide Composite by Sheet Stacking Method <i>Katsumi Yoshida, Hideki Mukai, Masamitsu Imai, Kazuaki Hashimoto, Yoshitomo Toda, Hideki Hyuga, Naoki Kondo, Hideki Kita, Toyohiko Yano</i>	253
12-90: Effect of Neutron Irradiation on Tensile Properties of Unidirectional Silicon Carbide Composites <i>Yutai Katoh, Takashi Nozawa, Lance Snead, Tatsuya Hinoki</i>	254
12-91: High temperature tests of 2D and 3D SiCf/SiC composites <i>J.B.J. Hegeman, M. Jong, P. ten Pierick, J.G. van der Laan</i>	254
12-92: Material Compatibilities Studies between SiC and Solid Breeding Materials for High-Temperature Gas Cooling Blanket System <i>A. Hasegawa, T. Murayama, M.Satou, T.Shikama, K.Abe</i>	255
12-93: Numerical Analysis of Mechanical Testing for Evaluating Shear Strength of SiC/SiC Composite Joints <i>H. Serizawa, D. Fujita, C.A. Lewinsohn, M. Singh, H. Murakawa</i>	255
12-94: Mechanical Properties of LPS-SiC Ceramics with Al₂O₃-Y₂O₃-SiO₂ System <i>Han Ki Yoon, Hun Chae Jung, Sang Pill Lee, Byung Chal Shin, Joon Soo Park, Akira Kohyama</i>	256
12-95: A comprising steady-state creep model for the austenitic AISI 316 L(N) steel <i>Michael Rieth</i>	257
12-96: Tensile Properties and Electrical Conductivity of Unirradiated and Irradiated Cu-Ni-Be <i>Steven J. Zinkle</i>	257
12-97: Influence of Mechanical Stresses on Radiation Swelling of an Austenitic Stainless Steel <i>I.A. Portnykh, A.V. Kozlov, V.L. Panchenko, V.M. Chernov, A.B. Sivak, F.A. Garner</i>	258
12-98: The Synergistic Influence of Irradiation Temperature and Atomic Displacement Rate on Microstructural Evolution of Ion-Irradiated Model Austenitic Alloy Fe-15Cr-16Ni <i>T. Sato, T. Okita, N. Sekimura, T. Iwai, F.A. Garner</i>	258
12-99: Effect Of Irradiation Dose On Mechanical Properties And Fracture Character Of Cu//SS Joints For ITER <i>A.S. Pokrovsky1 S.A. Fabritsiev, A. Peacock, A.Gerwash2 V.R.Barabash</i>	259
12-100: Novel Composite Heat Sink Material for the Divertor of future Fusion Reactors <i>A. Brendel, T. Köck, T. Brendel, H. Bolt</i>	260
12-101: Investigation of CuCrZr Alloys using Positron Annihilation Spectroscopy <i>Peter Domonkos, Morten Eldrup, Bachu N.Singh</i>	261
12-102: Characterization of 12Cr18Ni10Ti Stainless Steel Irradiated at Low Displacement Rates in BN-350 Reactor <i>O.P.Maksimkin, K.V.Tsai, L.G.Turubarova, T.A.Doronina, F.A.Garner</i>	261
12-103: Swelling and Microstructure of Cold-worked Austenitic Stainless Steel ChS-68 after High Dose Neutron Irradiation <i>S.I. Porollo, Yu.V. Konobeev, F.A. Garner</i>	262

12-104: Hold-Time Effects on the Fatigue Life of Copper and Copper Alloys for Fusion Applications <i>James Stubbins, Xianglin Wu, Xiao Pan, Meimei Li, Bachu Singh</i>	262
12-105: Microstructure of Austenitic Stainless Steel EC 316LN Irradiated in SINQ Target-4 up to 20 dpa and 1700 appm He at 430°C <i>X. Jia, M. Grosse, Y. Dai</i>	263
12-106: The Influence of Cold-work Level on the Irradiation Creep of AISI 316 Stainless Steel Irradiated as Pressurized Tubes in the EBR-II Fast Reactor <i>E.R. Gilbert, F.A. Garner</i>	264
PLENARY SESSION 14 – INTERGRATION OF EXPERIMENTS AND MULTISCALE MODELS TO SOLVE THE GRAND CHALLENGES OF FUSION MATERIALS AND THE MATERIALS DESIGN INTERFACE	
14.1: Integration of Experiments and Modeling in Fusion Materials Research <i>H. Matsui</i>	268
14.2: Bridges between Materials and Design <i>E. Diegele, Co-authors to be determined</i>	268
PARALLEL ORAL SESSION 15A – MATERIALS – DESIGN INTERFACE	
15A.1: Challenges of Structures and Materials Research in the Aerospace Industry <i>S. Jason Hatakeyama</i>	272
15A.2: In-Pile Performance of Pebble Bed Assemblies and Implications for the HCPB Blanket Concept <i>J.B.J. Hegeman, A.J. Magjelsen, J.G. van der Laan, J.H. Fokkens, B.J. Pijlgroms, D.J. Ketema, J. Reimann</i>	272
15A.3: Large-Scale Finite Element Modeling of the Thermo-Mechanical Behavior of the Dual Coolant US-ITER TBM Incorporating Damage Evolution <i>S. Sharafat, N. Ghoniem, J. El-Awady, S. Liu, R. Odette, T. Yamamoto, J. Blanchard³, E. Diegele, S. Zinkle</i>	273
15A.4: Materials and Design Interface of In-Vessel Components for Fusion Reactors <i>M. Akiba, S. Suzuki</i>	274
PARALLEL ORAL SESSION 15B – BLANKET ENGINEERING - II	
15B.1: Correlation Between Tritium Release and Thermal Annealing of Irradiation Damages in Neutron-Irradiated Li₂SiO₃ <i>Yusuke Nishikawa, Makoto Oyaidzu, Akira Yoshikawa, Kenzo Munakata, Moritami Okada, Masabumi Nishikawa, Kenji Okuno</i>	278
15B.2: A SIMS Study of the Distribution of Tritium in Neutron-Irradiated Beryllium Pebbles <i>E. Rabaglino, G. Tamborini, A. Moeslang, M. Betti</i>	278
15B.3: An Overview of Recent Progress in Studying Redox Control in FLiBe Using Dissolved Beryllium <i>M. Simpson, S. Fukada, G. Smolik, David Petti, John P. Sharpe, Robert Anderl, Y. Oya, T. Terai, D.-K. Sze, K. Okuno, Y. Hatano, A. Sagara</i>	279
15B.4: Swelling, Mechanical Properties and Microstructure of Beryllium Irradiated at 200 °C up to Extremely High Neutron Doses <i>V.P. Chakin, M.N. Svyatkin, A.O. Posevin, I.B. Kupriyanov</i>	280
15B.5: Recent Results on Beryllium and Beryllides in Japan <i>Y. Mishima, N. Yoshida, H. Kawamura, K. Ishida, Y. Hatano, T. Shibayama, K. Munakata, Y. Sato, M. Uchida, K. Tsuchiya, S. Tanaka</i>	281

PARALLEL ORAL SESSION 16A – MULTISCALE MODELING OF RADIATION EFFECTS

16A.1: Computational Modeling of Material Failure <i>Alan Needleman</i>	284
16A.2: An Atomic-Scale-Simulation Study of Hardening Due to Copper Precipitates in Iron <i>D.J. Bacon, Yu. N. Osetsky, R.E. Stoller</i>	284
16A.3: Modeling of Helium Bubble Formation in BCC Metals <i>Kazunori Morishita, Ryuichiro Sugano</i>	285
16A.4: Systematic Group-Specific Trends for Point Defects in BCC Transition Metals: An Ab-initio Study <i>Duc Nguyen-Manh, Sergei Dudarev, Andrew Horsfield</i>	286
16A.5: Multiscale Modeling of He Transport and Fate in Irradiated Nanostructured Ferritic Alloys <i>B.D. Wirth, T. Yamamoto, G.R. Odette, R.J. Kurtz, F. Gao, H.L. Heinisch</i>	286

PARALLEL ORAL SESSION 16B – HIGH HEAT FLUX PLASMA FACING MATERIALS - II

16B.1: Development of Candidate Plasma Facing Materials for Steady State Operation of the EAST Device <i>J.L. Chen, J.G. Li, X.D. Zhang, Q.G. Guo, C.C. Ge</i>	290
16B.2: The JET ITER-like Wall Project <i>G. Piazza, J. Pamela, A. Lioure, F. Le Guern, G.F. Matthews, V. Riccardo, J.P. Coad, J.D. Neilson, J. Likonen, C. Grisolia, H. Maier, T. Hirai, collaborators of the JET ITER-like Project</i>	291
16B.3: High Heat Flux Facility GLADIS – Operational Characteristics and Results of W7-X Pre-Series Target Tests <i>H. Greuner, B. Boeswirth, J. Boscardy, P. McNeely</i>	292
16B.4: Development of Ultra-Fine Grained Tungsten Alloys and Their Mechanical Properties for Fusion Applications <i>H. Kurishita, H. Arakawa, H. Matsui, K. Nakai, S. Kobayashi, Y. Hiraoka, T. Takida, K. Takebe</i>	292
16B.5: Thermal Stability, Nano Structure, and Chemical Erosion of Metal-Doped Carbon Films <i>M. Balden, C. Adelhelm, E. de Juan Pardo, I. Quintana, B.T. Ciecwiwa, J. Roth, M. Sikora</i>	293

POSTER SESSION 17

17-1: Microstructure and Mechanical Properties of Ausformed F82H <i>Tomotsugu Sawai, Eiichi Wakai, Nariaki Okubo, Shiro Jitsukawa</i>	296
17-2: Effects of Microstructure on Fatigue Fracture Mechanism of Reduced Activation Ferritic/Martensitic Steel <i>Sa-Woong KIM, Hiroyasu Tanigawa, Kiyoyuki Shiba, Takanori Hirose, Akira Kohyama</i>	297
17-3: Change of Microstructure and Mechanical Properties of Modified 9%Cr Martensitic Steel during Creep <i>Sung Ho Kim, B.J. Song, Woo Seog Ryu, Jun Hwa Hong</i>	298
17-4: Reduction of toroidal magnetic field ripple with ferritic steel armors in JT-60U <i>S. Sakurai, K. Masaki, Y. Kudo, K. Shinohara, Y. Suzuki, T. Sawai, T. Sasajima, A. Morioka, T. Hayashi, R. Takahashi, T. Fujita, N. Miya, Y. Miura</i>	298
17-5: Mechanical Characterization and Modeling of Brazed Joints of Refractory Alloys <i>Tz. Chehtov, O. Kraft, J. Aktaa</i>	299

17-6: The Effects of Specimen Size on the Cleavage Fracture Toughness of Eurofer97: A Single Variable Experiment <i>R. Yasudaa,b, J. Rensmana,c, G.R. Odettea, T. Yamamotoa, D. Gragga</i>	299
17-7: Precipitation in Ion-Irradiated Reduced-Activation Ferritic/Martensitic Steels <i>Hiroyasu Tanigawa, Hideo Sakasegawa, Hiroyuki Ogiwara, Hirotatsu Kishimoto, Akira Kohyama</i>	300
17-8: Long Term Stability of Finely Dispersed TaC Particle during Tempering 8%Cr-2%W Martensitic Steel <i>Manabu Tamura, Hiroyasu Kusuyama, Kei Shinozuka, Hisao Esaka</i>	300
17-9: Analysis of 300°C neutron irradiation response of Eurofer97 and F82H plate <i>J. Rensman, J.A. Vreeling, J. Boskeljon, R. den Boef, P. ten Pierick, M. Jong</i>	301
17-10: Progress in Development of China Low Activation Martensitic Steel for Fusion Application <i>Q.Y. Huang et. al.</i>	301
17-11: Effect of Dislocation Density on Hydrogen Retention Property of F82H <i>K. Matsuhiro, M. Sugimoto, Y. Ueda, M. Nishikawa</i>	302
17-13: Dynamic Strain Ageing Behavior on Tensile and Low Cycle Fatigue Properties of JLF-1 Steel in Vacuum <i>Huailin Li, Arata Nishimura, Takuya Nagasaka, Takeo Muroga</i>	302
17-14: On the Effects of Irradiation Induced Displacement Damage and Helium on the Yield Stress Changes and Hardening and Non-hardening Embrittlement of 8Cr Tempered Martensitic Steels: Compilation and Analysis of Existing Data <i>T. Yamamoto, G.R. Odette, H. Kishimoto, P. Miao, J-W. Rensman</i>	303
17-15: Microstructural evolution of a heavily neutron-irradiated ODS ferritic steel (MA957) at elevated temperature <i>S. Yamashitaa, N.Akasakaa, S. Ukaia, S. Ohnukib</i>	304
17-16: The Effects of Consolidation Temperature, Strength and Microstructure on Fracture Toughness of Nanostructured Ferritic Alloys <i>P. Miao a, G.R. Odette a, M. Alingera,b, D. Hoelzerc, T. Yamamotoa</i>	304
17-17: Fracture toughness and Tensile Properties of Nano Structured Ferritic Steel 12YWT <i>M.A.Sokolov, D.T.Hoelzer, R. Stoller, D.A.McClintock</i>	305
17-18: First Results On The Characterization Of The Advanced EU Reference RAFM ODS-EUROFER Steel <i>R. Lindau, M. Klimiankou, A. Möslang, M. Rieth</i>	305
17-19: Characterization of microstructural evolution in nanostructured ferritic alloys using positron annihilation <i>M.J. Alinger, S.C. Glade, G.R. Odette, Y. Nagai, M. Hasegawa, B.D. Wirth</i>	306
17-20: Solid State and Resistance Joining Technologies for Fusion Energy Systems <i>Robert Miller, Jerry Gould, Jeff Bernath, Robert Odette</i>	306
17-21: Effect of Irradiation on the Microstructure and the Mechanical Properties of Oxide Dispersion Strengthened EUROFER97 Steel <i>A. Ramar, N. Baluc, R. Schaeublin</i>	307
17-22: Water Corrosion Resistance of ODS Ferritic – Martensitic Steel Tubes <i>T.Narita, S.Ukai, T.Kaito, S.Ohtsuka, Y.Matsuda</i>	307
17-23: Thermal Aging Embrittlement of High Cr Oxide Dispersion Strengthened Steels <i>J.S. Lee, I.S. Kim, C.H. Jang, A. Kimura</i>	308

17-24: Low Cycle Fatigue Properties of ODS Ferritic-martensitic Steels at High Temperature <i>S.Ukai, S.Ohtsuka</i>	308
17-25: Effects of Neutron Irradiation on the Mechanical Properties of High-Cr Oxide Dispersion Strengthened Ferritic Steels <i>H.S. Cho, A. Kimura, S. Ukai, M. Fujiwara</i>	309
17-26: Pre- and Post-Deformation Microstructures of the Oxide Dispersion Strengthened Ferritic Steels before and after Neutron Irradiation <i>Ryuta KASADA, Naoki TODA, Hang-Sik CHO, Akihiko KIMURA</i>	309
17-27: Tritium distribution measurement of the tile gap of JT-60U <i>K.Sugiyama, T.Tanabe, K.Masaki, N.Miya</i>	310
17-28: Tritium Permeation Barrier for the First Wall of a Fusion Reactor <i>H. Maier, D. Levchuk, F. Koch, Th. Köck, H. Bolt</i>	310
17-29: Performance of a hydrogen sensor in Pb-16Li <i>Andrea Ciampichetti, Italo Ricapito, Gianluca Benamati, Massimo Zucchetti</i>	311
17-30: Reacton Rate of Be with Fluorine Ion for Flibe Redox Control <i>S. Fukada, G.R. Smolik, M. Simpson, R.A. Anderl, P. Sharpe, Y. Oya, T. Terai, K. Okuno, D.-K. Sze, D. Petti, Y. Hatano, A. Sagara</i>	311
17-31: Measurement of tritium trapped in the irradiation defects produced by high energy proton and spallation neutron in SS316 <i>Hirofumi NAKAMURA, Kazuhiro KOBAYASHI, Sumi YOKOYAMA, Shigeru SAITOH, Toshihiko YAMANISHI, Kenji KIKUCHI</i>	312
17-32: Behavior of Hydrogen Isotopes Irradiated in LiAlO₂ <i>Tianyong Luo, Takuji Oda, Yasuhisa Oya, Satoru Tanaka</i>	312
17-33: He-O Glow Discharge at Elevated Temperatures for the Removal of the Codeposited C/H Layer <i>C.L. Kunz, R.A. Causey, M.W. Clift, D.F. Cowgill, B.E. Mills, W.R. Wampler</i>	313
17-34: Thermal release rate of tritium trapped in bulk and plasma exposed surfaces of carbon specimens obtained from JET divertor <i>N. Bekris, J.P. Coad, C.H. Skinner, E. Damm, M. Glugla, W. Nägele</i>	313
17-35: Efficiency of tritium removal techniques in castellated structures <i>Jose Antonio Ferreira, F.L.Tabarés, D. Tafalla</i>	314
17-36: Applicability of Pd-Cu Alloy to Self-Developing Gas Chromatography of Hydrogen Isotopes <i>M. Matsuyama, H. Sugiyama, M. Hara, K. Watanabe</i>	314
17-37: Tritium Control Design of the HCPB Blanket Concept <i>L.V. Boccaccini, N. Bekris, M. Glugla, R. Meyder, J. Reimann, I. Ricapito, A.J. Magjelsen</i>	315
17-38: HTO Electrolysis Method by Using Proton Exchange Membrane Fuel Cell <i>Hiroki Takata, Masabumi Nishikawa, Takayuki Egawa, Nobukazu Mizuno</i>	315
17-39: Measurement System for In-pile Tritium Monitoring from Li₂TiO₃ Ceramics at WWRK <i>I.Tazhibayeva, Y.Chikhay, V.Shestakov, T.Kulsartov, H.Kawamura</i>	316
17-40: Erbium oxide as a new promising tritium permeation barrier <i>D. Levchuk, F. Koch, H. Maier, H. Bolt, A. Suzuki</i>	317
17-41: On the mechanism of the disproportionation of ZrCo hydrides <i>Sergey Beloglazov, N. Bekris, M. Sirch, R.-D. Penzhorn, M. Glugla</i>	317

17-42: Neutron Elastic Recoil Detection for Hydrogen Isotope Analysis in Fusion Materials <i>Naoyoshi Kubota, Kentaro Ochiai, Keitaro Kondo, Takeo Nishitani</i>	318
17-43: Characteristics on tritium release during plasma operation on JT-60U <i>Akira Oikawa, Hirofumi Nakamura, Atsushi Kaminaga, Kei Masaki, Takeshi Yamazaki, Tetsuo Tanabe</i>	318
17-44: Pulsed-laser ablation of co-deposits on JT-60 graphite tile <i>Y. Sakawa, K. Sugiyama, T. Shibahara, K. Sato, T. Tanabe</i>	319
17-45: Tritium recovery from isotropic graphite and CFC used to plasma facing material <i>Toshiharu Takeishi, Kazunari Katayama, Masabumi Nishikawa</i>	319
17-46: In-reactor vacuum RIED experiment on sapphire <i>Marc Decréton, Eric Hodgson, Hans Ooms</i>	320
17-47: Long Term Stability of Erbium Oxide Coatings <i>Akihiko Sawada, Bruce A. Pint, Akihiro Suzuki, Freimut Koch, Hans Maier, Takayuki Terai, Takeo Muroga</i>	320
17-48: Chemical Shift of Characteristic X-Ray Wavelength in Silicon-Containing Ceramics due to Neutron Irradiation <i>Toyohiko Yano, Saisyun Yamazaki, Hiroko Kawano, Keiichi Katayama</i>	321
17-49: Physical Property Changes of Crystalline and Non-Crystalline SiO₂ due to Neutron Irradiation and Recovery by Subsequent Annealing <i>Toyohiko Yano, Kunio Fukuda, Masamitsu Imai, Hiroyuki Miyazaki</i>	322
17-50: Thermally induced emf in unirradiated MI cables <i>R. Vila, E.R.Hodgson</i>	323
17-51: Thermal stability of neutron irradiation effects on KS4V and KU1 fused silica <i>Mónica León Pichel, A. Ibarra, D. Bravo, F.J. López, A. Rascón</i>	323
17-52: Anomalous Radioluminescence behaviour for KS-4V <i>A. Moroño, E.R. Hodgson</i>	324
17-53: Luminescence of Cr doped alumina induced by charged particle irradiation <i>A. Inouye, T. Suzuki, S. Nagata, K. Toh, B. Tsuchiya, T. Shikama</i>	324
17-54: Isotopic effect on Thermal Conductivities of Silicon Single Crystal and Diamond Film <i>Tetsuji Nodaa, Hiroshi Suzukia, Hiroshi Arakia, Wen Yanga, Takefumi Ishikurab</i>	325
17-55: In-situ Measurements of Optical Fibers under Gamma ray Irradiation <i>Dan Sporea, Adelina Sporea, Bogdan Constantinescu</i>	326
17-56: Research for High Temperature Measurement Using Fused Silica Core Optical Fiber <i>A. Honda, K. Toh, S. Nagata, B. Tsuchiya, T. Shikama</i>	327
17-57: The role of the fused silica stoichiometry on the intrinsic defects concentration <i>J. Mollá, F. Mota, M. León1, A. Ibarra, M.-J. Caturla, J.M. Perlado</i>	327
17-58: Search for Luminescent Materials under 14 MeV Neutron Irradiation <i>K. Toh, T. Shikama, S. Nagata, B. Tsuchiya, M. Yamauchi, T. Nishitani</i>	328
17-59: Deformation-induced Dislocation Channeling and Martensite Formation in Neutron-irradiated 316 Stainless Steel <i>N. Hashimoto, T.S. Byun</i>	328

17-60: The Penetration Of Tritium Through Aging Austenitic Radiation-Resistant Reactor Steel <i>Yu. N.Zouev, Yu. N.Dolinskii, I.V.Podgornova, V.V.Sagaradze</i>	329
17-61: On the Tearing Resistance of AISI-316L – Austenitic Stainless Steel <i>R. Chaouadi, J.L. Puzzolante, E. Lucon, M. Scibetta</i>	329
17-62: Ageing Effect on the Properties of CuCrZr Alloy Used for the ITER HHF Components <i>G.M.Kalinin, A.D.Ivanov, A.N.Obushev, B.S.Rodchenkov, M.V.Rodin, Y.S.Strebkov</i>	330
17-63: Dislocation structure in deformed irradiated single crystal Ni <i>Z. Yao, R. Schäublin</i>	330
17-64: Effects of Water Chemistry on Stress Corrosion Cracking Behavior of Unirradiated and Irradiated Type 316LN-IG <i>Yukio Miwa, Takashi Tsukada, Shiro Jitsukawa</i>	331
17-65: Low-temperature Embrittlement of Austenitic Steel Examined using Ring-pull Tensile Tests and Microhardness Measurements <i>V.S. Neustroev, E.V. Boev, F.A. Garner</i>	332
17-66: Atomic-scale modeling of primary damage in copper <i>R.E.Voskoboynikov, Yu. N.Osetsky, D.J.Bacon</i>	332
17-67: In-situ SCC Observation on Neutron Irradiated Thermally-sensitized Austenitic Stainless Steel <i>Junichi Nakano, Takashi Tsukada, Shinya Endo, Koichiro Hide</i>	333
17-68: Demonstration of the Separate Temperature and dpa Rate Dependencies of Void Swelling in 300 Series Stainless Steels <i>F.A. Garner, Pacific Northwest National Laboratory, Richland WA USA</i>	334
17-69: Preliminary Study on Thick Alumina Coating Fabricated by SHS Method for Fusion Application <i>Y.Feng, Q.Huang, M.Zhang, Y.Li, C.Li, M.Chen, Y.Wu</i>	335
17-70: Preliminary Experiment on Corrosion behavior of CLAM in Liquid LiPb Eutectic <i>M. Zhang, Q. Huang, Y Wu, Y.Feng, C. Li, Y..Li</i>	335
17-71: Residual Stresses in Vapor-Deposited Erbia Coatings <i>Alan F. Jankowski, Jeffrey P. Hayes</i>	336
17-72: Material Probe Study on Boronized Wall of LHD <i>Y. Nobuta, N. Ashikawa, T. Hino, Y. Hirohata, K. Nishimura, A. Sagara, S. Masuzaki, N. Noda, O.Motojima, LHD Experimental Group</i>	336
17-73: Charging of V-4Cr-4Ti by Oxygen to create in-situ Insulator Coating <i>O. Yeliseyeva, T. Muroga, A. Suzuki, Z. Yao, A. Lukyanenko</i>	337
17-74: Compatibility of Multi-Layer, Electrically Insulating Coatings For the Vanadium-Lithium Blanket <i>B.A. Pint, J.L. Moser, A. Jankowski, J. Hayes</i>	337
17-75: Correlation of Yield Stress and Microhardness in 08Cr16Ni11Mo3 Irradiated to High Dose in the BN 350 Fast Reactor <i>M.N. Gusev, O.P. Maksimkin, O.V. Tivanova, N.S. Silnaygina, F.A. Garner</i>	338
17-76: A Semi-automated Small Specimen Fracture Testing Instrument <i>K.A. Fields, G.R. Odette, T. Yamamoto, D. Gragg, D. Klingensmith</i>	339
17-77: New microscopic specimen testing method using a bending of micro cantilever specimen <i>S. Fujita, N. Nita, Y. Satoh, H. Matsui</i>	339

17-78: Miniaturized Fracture Stress Tests for Thin-Walled Tubular SiC Specimens <i>Thak Sang Byun, Edgar Lara-Curzio, Lance L. Snead, Yutai Katoh</i>	340
17-79: An Application of the Gurson's Model to Small Scale Fracture-Mechanical Specimens <i>R. Sunyk, F. Reusch</i>	340
17-80: Master Curve Evaluation of Transition Fracture Toughness of a JLF-1 Reduced-Activation Ferritic Steel <i>Ryuta KASADA, Akihiko KIMURA</i>	341
17-81: Fracture Behaviors of F82H Steel for Small Size Specimens <i>E. Wakai, H. Ohtsuka, S. Matsukawa, S. Jitsukawa</i>	341
17-82: High temperature FIMEC tests on NFR materials <i>R. Montanari, G. Filacchioni, B. Iacovone, P. Plini, B. Riccardi</i>	342
17-83: Small Fracture-toughness Specimen for Post-irradiation Experiments: Validation and Results <i>H.-C. Schneider, R. Rolli, J. Aktaa</i>	342
17-84: A Universal Relationship Between Hardness and Flow Stress <i>M.Y. He, G.R. Odette, T. Yamamoto, D. Klingensmith</i>	343
17-86: The Effects of Specimen Size on the Cleavage Fracture Toughness of Eurofer97: A Model Based Analysis <i>W.J. Yanga,b, R. Yasudaa,c, G.R. Odette, T. Yamamoto, J. Rensmana,d</i>	343
17-89: Manufacturing of W/Cu Mock-ups for Plasma Facing Components <i>Shen Wei ping, Li Qiang, Ge Chang chun</i>	344
17-90: Effects of helium and hydrogen implantation on damage during pulse high heat loading in tungsten <i>K. Tokunaga, T. Fujiwara, K. Ezato, S. Suzuki, M. Akiba, N. Yoshida</i>	344
17-91: CFC/Cu Joints for ITER Divertor: New Developments <i>M.Ferraris, M.Salvo, V.Casalegno, M.Merola</i>	345
17-92: Development of A Helium-Cooled Divertor: Material Choice, Technological Studies, and Proof Of Principle <i>P. Norajitra, U. Fischer, A. Gervash, R. Giniyatulin, N. Holstein, T. Ihli, G. Janeschitz, W. Krauss, R. Kruessmann, V. Kuznetsov, A. Makhankov, I. Mazul, A. Moeslang, I. Ovchinnikov, M. Rieth, B. Zeep</i>	346
17-93: Features of Plasma Sprayed Beryllium Armor for the ITER First Wall <i>R.E. Nygren, D.L. Youchison, K.J. Hollis</i>	347
17-94: Methods of Elastic Modulus Determination for Irradiated Porous Tungsten Coatings <i>J.H. You, T. Höschen, S. Lindig</i>	347
17-95: Silicon Doped Carbon/Cu Joints Based on Amorphous Alloy Brazing for First Wall Application <i>Zhang-jian Zhou, Zhi-hong Zhong, Chang-chun Ge</i>	348
17-96: Effects of Chemical Status of Carbon on Deuterium retention in Carbon Related Materials <i>Makoto Oyaidzu, Hiromi Kimura, Toshihiko Nakahata, Yusuke Nishikawa, Masayuki Tokitani, Yasuhisa Oya, Hiroto Iwakiri, Naoaki Yoshida, Kenji Okuno</i>	348
17-97: Hydrogen isotope behavior in the first wall of JT 60U after DD discharge <i>Y.Oya, M.Oyaidzu, T.Shibahara, K.Sugiyama, A. Yoshikawa, Y. Onishi, Y.Hirohata, Y. Ishimoto, J. Yagyu, T.Arai, K.Masaki, K.Okuno, N.Miya, S.Tanaka, T.Tanabe</i>	349

17-98: Manufacturing of a small scale CFC armored monoblock mockup by hot radial pressing <i>Eliseo Visca, S. Libera, A. Mancini, A. Pizzuto</i>	350
17-99: Damage Evaluation Under Thermal Fatigue of a Vertical Target Full Scale Component for ITER Divertor <i>M. Missirlian, F. Escourbiac, M. Merola, A. Durocher, I. Bobin-Vastra, B. Schedler</i>	351
17-100: Material Damage due to Edge Localized Modes in ITER <i>J. Linke, T. Hirai, O. Ogorodnikova, M. Rödiger</i>	352
17-101: Dependence of oxygen concentration on chemical behavior of deuterium implanted into oxygen contained boron thin film <i>Akira Yoshikawa, Makoto Oyaidzu, Hideo Miyauchi, Yasuhisa Oya, Akio Sagara, Nobuaki Noda, Kenji Okuno</i>	352
17-102: Recent Progress of Plasma Sprayed Tungsten Coated Carbon and Copper <i>Xiang Liu, Zengyu Xu, Shunyan Tao, Yunzhen Cao</i>	353
17-103: Simulation of Redeposition Patterns in the Gaps between Divertor Tiles <i>Kaoru Ohya, Kensuke Inai, Jun Okada, Shinji Ebisu</i>	353
17-104: W-macrobush melt damage simulation after multiple transient events in ITER <i>B.N. Bazylev, G.Janeschitz, I.S. Landman, A.Loarte, S.E. Pestchanyi</i>	354
17-105: IASCC Behavior of the Highly Irradiated Stainless Steel at relatively low temperature, 323K at JMTR <i>T. Ishii, M. Ohmi, J. Nakano, F. Takada, T. Saito, Y. Nagao, M. Shimizu, Y. Miwa, T. Tsukada</i>	354
17-106: Dynamics of deuterium implanted in boron coating film for wall conditioning <i>Toshihiko Nakahata, Akira Yoshikawa, Makoto Oyaidzu, Yasuhisa Oya, Yuki Ishimoto, Kaname Kizu, Jyunichi Yagyu, Naoko Ashikawa, Kiyohiko Nishimura, Naoyuki Miya, Kenji Okuno</i>	355
17-107: In-pile Testing of the ITER First Wall Mock-Ups at Relevant Thermal Loading Conditions <i>N. Litunovsky, A. Gervash, V. Komarov, P. Lorenzetto, I. Mazul, R. Melder</i>	355
PARALLEL ORAL SESSION 18A – RADIATION EFFECTS IN FCC METALS	
18A.1: In-Reactor Deformation Behaviour of Copper and CuCrZr Alloy: Experiments, Results and Implications <i>B.N. Singh, S. Tähtinen, P. Moilanen, P. Jacquet, J. Dekeyser, D.J. Edwards</i>	358
18A.2: Disappearance of the Dose Rate Effect on Swelling Upon C Addition to Fe-Cr-Ni-Ti Alloys <i>N. Sekimura, T. Okita, F.A. Garner</i>	359
18A.3: Effect of Heat Treatments on Precipitate Microstructure and Mechanical Properties of a CuCrZr Alloy <i>D.J. Edwards, B.N. Singh, S. Tähtinen</i>	359
18A.4: Study on the Interaction Between Dislocations and Helium Bubbles in Copper by In-Situ Straining Experiments in Transmission Electron Microscopy <i>N. Nita, H. Matsui</i>	360
18A.5: Atomic-Scale Mechanisms of Cleared Channels Formation in Neutron-Irradiated Low Stacking Fault Energy fcc Metals <i>Yu. N. Osetsky, R.E. Stoller, S. Zinkle, B.N. Singh</i>	361

PARALLEL ORAL SESSION 18B – DEFORMATION AND FRACTURE

18B.1: Plastic Flow Properties and Fracture Toughness Characterization in the Lower Transition of Unirradiated and Irradiated Tempered Martensitic Steels <i>P. Spätig, R. Bonadé, E.N. Campitelli, P. Mueller, G.R. Odette, J.W. Rensman</i>	364
18B.2: Effect of Test Temperature and Strain Rate on Radiation Hardening <i>Steven J. Zinkle, Bachu N. Singh</i>	365
18B.3: Effect of Temperature Change on the Irradiation Hardening of the Structural Alloys for ITER Blanket and ITER TBM Irradiated to 1.5 dpa in JMTR <i>S. Jitsukawa, E. Wakai, N. Okubo, M. Ohmi</i>	365
18B.4: Qualification of CuCrZr Alloy to Stainless Steel Joints <i>S. Tähtinen, P. Moilanen, B.N. Singh</i>	366
18B.5: Mechanical Property Degradation of Ferritic/Martensitic Steels after the Fast Reactor Irradiation “ARBOR 1” <i>C. Petersen, A. Povstyanko, V. Prokhorov, A. Fedoseev, O. Makarov, B. Dafferner</i>	367

PARALLEL ORAL SESSION 19A – VANADIUM AND REFRACTORY METALS

19A.1: Advances in Development of Vanadium Alloys and MHD Insulator Coatings <i>OT. Muroga, J.M. Chen, V.M. Chernov, K. Fukumoto, D.T. Hoelzer, R.J. Kurtz, T. Nagasaka, B.A. Pint, M. Satou, A. Suzuki, H. Watanabe</i>	370
19A.2: Thermal Creep of Two Heats of V-4Cr-4Ti in a Liquid Lithium Environment <i>Meimei Li, T. Nagasaka, D.T. Hoelzer, M.L. Grossbeck, S.J. Zinkle, T. Muroga, K. Fukumoto, H. Matsui, M. Narui</i>	371
19A.3: Influence of Cr and Ti Content on Compatibility of V-Cr-Ti Type Alloys with Liquid Lithium <i>M. Fujiwara, B.A. Pint, T. Muroga, M. Satou, A. Hasegawa, K. Abe</i>	372
19A.4: The Microstructure of Laser Welded V-4Cr-4Ti Alloy after Neutron Irradiation <i>H. Watanabe, K. Yamasaki, N. Yoshida, T. Nagasaka, T. Muroga</i>	372
19A.5: Fracture Toughness Investigations of Tungsten Alloys and Severe Plastic Deformed Tungsten Alloys <i>M. Faleschini, H. Kreuzer, D. Kiener, W. Knabl, R. Pippan</i>	373

PARALLEL ORAL SESSION 19B – CHEMICAL COMPATIBILITY & COATINGS

19B.1: Multilayer Systems for Sustaining High Heat Flux <i>A. Evans</i>	376
19B.2: Coatings and Joining for SiC/SiC Composites for Nuclear Energy Systems <i>C.H. Henager, Jr., Y. Shin, Y. Blum, L.A. Giannuzzi, S.M. Schwarz</i>	376
19B.3: Comparison of Corrosion Behavior of Bare and Hot-Dip Coated EUROFER Steel in Flowing Pb-17Li <i>J. Konyas, W. Krauss, Z. Voss, O. Wedemeyer</i>	377
19B.4: Research on Calcium Zirconate as a Insulating Coating for Li Blanket <i>Akihiro Suzuki, Akihiko Sawada, Freimut Koch, Hans Maier, Takayuki Terai, Takeo Muroga</i>	378
19B.5: Investigation of Pb-Li Compatibility for the Dual Coolant Test Blanket Module <i>B.A. Pint, J.L. Moser, P.F. Tortorelli</i>	379

Plenary Session 01 – Perspectives on Fusion Energy

01.1

Vision for a Sustainable Energy Future

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Sustainable and environmentally benign energy supply has emerged as an issue of national strategic significance. Both electricity generation and transportation require enormous sources of primary energy that only nuclear power appears capable of providing while satisfying the multiple criteria of safety, environment, security, cost, etc. Spent nuclear fuel disposition remains an unresolved issue, however, and it is one which will only worsen with increased nuclear power without radical changes in our approach to the problem. This talk describes how the high-temperature gas reactor, when properly utilized in conjunction with LWRs, can play an important role in a strategy to meet the nation's energy demands for both electricity and transportation for the next century, while requiring only one Yucca Mountain (or its equivalent) for spent fuel storage. That time scale permits an orderly development of fusion, including the important issues of materials selection and qualification, maturation and optimization of the confinement configuration, etc. Seen this way, both fission and fusion are nuclear-based technologies, with fission the bridge to fusion and fusion the ultimate solution to the spent fuel issue.

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01.2

Material science and technology in the roadmap towards a fusion reactor

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The overall objective of the European fusion program is the realization of reactors for power stations, which are economically viable and compatible with a sustainable development, i.e. safe and environmentally responsible. In this strategy, ITER is the single step between the present devices and DEMO (a "demonstration" fusion power station). Its physics and technology objectives insure that the knowledge basis acquired during the construction and exploitation phases will contribute to the overall goal of the program. Among the different elements to be tested, one can cite the Test Blanket Modules, which will be the first nuclear element to be inserted in ITER. The validation of the proposed concepts will be a major step towards a future power plant.

The advancement of the material science and technology is the second critical element of the European strategy. Materials to be assessed encompass structural material, (e.g., RAFM, ODS steels,) SiCf /SiC, refractory metal as W, high heat flux components. In view of the licensing and construction of DEMO, the construction of the IFMIF in the same time scale as ITER is a key element of the present roadmap. The basic understanding of material properties will also require a strong effort in numerical modeling, which is now feasible with the advent of massively parallel computers.

The talk will present the present European view of the roadmap towards a DEMO reactor. The main lines of a program in parallel with the ITER construction (the "Accompanying Program"), in particular those pertaining to material science and technology, will be outlined.

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Parallel Oral Session 02A – Ferritic Steels - I

02A.1

Status of Reduced Activation Ferritic/ Martensitic Steel Development

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Reduced activation ferritic/martensitic (RAFM) steels are the reference structural materials for future fusion power reactors, as they have achieved the greatest technology maturity, i.e., qualified fabrication routes, welding technology and a general industrial experience are already available. However, the temperature window of use of RAFM steels is presently about 350-550°C, the lower value being limited by irradiation-induced embrittlement effects and the upper value by a strong reduction in mechanical strength.

Most irradiation campaigns on large heats of RAFM steels have been completed, but many post-irradiation examinations are still in progress. These examinations include specimens from recent irradiations of the European RAFM steel, i.e., the EUROFER 97, and from earlier irradiated IEA F82H specimens. Recent results from these studies will be summarized. For instance, neutron irradiation of the EUROFER 97 at about 325°C to 32.5 dpa in the BOR60 reactor recently raised some questions on the common expectation that the radiation hardening should saturate at about 15 dpa. Apart from irradiation effects, recent results of activities being pursued in Europe, Japan, and the U.S. will be also summarized and discussed, which relate to the mechanical properties of RAFM steels, their compatibility with H, He and liquid Pb-Li, as well as to manufacturing of Test Blanket Modules (TBMs) and status of data bases and design rules. Note that results of such activities on unirradiated and irradiated RAFM steels have led to heats of new RAFM steels being produced.

In addition to the development of conventional RAFM steels, work on the development of oxide dispersion-strengthened (ODS) RAFM and RAF steels is being pursued. Such steels would allow higher operating temperatures for increased efficiency of future fusion power reactors. Significant progress has been made in the ODS development programs in Europe, Japan, and the U.S. since ICFRM-11 two years ago.

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02A.2

Irradiation Effects on Precipitation and its Impact on the Mechanical Properties of Reduced-Activation Ferritic/Martensitic Steels

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It was previously reported that reduced-activation ferritic/martensitic steels (RAFs), such as F82H-IEA and its heat treatment variant, ORNL9Cr-2WVTa, JLF-1 and 2%Ni-doped F82H, showed a variety of changes in ductile-brittle transition temperature (DBTT) and yield stress after irradiation at 573K up to 5dpa. These differences could not be interpreted solely as an effect of irradiation hardening caused by dislocation loop formation. To address these observations, the precipitation behavior of the irradiated steels was examined by weight analysis, X-ray diffraction analysis and chemical analysis on extraction residues. The results suggested that irradiation affects precipitation as if it was forced to reach the thermal equilibrium state at irradiation temperature 573K, which usually never be achieved by aging. The details of precipitates in the irradiated RAFs were examined to determine their impact on the mechanical properties, which obtained by tensile, Charpy impact, and bend bar toughness tests. Transmission electron microscopy was performed on thin films and extraction replica specimens to analyze the size distribution, chemical composition and crystal structure of precipitates. It turned out that the hardening level normalized by square root of average packet size showed a linear dependence on the increase of extracted precipitate weight. This dependence suggests that the difference in irradiation hardening between RAFs was caused by the different precipitation behavior on packet, block and prior austenitic grain boundaries during irradiation. The simple Hall-Petch law could be applicable to interpret this dependence. Detailed analytical results will be presented and their interpretation discussed.

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02A.3

New Nano-Particle-Strengthened Ferritic/Martensitic Steels by Conventional Thermomechanical Processing

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Martensitic steels are presently the preferred structural materials for elevated-temperature applications for fusion power plants. Their major advantage is good thermal properties relative to other elevated-temperature alloys. A major shortcoming is high-temperature strength, which places a limit on the maximum operating temperature. The reduced-activation steels developed for fusion applications have maximum operating temperatures of 550-600°C, but for increased efficiency of a fusion plant, designers require higher operating temperatures.

The search for new elevated-temperature steels has led to work to develop oxide dispersion-strengthened (ODS) steels, which were introduced in the 1960s. These steels, strengthened by small oxide particles, are produced by complicated and expensive mechanical-alloying, powder-metallurgy techniques, as opposed to conventional processing (melting, casting, forming—hot rolling, cold rolling, extruding, etc.) techniques used for present-day elevated-temperature steels. ODS steels are also plagued by anisotropy in mechanical properties caused by the processing.

Although successful development of ODS steels would be a significant advancement, the need to develop the manufacturing capability for the

production of large quantities of such materials would be expensive. This indicates that a more realistic solution to meet the demand for such steels would be the development of steels with higher operating temperatures that can be produced by conventional processing techniques.

Based on the science of precipitate strengthening (the need for large numbers of small precipitate particles) and thermodynamic modeling to explore possible optimum compositions, a thermomechanical treatment (TMT) was developed that increased the yield stress of commercial 9-12% Cr nitrogen-containing martensitic steels up to 88% at 700°C. Preliminary creep-rupture tests indicate a commensurate increase in rupture life. Steels designed and produced specifically for the TMT have yield stresses at 700°C up to 200% greater than conventional steels. The strength of these new steels are comparable to the strongest experimental ODS steels that have been developed. Characterization of the precipitates in the new steels by transmission electron microscopy indicated the precipitates had characteristics similar to the experimental ODS steels, and they were eight-times smaller at a number density over three orders of magnitude greater than in the conventional steels they would replace.

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02A.4

Mechanical Properties of 9Cr Martensitic Steels and ODS-FeCr Alloys After Neutron Irradiation at 325°C up to About 40 dpa

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Martensitic steels with 9 wt% Cr as well as ODS-FeCr alloys are considered as the main candidates for the internal structures of future fusion reactors. Many studies are in progress with the main objective of understanding and predicting their behaviour during service. In particular, a large programme of neutron irradiations is carried out to study the susceptibility of these materials to the irradiation-induced hardening and embrittlement at high doses and in the range of low temperatures ($T < 400^{\circ}\text{C}$) where these phenomena could reach a high detrimental level. At the present time, results are available from experiments performed in Osiris reactor at CEA-Saclay, and in SM and BOR60 reactors at RIAR, with irradiation temperatures ranging from 300 to 325°C and doses from 2 to 42 dpa.

Several ferritic/martensitic steels such as Reduced Activation alloys (F82H, JLF-1, 9Cr2W1TaV) including the European candidate EUROFER (9Cr1W1TaV), advanced ODS Fe-14%Cr ferritic alloys and 9Cr1Mo (VNB) conventional martensitic steels were irradiated as specimens for mechanical tests.

The objective of this paper is to present the main results obtained from SM and BOR60 experiments concerning the evolution of tensile properties and the irradiation creep. Tensile properties were measured at 20°C and at the irradiation temperature. Fractographic examinations of the rupture surface were performed as well. Data are compared with previous results coming from Osiris irradiation and discussed as a function of the dose for the different classes of materials.

Reduced Activation and conventional martensitic steels, which presented before irradiation similar tensile properties, behave in a different manner after irradiation. 9Cr1Mo conventional steels seem to be more sensitive to irradiation-induced hardening. The hardening rate decreases for doses higher than 10-15 dpa, but a continuous loss of ductility is observed with the dose up to 42 dpa. Parameters describing ductility could reach sometimes negligible values.

However, a very low irradiation-creep deformation was obtained at 325°C for all 9Cr reduced activation and conventional martensitic steels.

On the other hand, ODS Fe-14%Cr alloys present a moderate irradiation-induced hardening and a relatively high level of ductility after irradiation up to 40 dpa.

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02A.5

Friction Stir Welding of Oxide Dispersion Strengthened Steels

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Joining of advanced oxide dispersion strengthened first wall materials represents a major challenge because melting segregates the dispersoid. Several solutions to this problem exist, including explosive bonding, resistance welding, diffusion welding and friction stir welding. The purpose of this effort is to demonstrate that friction stir welding can be used to bond oxide dispersion strengthened steels. Friction stir welding is a solid state process that employs severe plastic deformation to create joints between wide varieties of different materials. The

weld is created by clamping the materials to be joined, and plunging a spinning tool into the surface. The spinning tool is then translated down the joint line leaving behind a weld zone characterized by a fine-grained, dynamically recrystallized microstructure. Typically, the tool is spun at 400 rpm to 2000 rpm, and translated down the joint line at a rate of 4 in/min to 300 in/min depending on tool design, base material, and thickness. As the tool rotates and translates, complex flow patterns develop in the base material that creates an intimate mixing of materials from both sides of the weld. Heat input during plastic deformation generally creates a temperature in the weld between 0.6 and 0.8 of the absolute melting temperature, so no liquid phase is generated.

It will be shown that friction stir welding can successfully create weld zones in both MA957 and ODS Eurofer. Weld zone properties will be described in detail.

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Parallel Oral Session 02B –High Heat Flux Plasma Facing Materials - I

02B.1

High Heat Flux Testing of Plasma Facing Materials and Components - Status and Perspectives for ITER Related Activities

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Extensive R&D programs have been initiated by all ITER partners to develop reliable plasma facing components for the next step fusion experiment. These activities focus on the selection and fabrication of new, improved plasma facing materials (PFM), primarily based on beryllium for the first wall and on carbon or tungsten for the divertor region. Another material related issue is the joining of these PFMs to an effective water cooled heat sink made from copper alloys and/or stainless steel. The joining techniques which have been applied include high temperature brazing, diffusion bonding, HIPping, and electron beam welding. Coatings (CVD, PVD, and 5-10 mm thick coatings applied by plasma spraying) also play an important role, particularly with regard to future repair processes after long term plasma exposure.

Existing fusion devices do not provide the conditions needed to evaluate the performance of PFMs and plasma facing components (PFC) under ITER specific thermal loads, i.e., quasi stationary heat fluxes up to approx. 20 MWm⁻² and sufficiently large cycle numbers. Hence, high heat flux test facilities based on intense electron and ion beams have been utilized successfully to assess the efficiency and the fatigue life time of different material approaches and design concepts. Modeling and experiments with both normal operation scenarios and transient events, such as plasma instabilities (vertical displacements, plasma disruptions) or edge localized modes (ELMs), are being performed to evaluate and to quantify the resulting material erosion or damage and thus to assess the life time of the components.

Additional research activities are focused on the degradation of materials and joints due to energetic neutrons. To quantify irradiation induced property changes and to evaluate the overall performance of neutron irradiated components, thermal load tests have been performed in-pile or were subject of detailed post-irradiation campaigns with actively cooled plasma facing components which have been produced using different materials and design options. Furthermore, the needs for extensive quality control methods and non-destructive analyses during the procurement phase will be highlighted.

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02B.2

Overview of Codeposition and Fuel Inventory in Castellated Divertor Structures at JET

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The castellated structure of tiles in the ITER divertor is thought to be the best solution to ensure the thermo-mechanical durability and integrity of materials under high heat flux loads, especially when considering the use of metals (tungsten and beryllium). It is known that in an environment in which low-Z elements (e.g. carbon) are also present, eroded material may be transported and co-deposited together with fuel species in areas shadowed from the direct plasma impact [1-3]. As a consequence, re-deposition occurs in grooves of castellation and in gaps separating plasma facing components (PFC).

Until recently, the only large-scale castellated structure used in fusion experiments was the Mk-I divertor of the JET tokamak. This water-cooled divertor was composed of small (5x10 cm) roof-shaped tiles separated by 6 or 10 mm gaps. It was operated first with carbon fibre composite tiles (CFC) and then with castellated (6x6 mm with 0.6 mm deep groove) beryllium blocks. After long-term plasma operation periods (~60 000 s with CFC and ~20 000 s with Be), detailed ex-situ examination of the tiles with nuclear reaction analysis (NRA) has

permitted the assessment of material transport to the gaps between tiles and grooves of the castellation. Significant co-deposition associated with fuel inventory has been observed up to a few cm deep in the gaps between tiles (up to twice greater than on plasma facing surfaces), whereas in the narrow castellated grooves the inventory was around 30 % of that found on top surfaces. Deuterium migration into the bulk of CFC tiles was also determined.

The intention of this work is to provide an overview of results obtained. Particular emphasis is on: (i) the impact of the gap width and its orientation in the torus (poloidal/toroidal) on the inventory; (ii) comparison of carbon and beryllium behaviour; (iii) the mechanisms of material transport to the gaps. The influence of PFC structure in other JET divertors on the co-deposition between the tiles will be addressed and the implications for next-step fusion devices discussed.

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02B.3

Development of Brazing Foils to Join Monocrystalline Tungsten Alloys with Eurofer Steel

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The targeted development of a helium cooled high performance divertor requires the selection and combination of refractory materials and advanced oxide-dispersion-strengthened, reduced activation ferritic steels. The joining of the different materials is challenging, as it is exposed to mechanical and thermal loading not only during the manufacturing but also during the long term operation of the divertor. Within the frame of a recently started helium gas cooled divertor for a "DEMO" thermonuclear reactor, investigations on various types of rapidly quenched brazing foils have been carried out to manufacture non-detachable tungsten – ferritic/martensitic steel EUROFER joints that are able to survive cyclic thermomechanical loading.

Based on Ti_{bal}-V-Cr-Be and Fe_{bal}-Ta-Ge-B-Pd alloys, brazing foils in the form of fragment ribbons of 30-50 μm thickness were obtained by "melt spinning"

(Forschungszentrum Karlsruhe, Germany) and "planar flow casting" (MEPhI, Russia) methods. The fabrication of small mock-ups have shown that amorphous brazing foils are most promising to join tungsten with chromium steel at temperatures between 1373-1423K. Owing to a great difference between the coefficient of linear-thermal expansion of tungsten and chromium steel, a spacer of 0.5 mm in thickness made of Ta was inserted into the construction of a brazed joint. 40×8×5 mm³ EUROFER samples and 40×1.5×5 mm³ monocrystalline tungsten samples were assembled in a special conductor and brazed in a vacuum furnace with a resistance-type heater at 1423K. Thermocycling tests of the brazed samples in a hermetic argon filled steel container have been performed – 200 cycles of heating to 1073K and cooling in water to room temperature. It has been found that the brazed joints do not contain any pores, dry joints, or cracks. The distribution of the main and alloying elements of filler material in the brazing zone is rather uniform.

Thus, it has been shown that these rapidly quenched filler metals meet initial divertor requirements of reliable «tungsten - EUROFER steel» brazed joints. Finally, results on metallography, electron microscopy, X-ray spectroscopy and differential scanning calorimetry will be shown and discussed.

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02B.4

Thermal Stability of Tungsten - Clad Low Activation Steel for Fusion Energy

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The first wall of a laser inertial fusion energy reactor must withstand the repetitive pulse of target emissions including x-rays, ions, alpha particles and neutrons. The primary candidate is tungsten-clad (100 μ m to 250 μ m thickness) low activation ferritic steel, F82H. The first few microns of the tungsten cladding absorbs the ion pulse resulting in an intense temperature peak (2400°C to 2700°C) at the tungsten surface that rapidly attenuates with time and distance into the cladding. Target values for the tungsten-steel interface temperature are around 500°C to 600°C. The durability of the tungsten cladding will ultimately depend on maintaining chemical stability and adhesion at the interface. The long

term thermal stability of the F82H alloy has been previously investigated by evaluating material from long term creep rupture and aging studies. A variety of phases were identified including a range of carbides and intermetallic compounds such as laves phase, Fe₂W.

The tungsten - F82H alloy interface represents a nonequilibrium material condition at elevated temperatures. This paper presents the results of long term aging experiments on tungsten-clad F82H steel. Diffusion couples were aged at temperatures ranging from 500°C to 900°C and times approaching 5000 hours. X-ray diffraction was used to identify interface phases. Solute concentration profiles were determined to assess the kinetics of second phase formation. The potential for using a platinum diffusion barrier to impede second phase formation and to improve adhesion was explored.

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02B.5

Effects of Solid Transmutation Elements on Defect Structure Development of W using W-Re and W-Re-Os Model Alloys

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Tungsten(W) has been considered as plasma facing materials of high heat flux components in fusion reactors because of its high melting temperature and low sputtering yield. Under fusion reactor irradiation conditions, large amount of solid transmutation products, such as rhenium(Re) and osmium(Os), will be produced by nuclear reactions by 14MeV neutrons with tungsten. These products and displacement damage may cause irradiation induced precipitates, characteristic damage structures, irradiation induced embrittlement and irradiation induced degradation of thermal conductivity. It is well known that addition of Re into W improved ductility and reduced thermal conductivity significantly even with a small amount of Re addition(i.e., 3wt%Re). Whereas, the effect of Os combined with Re have not been clarified yet.

In this work, to study the effects of transmutation products on microstructural stability of tungsten under fusion reactor conditions, a series of W-Re, W-Os and

W-Re-Os alloys were prepared and their microstructural development were examined by accelerator irradiation.

The examined alloys were prepared by arc-melting. TEM disks were cut from the ingots using Electro Discharging Machining and their surface were mechanically polished, and were annealed at 1400C for 1h in vacuum. Irradiation experiments were conducted by 1MeV-proton using a Dynamitron accelerator of Tohoku University. The irradiation temperature was about 500C and 600C, and irradiation dose was about 0.15dpa(E_d :90eV). Microstructural observation was carried out using a HF-2000 at 200eV. Irradiation hardening was measured by Vickers hardness test.

Formation of cavities was suppressed by additions of 3%Re or 3%Os. It is found that the suppression effects of Os on cavity growth was more remarkable compared to Re. In the case of W-5Re-3Os, very small visible voids were observed. It is proposed that the major reason the atomic size factor of Os larger than that of Re resulted in the prominent trapping of interstitial atoms. Irradiation induced precipitation was not observed in all the examined specimen.

Above mentioned behavior of defects clusters corresponded to the smaller radiation hardening in the binary alloys compared to tungsten. Correlation between previous neutron irradiation results and this work will be presented.

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Parallel Oral Session 03A – Fundamental Radiation Effects

03A.1

Ab initio Study of Helium in α -Fe: Dissolution, Migration and Clustering

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The atomistic behavior of helium in pure iron was investigated by *ab initio* electronic structure techniques. The calculations were performed on 128 atom supercells at constant pressure using the SIESTA DFT-code. Substitutional and interstitial configurations of He are found to have similar energetic stabilities. The tetrahedral configuration is more stable than the octahedral one by 0.18 eV for interstitial He. These results are at variance with predictions from empirical potentials which significantly overestimate the helium-to-vacancy binding-energy and give the opposite site preference for interstitial helium.

The fast migration of interstitial helium – with an energy barrier of only 0.06 eV – together with the significant binding energy between interstitial He atoms (0.45 eV) support the self-trapping effect proposed for the formation of He bubbles observed at low temperature in initially vacancy-free lattices. The effective migration energies of substitutional He are estimated for the vacancy and the dissociation mechanisms in the limiting cases dominated by thermal vacancies or high supersaturation of vacancies. The migration of substitutional helium by the vacancy mechanism is governed by the migration of the HeV₂ complex, with an energy barrier of 1.08 eV. The structure and stability of small He_nV_m clusters ($n, m=1$ to 4) were determined. The trends of the binding energies of helium and vacancies to these clusters are discussed. We show how the shift with respect to empirical potential results of the corresponding dissociation energies may impact the interpretation of thermal desorption spectroscopy experiments.

The major approximations in the calculation include: basis set, exchange-correlation functional, pseudopotential, supercell size and neglect of finite temperature contributions. The estimated effects of these approximations are critically discussed.

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03A.2

Direct Observation of Dislocation-Stacking Fault Tetrahedra Interaction

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Stacking fault tetrahedra (SFTs) are directly produced in high-energy cascades during neutron irradiation of fcc metals with low stacking fault energy, e.g., copper and gold. The high density of SFTs causes significant changes in mechanical properties such as strengthening, loss of ductility, plastic instability, and formation of cleared channels (dislocation channeling). The interaction of gliding dislocations with SFTs and the other defect clusters is a key physical mechanism for these macroscopic phenomena. The dislocation-SFT interaction is a classic topic; however, the detailed processes and their underlying physical mechanisms are still unknown. We have investigated the dislocation-SFT interaction by *in situ* straining experiments in a transmission electron microscope (TEM), focusing on the mechanism of SFT collapse.

The SFTs collapsed through direct interaction with dislocations. Although dislocation-pileups produce significant stress concentration at the pileup front, proportional to the number of dislocations consisting the pileup, some small SFTs (≈ 5 nm) did not collapse during interaction with the leading dislocation at the front of a 150-dislocation pileup. On the other hand, SFTs often collapsed during interaction with a single dislocation that was not associated with a pileup. This indicates that the stress-field is not the crucial factor for the SFT collapse. Instead, the manner of interaction with the dislocation core is most important. At least three distinct mechanisms of dislocation-SFT interaction were identified in terms of remnants following the collapse of an SFT. The occurrence of different SFT collapse processes depended on the dislocation-type (screw vs. 60 degree mixed), the position of interaction (near the apex vs. near the base triangle), and temperature (298K vs. 100K). Also, small SFTs (≈ 5 nm) were more difficult to collapse upon interaction with gliding dislocations, compared with large SFTs (> 25 nm).

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03A.3

A Magnetic Interatomic Potential for Molecular-Dynamics Simulations of Body-Centered Cubic Iron

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Body-centered cubic (bcc) iron stands out as an anomalous case among the group of bcc metals. The bcc structure of iron owes its stability to ferromagnetism and many fundamental properties of iron and iron-based alloys characterizing their microstructural evolution under irradiation are radically different from those of non-magnetic bcc metals (V, Nb, Ta, Mo, W). The mobility of self-interstitial atom defects in iron is significantly lower than in the non-magnetic bcc metals and the frequent occurrence of two types of dislocation loops in Fe is not common for other bcc metals. Density functional calculations of energies of formation of self-interstitial atom defects in Fe show that these energies follow a pattern that is different to that observed in the non-magnetic bcc metals. The origin of these anomalies is associated with the effect of magnetism on interatomic interactions, which is not included in the currently available many-body empirical potentials.

We develop a new approach to molecular dynamics simulations of bcc iron that includes the effect of magnetism on interatomic interactions. Electron correlation effects responsible for magnetism become more significant in the limit of low electron density. In iron this gives rise to a magnetic phase transition and to a symmetry-breaking branching of the energy surface. Using the Ginzburg-Landau-Stoner model of a magnetic phase transition we derive a many-body potential of interaction between atoms in bcc iron. The new potential takes into account the magnetic contribution to the total energy and is suitable for large-scale molecular dynamics simulations.

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03A.4

Molecular Dynamics Simulation of Screw and Mixed Dislocation Interaction with Stacking Fault Tetrahedron in FCC Cu

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¹Nuclear Engineering Department, University of California, Berkeley, California; ²Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; USA; ³Department of Materials Science and Engineering, University of Illinois, Urbana, Illinois, USA The interaction between moving dislocations and radiation defect clusters controls the yield strength, ductility and flow localization behavior of structural materials under irradiation. Using molecular dynamics (MD) simulations, one can directly study atomistic processes and determine the sequence-of-events controlling dislocations and obstacle interactions. Such information is crucial in understanding the relationship between the mechanical and structural properties and should be incorporated in improved physically-based models to better predict the mechanical properties under various operating conditions and to better understand the initial dislocation – defect interaction mechanisms controlling the onset of plastic flow localization in dislocation channels. To this end, we present the results of MD simulations to study the atomic-scale interactions of dislocations with stacking fault tetrahedra (SFT) in face centered cubic (FCC) metals using MD simulation methods. An examination of various factors, including dislocation type (edge, screw and mixed), temperature, SFT size, dislocation velocity and the interaction geometry lead to the conclusion that SFT are very strong obstacles to dislocation motion, with SFT shearing the most common result of dislocation interaction. Complete SFT annihilation and absorption into the dislocation core may be possible for some specific interactions. The resulting SFT – dislocation interactions are analyzed in terms of the governing partial dislocation reactions and are discussed in the context of recent experimental in-situ TEM studies at the University of Illinois and Oak Ridge National Laboratory.

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Feature of Damage Accumulation in Be₁₂Ti Intermetallic Compound

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Be₁₂Ti is a potential candidate for neutron multiplier material of fusion reactors. It was reported that no interstitial loops and no voids are formed by high energy electron beam irradiation [1], while this material showed unusually active formation of cavities under 8keV deuterium ion irradiation at room temperature at high dpa [2]. Another feature of the damage from deuterium ion irradiation was interstitial type defect clusters such as dislocation loops were not formed. It is considered that aggregation of interstitial atoms is energetically difficult even if they are formed by the displacement damage, because its crystal structure is complicated (tI26) and lattice constant is large.

In the present work, accumulation of lattice defects is calculated using a simple rate theory model for both the interstitial clustering case and the non clustering case to explain the features of deuterium irradiation damage mentioned above. Behavior of defect accumulation under neutron irradiation is also discussed by applying the rate theory.

The fact that interstitial atoms can not aggregate characterizes the defect accumulation of this material. In case of 8keV deuterium irradiation at room temperature, the mobile interstitial atoms escape to the surface and only immobile vacancies are highly accumulated in the damaged area. It is likely that the vacancies accumulated to a saturation level can aggregate and form large cavities by athermal processes such as cascade collisions. In the case of interstitial loop formation, however, the amount of the vacancies and interstitial atoms aggregated as interstitial loops are comparable and thus the evolution of their clusters may be suppressed by their mutual annihilation.

The radiation damage in the neutron multiplier of a fusion reactor is simulated by the taking parameters of low sink concentration, low damage rate and high temperature. Due to no interstitial loop formation and low preexisting sinks such as grain boundaries and dislocations, the situation where the interstitial concentration is comparable with that of vacancies continues for long times and thus it is expected that nucleation and growth of voids will be suppressed.

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Parallel Oral Session 03B – Blanket Engineering - I

03B.1

Material System Integration and Irradiation Test for Fusion Blanket

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Material developments for a blanket system of fusion reactor and evaluation of their performance changes after irradiation have been studied for a couple of decades. Based on these research activities, three types of low activation materials such as ferritic/martensitic steels, vanadium alloys and SiC/SiC composites have been developed successfully. These materials fabrication process and construction techniques for components including welding and coating have also been studying by many researchers. Recently, design activities of ITER test blanket modules (TBM) using these low activation structural materials started and design and construction of TBMs are discussing by ITER-TBM working groups to start blanket system experiments at ITER first day.

Material data of unirradiated and irradiated conditions have been collecting, but data of integrated material system behaviors of blanket component under neutron irradiation such as compatibility between structural materials and coolant or breeding materials, tritium transport between component materials are not sufficient. Life time prediction of structural materials under fusion neutron irradiation conditions are expected to be clarified using IFMIF, however, the issues of material system integration should be continued using fission reactors, because irradiation volume of IFMIF is limited.

This paper will review the structural material data from the view point of material integration for a fusion blanket system and show on-going irradiation program on material integration behavior, and discuss required irradiation experiments using fission reactors.

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03B.2

On Qualification, Codes and Standards, Quality Assurance and Licensing

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The world-wide focus of fusion activities is turning largely to activities associated with the next step burning plasmas (e.g., ITER). The design of ITER is such that the hazards addressed are similar to those of a future fusion power plant; ITER will have large inventories of tritium and activation products. As a result, nuclear safety and licensing issues are receiving increased attention. In particular requirements related to materials qualification, codes and standards, and quality assurance (QA) need to be defined for the procurement of ITER components and for the fabrication of the ITER Test Blanket Modules. In this paper, the following items will be discussed:

- philosophy behind qualification requirements
- strategies designed to accommodate the experimental nature of ITER
- additional qualification requirements for experimental components
- appropriateness of current codes and standards and areas where changes are needed
- quality assurance concerns
- recommendations/path forward

The focus of the discussion will be on materials structural performance and the impact of these items on current research activities.

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03B.3

The Feasibility of Recycling and Clearance of Active Materials from a Fusion Power Plant

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The inherent favorable safety and environmental characteristics of fusion power can be fully exploited only in a power plant design that pays careful attention to the disposition of active materials arising during operation and at end-of-life. In order to minimize –(or eliminate) the quantity of such material that remains as waste requiring long-term storage (> 100 y), maximum use should be made of both recycling within the nuclear industry and “clearance” or release to the commercial market as non-radioactive products. This approach has typically been taken in conceptual fusion power plant studies in recent years.

For the clearance of materials, i.e., the removal from regulatory control when activity has fallen to very low levels, it has been conventional to compute a Clearance Index by application of nuclide-by-nuclide clearance levels, based on various national or international guidelines. Over the past five years, the US Nuclear Regulatory Commission, European Commission, and International

Atomic Energy Agency issued revised clearance levels, taking into account the previous guidelines. In this paper the implications for fusion materials of these new levels are considered, by re-evaluation of the Clearance Index for selected power plant concepts. In addition, the issue of public acceptability of cleared material is considered, since it is not assured that formerly radioactive material would be accepted for unrestricted use, despite its extremely low level of activity.

When determining the suitability of active material for recycling within the nuclear industry, power plant studies have employed criteria based solely on radiological parameters such as contact gamma dose-rate, intended to reflect the ability to handle the materials for processing, by remote handling means if necessary. Reviews of remote procedures currently used within the nuclear industry suggest that these criteria have been unduly conservative, and this paper makes recommendations for revised criteria, and assesses the implications of their use.

The feasibility of recycling active materials from a fusion power plant depends not only on these radiological criteria, however. The possibility of waste reprocessing and isotope separation systems being available on the industrial scale, for fabrication of new components, as well as the economic viability of these processes, will ultimately determine the extent of fusion materials recycling. For key power plant structural materials, these issues are reviewed.

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03B.4

Estimation of Tritium Release Behavior from Solid Breeder Materials Under the Condition of ITER Test Blanket Module

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Tritium liberation model to represent the release behavior of bred tritium from solid breeder materials has been developed by the present authors. The model takes into account tritium diffusion in solid breeder grain, tritium transfer at interfacial zone of grain to surface water and liberation of tritium from surface water to purge gas through such surface reactions as water adsorption/desorption, isotope exchange reaction with hydrogen in purge gas, isotope exchange reaction with water vapor in purge gas and water formation reaction. Based on the model, a calculation code is composed to estimate the tritium behavior and it is named as the TRITON QUEST code-liberation from SB-. The properties required to estimate the tritium release behavior to dry purge gas, dry

purge gas with hydrogen or purge gas with water vapor have already been evaluated by analyzing data from tritium release experiments using Li_4SiO_4 (from FzK), LiAlO_2 (from JAERI), Li_2TiO_3 (from CEA) and Li_2ZrO_3 (from MAPI or JAERI) by the present authors. Tritium release curves estimated using this code give good agreement with the tritium release curves obtained from neutron irradiated solid breeder materials in experiments by the present authors for various solid breeder materials under various release conditions.

In this study, tritium release behavior from various breeder materials including world candidate breeder materials are estimated in the neutron irradiation condition and operation sequence of the ITER test blanket module test program (neutron wall loading of 0.78 MW/m^2 and repetition of burn time for 400 seconds and dwell time for 1400 seconds). Obtained results are compared and evaluated to discuss the tritium release and inventory performance of different breeder materials.

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03B.5

Material Compatibility Issues in Fusion Fuel Cycle R&D and Design

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Many material selections for fusion Fuel Cycle systems are determined by the properties of tritium, including its behavior as a hydrogen isotope, and its decay product, ³He. Within the EU R&D program, mainly oriented towards ITER, the following issues have been addressed.

Torus and Neutral Beam pumping is by cryosorption on activated charcoal, bonded to a stainless steel substrate by inorganic cement. The sorbent-bond system has been extensively tested to ensure its mechanical integrity and longevity under tritium exposure. Conditions have been established for cryopanel regeneration, to release quantitatively the diverse tritiated gas species originating predominantly from plasma wall interaction.

Processing of highly tritiated gases includes separation of the hydrogens from impurities by Pd/Ag membranes. Extended testing has been carried out to study decay helium bubble formation within the membrane lattice, with the conclusion that adequate lifetimes can be achieved. Tritium which permeates through the primary containments of tritium plant components, operating at temperatures of ~400°C and higher, is confined by outer (room temperature) jackets, which are evacuated periodically.

Water detritiation, by the CECE (Combined Electrolysis Catalytic Exchange) process, relies on wetproof catalysts with a teflon matrix, and tests are in progress to determine the operating life as a function of tritium concentration in the water. Similar tests on the nafion membranes in solid polymer electrolyzers are under way; initial results are positive. For very high tritium-in-water concentrations in other systems, materials such as hastelloy need to be used to avoid the corrosion observed with stainless steel.

Tritium storage systems, for both static and transport applications, use metal getters, which must release tritium rapidly and quantitatively at moderate conditions. Testing of the ITER reference ZrCo getter material has shown that significant disproportionation, resulting in trapping of tritium, occurs under normal ITER operating conditions. Alternative getters, including uranium, should be reconsidered.

A comprehensive study of tritiated waste quantities and types, ranging from soft housekeeping waste to metals, is under way and a strategy for treatment and disposal is being developed. For stainless steel, high temperature processes, including purging of the molten metal with argon/protium to release tritium by isotopic exchange, are being investigated.

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Poster Session 04

04-1

Fracture Toughness Vis-a-Vis the Master Curve for Some Advanced Reactor Pressure Vessel and Structural Steels

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The evolutionary concepts under consideration for advanced nuclear reactors naturally compel the need for advanced structural materials. This can be applied to advanced light-water reactors (Generation III), to the various advanced high-temperature reactors under consideration for the Generation IV reactor program, and to fusion reactors. In all of these reactors systems, ferritic and ferritic-martensitic steels will play a major role for both pressure containment components and reactor internal components. The preliminary designs for the RPVs in the Generation IV systems envision operating temperatures from 280 to 850°C, operating pressures from atmospheric to 25 MPa, and radiation doses from 0.01 to 40 dpa. The RPVs that will operate at very high pressures are also conceived to have very large vessel sizes that will require scale-up of ring forging and joining technologies and ensuring thick-section properties. In these cases, high-strength alloys can reduce sections sizes with concomitant advantages to through-thickness properties, welded construction, transportation, etc. In this study, the fracture toughness of four different structural materials of varying structural experience will be discussed: (1) a 9Cr-1MoV (Grade 91) ferritic-martensitic alloy used for high temperature structures, including pressure vessels, and approved in the ASME Code to 649°C, (2) F82H, a 7.5Cr2WV ferritic-martensitic alloy, with a smaller database than the 9Cr alloy, but good potential for higher strength and one of the alloys developed to have reduced activation under neutron irradiation with resultant advantages for decommissioning, (3) 12YWT, an oxide-dispersion-strengthened (ODS) alloy with very good high temperature strength, but a very new alloy with essentially no database, and (4) HSLA-100, a high strength, low alloy structural steel with improved toughness relative to HY-100 steel, often used for naval structures. It is shown in this paper that, in general, the master curve appears to adequately describe fracture toughness vs temperature behavior for ferritic-martensitic steels, although some steels tend to exhibit relatively high scatter outside of tolerance bounds, and that fracture toughness data in the ductile-brittle transition region are sparse, especially in thick sections. Results are also presented regarding specimens of different types and sizes. Available data on fracture toughness in the irradiated condition for all materials are also discussed.

04-2

In Situ Fatigue of the Eurofer 97 steel

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In order to investigate the fatigue behaviour of the EUROFER 97 steel in the condition of simultaneous irradiation, similar to what would occur in a thermal fusion reactor, a series of six in situ experiments were conducted using a specially designed testing device. The irradiation was carried out with 590 MeV protons, which have been shown in the past to be, at low doses, an acceptable simulation for 14 MeV neutrons. Depending on the achieved cycles to failure, the final dose reached levels between 0.15 and 0.25 dpa. All tests were performed at a total strain range of 0.8%.

The aim of the experiments was to study the synergetic effects of temperature and microstructure on fatigue performance, in particular in view of the possible embrittlement from atomic hydrogen produced by spallation reactions. It is known that hydrogen effects in steels are strongly temperature dependent and effective up to 300°C. The first test was run at 150°C and the second at 250°C. Increasing the irradiation temperature decreased the fatigue life. This is an indication of a limited role of hydrogen since hydrogen embrittlement should be more severe at low temperatures. In order to see the influence of the primary dislocation structure on crack propagation, two further tests were conducted with pre-deformation, at the same irradiation temperatures. The specimens were fatigued before irradiation, at 0.8% to about 10% of the available fatigue life and then in-beam fatigued to rupture. Again, no dramatic effects on crack propagation occurred, since the total life remained unchanged at 150°C and was longer at 250°C. Nevertheless, the in-beam part of the specimen tested at 150°C was shorter. The comparison with unirradiated experiments indicated nevertheless that life is reduced by a factor of approximately two during in-beam fatigue.

Secondary experiments were conducted for measuring the irradiation hardening under positive, zero and negative stresses, as well as for measuring the flow stress under irradiation. These experiments will be briefly reported.

The results will be discussed as a function of the observed microstructure.

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04-3

Interaction of $1/3\langle 11\bar{2}0\rangle(0001)$ and $1/3\langle 11\bar{2}0\rangle\{1\bar{1}00\}$ Edge Dislocations with Self-interstitial-atom Loops in alpha-Zirconium

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Atomic-scale computer modelling has been carried out to investigate dislocation-loop interaction in the HCP structure. The interaction of edge dislocations with the same Burgers vector (BV) $1/3\langle 11\bar{2}0\rangle$ but different glide planes, (0001) and $\{1\bar{1}00\}$, with self-interstitial loops has been considered. Such loops with perfect BV equal to one of the three $1/3\langle 11\bar{2}0\rangle$ vectors of the lattice are created in high-energy displacement cascades and are known to be a feature of irradiated zirconium. The current work focuses on the effect on dislocation glide of a

prismatic loop which is rectangular in shape and bounded by two basal- and two prism-plane segments. During motion of the dislocation line, and depending on the geometry and loop BV, it interacts with an interstitial loop by either dragging or pushing it, absorbing it or reacting to form a segment with a different BV. The reactions have been analysed by atomic visualization and dislocation theory. Stress-strain curves, and hence the critical stress for dislocation motion and breakaway, have been obtained for all the cases simulated.

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Analysis of Recovery Process of Neutron-Irradiation-Induced Defects in SiC by Isothermal Annealing up to 1400°C

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SiC is expected as a first wall and blanket material of advanced fusion reactors. It receives extremely large quantity of fast neutrons generated by a nuclear fusion reaction. Therefore, it is very important, when comprehending the safety and life of a reactor, to clarify the influence of the neutron irradiation on various physical properties of SiC. However, both the generation mechanism of irradiation-induced defects and the details of the recovery process by heat-treatment are not fully clarified. The volume expansion by fast neutron irradiation of SiC and the recovery by post-irradiation heat-treatment are deeply related to the behavior of crystalline defects such as interstitial atoms and vacancies. Therefore, it is effective to measure changes in lattice constant and macroscopic length to know concentration of defects and stability of them.

In this research, the effects of neutron irradiation on physical property change of SiC were examined. Changes of macroscopic length, thermal diffusivity, and lattice constant due to neutron irradiation and due to post-irradiation isothermal annealing up to 6 h between room temperature and 1450°C were measured.

Recovery rates at each temperature were determined and compared each other. One group of specimens was irradiated to a fluence of 5.3×10^{24} n/m² ($E > 0.1$ MeV) at 470°C, and the other group was irradiated to a fluence of 1.9×10^{23} n/m² at ~200°C.

The annealing effects on physical property change of neutron-irradiated SiC are including:

- (1) The recovery of length change and lattice constant change was not saturated by annealing for 1~2 h at each annealing temperature between irradiation temperature and ~1400°C.
- (2) Irradiation-induced defects in SiC were considered to be recovered basically by the re-combination of vacancies with interstitial atoms, since changes in both properties up to ~1200°C could be fitted almost a straight line against the square root of isothermal annealing time.
- (3) When the Arrhenius plot of recovery rates was seen in detail, there should be some recovery mechanisms in which activation energies differ up to 1200°C.
- (4) A large change of the Arrhenius plot of both samples near 1200°C was considered to correspond to vacancy migration.

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04-5

Neural Network Analysis of Charpy Toughness of Neutron Irradiated Low-activation Martensitic steels

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Previous work on the analysis of a large multi-variable mechanical property database of neutron-irradiated steels has revealed a strong correlation between a change in the Charpy impact transition temperature and the corresponding change in yield stress. However, it is likely that other variables such as the irradiation temperature and the helium (or hydrogen) content may also play significant roles. To examine this, we have used an artificial neural network to re-analyse the data. Neural networks are useful in multi-variable problems of this type, where the complexity is overwhelming from a fundamental perspective (i.e. there are no appropriate physical models) but where it is important to retain detail in order to capture subtle, possibly nonlinear, interactions between the variables. The data used to create the model span ranges of displacement damage of up to 90 dpa and temperature 273 K - 973 K. The trained Bayesian model has been able to capture the non-linear dependence of transition temperature on the chemical composition and irradiation parameters. The ability of the model to generalise on unseen data has been tested and regions within the input domain that are sparsely populated have been identified. These are the regions where future experiments could be focused. The model data has also been compared with earlier models of unirradiated data.

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04-6

Atomistic Insight into Thermal Conductivity Degradation and Swelling of Irradiated SiC

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Thermal conductivity degradation and swelling of SiC under irradiation are two key issues, which limit its application as a structural material in fusion reactors. At present, the microstructural causes of degradation in thermal conductivity and dimensional stability are not well understood or even if the same defect features are responsible for the changes in both properties. We present the results of atomistic simulations using Molecular Dynamics (M.D.) and Molecular Statics (M.S.) techniques and the empirical interatomic potentials developed by Tersoff to investigate defect evolution and impact on SiC properties under fusion relevant irradiation conditions.

The results will be discussed in the light of experimental work implying that the same defects are responsible for both conductivity degradation and swelling under neutron irradiation at temperatures below 1000°C.

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04-7

Proton Irradiation Modifications in Ultraviolet Transmission on KU1 Quartz Glass

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The optical transmission components of the future thermonuclear reactor will be expected to maintain their transmission properties under high levels of ionizing radiation (≈ 5 Gy/s) during hundreds of hours. For such applications, radiation-induced optical absorption imposes a severe limitation. It is therefore necessary to study the optical degradation of suitable candidate materials, to assess the system lifetimes. KU1 quartz glass is known to be radiation-resistant, so, KU1 pellets (approx. 5 mm diameter) samples prepared in CIEMAT, Madrid, in the frame of Romania - EURATOM co-operation, and a CARY 4 VARIAN spectrophotometer have been used. The irradiation was performed using 12.6 MeV protons at Bucharest TANDEM accelerator, in the following conditions: 0.8 mm thick KU1 samples at a total dose of 2×10^{14} protons, in air irradiation at a temperature of 50 C on the target. The presence of a 215 nm peak (due to both electron and nuclear collisions stopping) and a big reduction (as compare to a similar gamma irradiation) of the 270 nm peak (due only to nuclear collisions) were observed. Using the data from AEA Harwell FUS86 EURATOM report, the dose rate of our 12.6 MeV proton irradiations (1 nA beam intensity on a 3×3 mm² area during 15 hours) was evaluated at 200 Gy/s and the total irradiation dose at 10 MGy. Comparing our spectra (mainly the intensity of 215 nm peak) with the results for gamma and high energy electron irradiations, we can conclude for the 12.6 MeV proton irradiation at 50 C that the saturation effect in absorption is obtained after a 10 MGy dose, as compared with 4-5 MGy for gamma and with 11-12 MGy for electrons, suggesting the ionization process is essential for defect absorption centers in all the cases. Preliminary studies using high energy proton irradiations for a new type of quartz glass – KS-4V (dose and temperature dependence of absorption behavior in UltraViolet and Visible regions) as a part of the efforts related to the selection of the most resistant in various radiation fields optical transmission materials to be used in the future International Experimental Thermonuclear Reactor (ITER) project are also reported.

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04-8

Plastic Deformation of SUS304 under In-situ and Post-Irradiation Fatigue Loadings

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Structural materials in fusion reactors will receive severe displacement damage and thermal/magnetic stresses, simultaneously. Since these stresses can include cyclic component due to the periodic operation of fusion reactors, it is important to understand the radiation effects on fatigue behavior of structural materials. In the present study, plastic deformation of solution-annealed SUS304 under in-situ and post-irradiation fatigue loadings at 300°C was examined by means of the nano-indentation technique. The objective of this study is to improve the understanding of radiation effects on fatigue behavior.

Miniaturized specimens with a side notch in gauge were tested in stress control mode with triangular wave of $122.4 \leftrightarrow 24.5$ MPa under constant loading rate of 50 MPa/s. Irradiation dose rate was 1.0×10^{-7} dpa/s for both in-situ and post-irradiation tests. Nano-indentation hardness tests at notch tip were conducted for the specimen fatigued for 24 hours under irradiation (in-situ irradiation condition) as well as for the specimen irradiated for 24 hours and then fatigued for 200 hours (post-irradiation condition).

As compared with the unirradiated specimen fatigued for 24 hours, less significant change of hardness was detected even at notch tip for the in-situ irradiation specimen. As for the post-irradiation specimen, the hardened area at notch tip was smaller than that of unirradiated specimen fatigued for 200 hours. These results suggest the delay in the development of dislocation structures under both in-situ and post-irradiation fatigue loadings. The interaction of radiation-induced defect clusters and moving dislocations may play an important role in the radiation effects on fatigue behavior.

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04-9

Embrittlement Behavior of Neutron Irradiated RAFM Steels

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The objective of this work is to study the effects of neutron irradiation on the embrittlement behavior of different reduced activation ferritic/martensitic (RAFM) steels. The irradiation was carried out in the Petten High Flux Reactor (HFR) in the framework of the HFR Phase IIb (SPICE) irradiation project at a dose of 15 dpa and at different irradiation temperatures (250, 300, 350, 400, and 450°C). The embrittlement behavior and mechanical properties are investigated by instrumented Charpy-V tests with subsized specimens (KLST-type).

The emphasis is put on the investigation of irradiation induced embrittlement and hardening in the newly developed reduced activation steel EUROFER 97 for different heat treatment conditions and for HIP powder steels. The alloy is a result of the development from OPTIFER I to OPTIFER VII intensively studied in previous irradiation programs (MANITU, HFR Ib) that already showed promising embrittlement behavior after neutron irradiation up to doses of 2.4 dpa. The embrittlement behavior of EUROFER 97 is compared with the results on international reference steel F82H mod. included in the SPICE project. A role of He in a process of non hardening embrittlement is investigated in EUROFER 97 based steels, that are doped with different contents of natural boron and the separated ¹⁰B-isotope (0.008-0.112 wt.%).

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04-10

Strain Hardening and Long Range Internal Stress in the Localized Deformation of Irradiated Polycrystalline Metals

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Low temperature irradiation can harden metallic materials and often results in microscopic strain localization such as dislocation channeling and mechanical twinning, however, it does not significantly affect macroscopic strain hardening at a given true stress level. In this study it was attempted to describe the strain hardening behavior during strain localization in terms of long range back stress. Deformation microstructures show that local strain hardening can be largely affected by the orientation relationship between adjoining grains because the total plastic incompatibility, or total number of pileup dislocations, should be determined by the orientation relationship. Long range back stress was formulated as a function of total number of pileup dislocations and number of localized bands formed in a grain. Strain hardening rate was calculated for distributed and localized dislocation pileups. It was found that strain hardening rate was not sensitive to the strain localization when grain size was large; the long range back stress at a possible dislocation source is almost independent of the localization unless it is very close to the pileups at grain boundary. Also, calculation of the stress field from pileup dislocations indicated that the pileup behavior in deformation bands can be different for different dislocation types.

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04-11

Inner structure of dislocation channels in neutron-irradiated V-Cr-Ti alloys

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Dislocation channels have been observed in deformed V-4Cr-4Ti alloys irradiated with neutrons below 300°C. The local deformation due to the formation of dislocation channels gives rise to plastic instability and leads to significant loss of ductility like brittle fracture. We have reported the dislocation channeling processes after neutron irradiation by using AFM [1]. In this study we focus the microstructures in neutron-irradiated V-4Cr-4Ti alloys after mechanical tests by using TEM.

Small size tensile specimens of V-(3-5)Cr-(3-5)Ti-(0-0.1)Si alloys with gauge length of 5 mm with thickness of 0.25mm were irradiated in the HFIR-11J at 300°C with a damage level of 5dpa. After the irradiation, tensile tests were performed at RT. The UTS and uniform elongation of the neutron irradiated V-4Cr-4Ti alloys at 300°C was about 750MPa and less than 0.5%, respectively. In the tensile test, the test was interrupted at the stress level reached to ultimate tensile strength (UTS) level. TEM observations were performed for the deformed area close to the fractured part.

From the TEM observation of dislocation channels, no dislocation wall and network can be observed on the interface between the matrix and deformation band. Some deformation bands traversed in grains entirely and extended to the other grains beyond the grain boundaries. On the other hand, some deformation bands stopped at the grain boundary and there were dislocations piled up close to the grain boundary inside the deformed channel. However there were few secondary dislocation channels generated from primary dislocation channels and no intersection both primary and secondary dislocation channels through the TEM observation. It is suggested that the generation frequency of secondary dislocation channels was suppressed during deformation and lead to significant loss of ductility. We will discuss the correlation between the lack of dislocation network on the interface of dislocation channels and the suppression of the secondary dislocation channel generation in the conference.

[1] M. Sugiyama, K. Fukumoto, et al. J. Nucl. Mater. 329-333 (2004) , 467-471

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04-13

Small Punch Creep Properties of Reduced Activation Ferritic Steel

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Reduced activation ferritic/martensitic steels (RAFs) are leading candidates for structural materials of fusion reactors. One of the RAFs, 9Cr-2W-V-Ta steel (JLF-1) has an excellent high temperature creep strength. It is essential to understand an influence of irradiation on mechanical properties such as impact properties and creep properties to put the steel in practical use. However, since there are severe limitations on specimen capacity at material irradiation testing facilities, it is required to use miniaturized specimens. It would be very helpful if the creep properties could be evaluated using the SP specimen, which is much smaller than the standard specimens.

In this paper, creep properties of JFL-1 were evaluated by recently developed small punch (SP) creep test using a miniaturized specimen measuring 10x10x0.5 mm. The tests were carried out at temperatures of 600-700°C and at load levels of 150-500 N in a vacuum. The test results were correlated with those of standard uniaxial creep test in terms of the conversion of applied load to stress. Experimental results revealed that the relationship between minimum creep deflection rate and rupture life measured by the SP creep test followed the Monkman-Grant's law as well as those of uniaxial test. The rupture lives of uniaxial and SP tests were arranged against the applied stress and load using Larson-Miller parameter, respectively. Then, the ratio of load (F , N) to stress (σ , MPa) was calculated so that both curves might be in agreement. As a result, the relation was estimated to be $F=2.4\sigma$ and was consistent with that reported in conventional CrMoV casting steels.

These results suggested that the uniaxial creep properties of JFL-1 could be evaluated by a plate-type miniaturized specimen. Consequently, the SP creep testing technique seemed to be appropriate tool for evaluating creep properties of not only general heat-resistant steels but also RAFs.

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04-13

Helium Effect of Microstructural Evolution in Ion-irradiated Reduced Activation Ferritic/Martensitic Steel to High Fluences

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Mechanical property changes of Reduced Activation Ferritic/Martensitic steels (RAFTs) by neutron irradiation have been extensively studied in these decades. However, irradiation hardening and embrittlement is not clarified yet. The objective of this work is to figure out role of microstructural evolution on irradiation hardening and embrittlement with the special care on dislocation, lath/grain boundaries and precipitates.

The objective of this work is to clarify radiation-hardening mechanism by means of ion irradiation and nano indentation. JLF-1 (9Cr-2W-V, Ta) steel was

irradiated up to 60 dpa at 693 K, 743 K and 793 K. Single-ion irradiation was performed with 6.4 MeV Fe³⁺. The Fe³⁺ ions and the energy-degraded 1.0 MeV He⁺ ions were simultaneously irradiated for dual-ion irradiations. The displacement damage rate and helium injection rate were up to 1.0×10⁻³ dpa/s and 1.5×10⁻² appm He/s, respectively. As the post-irradiation examination, transmission electron microscopy (TEM) observations and nano-indentation measurements were carried out.

Small differences in micro-hardness between these irradiation temperatures were measured under single-ion irradiation. Helium effect of microstructural evolution at 693 K under dual-ion irradiation was found to contain a finer defect clusters than that under single-ion irradiation. The microstructures in the dual-ion irradiated JLF-1 consisted of the fine defect clusters like dislocation loops and precipitates, and a large increment of radiation-hardening was measured by a nano-indentation technique. In this work, helium effects on radiation-induced precipitate were investigated in detail with TEM observation. It is attempted to quantitatively relate the dislocation loops and the precipitates to the irradiation induced hardening. A discussion of the contributions of the microstructure to the radiation hardening will be presented.

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04-14

Stress Corrosion Cracking Susceptibility of Ferritic/Martensitic Steel in Super Critical Pressurized Water

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Reduced activation ferritic/martensitic (RAF/M) steels is the leading candidate as the structural materials for breeding blanket components. JAERI has proposed a ceramic breeding, water cooled, blanket concept for the ITER test blankets module and for the DEMO reactor. High temperature SCPW (super critical pressurized water) would be an attractive option from the viewpoint of higher thermal efficiency. Therefore it is necessary to check the compatibility of the steel and SCPW.

In this work, Japanese reduced activation ferritic/martensitic steel, F82H has been tested through slow strain rate tests (SSRT) with strain rates of 3×10⁻⁷ s⁻¹ in SCPW environment. The water was purified by ion-exchange resin, and oxygenated up to 2-20 wt. ppms. It was also pressurized up to 23.5 MPa and the range of its temperature was from 280 °C to 550 °C. The flow rate of the SCPW was under 6×10⁻⁷ m3s⁻¹.

The stress drop and the loss of ductility, both due to the stress corrosion cracking, have not been observed in all the specimens. Also the fracture surface showed no brittle fracture. The weight change during the SSRT depends strongly on the test temperature, but a dissolved oxygen content does not have significant effects. The time dependence of weight change has been described through the plot of some parabolic curves.

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04-15

Optimization of the EUROFER Uniaxial Diffusion Weld Process

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EUROFER steel plates with cooling channels are accepted as most important structural elements of the helium cooled blanket box (HCPB) and the breeder unit (BU) as well. The fabrication of such plates is based on a hot isostatic pressing (HIP) or a uniaxial diffusion weld (U-DW) process of two half plates structured with the half of the cooling channels, respectively. This contribution presents our results for the optimization of the U-DW process for EUROFER. The process consists of high speed dry milling of the joining surfaces, cleaning, pressing of the hot work pieces and a well known post weld heat treatment. The main goal of the optimization is a weld with mechanical properties like those of the base material by means of a process with low temperature, avoiding any change in the base material properties, and low compression, avoiding any damage of the cooling channels side walls.

For the optimization a numerical simulation tool of the U-DW process has been developed. The tool needs among others a reliable creep model for EUROFER at the diffusion welding temperatures which has been realized with a differential equation based visco-plastic model. The model parameters have been determined by fitting compression experiments. The U-DW process simulation tool yield a range of U-DW parameters like surface roughness, pressure, temperature and duration.

The paper will present in addition results of samples produced by different one step and two step U-DW processes. The samples are examined by tensile and Charpy impact tests regarding strength, toughness and ductile to brittle temperature (DBTT). New results for the weld quality and a comparison between experimental and theoretical results will be discussed. The outlook will publish first results of scalability in terms of a future application.

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04-16

Activation and Radiation Damage Behaviour of Russian Structural Materials for Fusion Reactors in the Fission and Fusion Reactors

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Various structural low (reduced) activated materials have been proposed as a candidate for the first walls-blankets of fusion reactors. One of the main problems connected with using these materials - to minimise the production of long-lived radionuclides from nuclear transmutations and to provide with good technological and functional properties. The selection of materials and their metallurgical and fabrication technologies for fusion reactor components is influenced by this factor. Accurate prediction of induced radioactivity is necessary for the development of the fusion reactor materials.

Low activated V-Ti-Cr alloys and reduced activated ferritic-martensitic steels are a leading candidate material for fusion first wall and blanket applications. At the present time a range of compositions and an impurity level are still being investigated to better understand the sensitive of various functional and activation properties to small compositional variations and impurity level.

For the two types of materials mentioned above (V-Ti-Cr alloys and 9-12% Cr f/m steels) and manufactured in Russia (Russia technologies) the analysis of induced activity, hydrogen and helium-production as well as the accumulation of such elements as C, N, O, P, S, Zn and Sn as a function of irradiation time was performed. Materials "were irradiated" by fission (BN-600, BOR-60) and fusion (Russian DEMO-C Reactor Project) typical neutron spectra with neutron fluency up to 10^{22} n/cm² and the cooling time up to 1000 years. The calculations of the transmutation of elements and the induced radioactivity were carried out using the FISPACT inventory code, and the different activation cross-section libraries like the ACDAM, FENDL-2/A and the decay data library FENDL-2/D.

It was shown that the level of impurities controls a long-term behaviour of induced activity and contact dose rate for materials. From this analysis the concentration limits of impurities were obtained. The generation of gas and solid transmutants can play a large role in changing of the properties of alloys to irradiation. Neutron-induced transmutations lead to substantial changes in elemental composition. Especially, for vanadium material the large level of solid transmutation occurs both in fission and fusion spectra. The obtained results give a complete picture about the values of induced radioactivity, dose rate, decay heat, element and gas production.

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04-17

A Closer Look at the Fracture Toughness of Ferritic/Martensitic Steels

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Ferritic/Martensitic (F/M) steels, with Chromium content between 7% and 12%, are closely investigated both in the Fusion and ADS (Accelerator-Driven Systems) communities. More specifically, Reduced-Activation F/M (RAFM) steels are now being considered as primary candidate structural materials for a demonstration fusion plant (DEMO), in view of their favourable mechanical properties and good resistance to swelling. For ADS systems, high Cr steels such as 9Cr1Mo (EM10), 9Cr1MoVNb (T91) or 12Cr1MoVW (HT9) represent the most promising choice as structural materials, in consideration of the severe service conditions (irradiation damage and production of spallation elements).

In the framework of collaborative projects with other European institutions, SCK•CEN has characterized the mechanical properties of several F/M steels, both in the unirradiated and irradiated condition: EUROFER97 (the European reference fusion RAFM steel), EM10, T91 and HT9. For all materials, in addition to tensile properties, fracture toughness has been evaluated before and after irradiation, using both Charpy impact and fracture mechanics tests. Two common, interesting features have emerged from these investigations: (a) irradiation embrittlement is systematically larger when quantified in terms of quasi-static fracture toughness (shift of Master Curve reference temperature) than when measured from Charpy tests (shift of ductile-to-brittle transition temperature, DBTT); (b) the applicability of the widely used Master Curve approach (ASTM E1921) appears questionable for these steels. Both issues, which bear obvious significant safety implications, are examined in detail and possible interpretations are attempted. In addition, potential improvements given by the application of more advanced fracture toughness analysis methodologies (SINTAP Lower Tail analysis, Single Point Estimation method, Multi-Modal Master Curve) are discussed.

Finally, in order to clarify whether the significant difference in embrittlement measured from Charpy and fracture toughness tests is due to dynamic (loading rate) effects, impact toughness tests on precracked Charpy-V specimens of EUROFER97 have been performed in the unirradiated condition and for two irradiation doses (0.35 and 1.6 dpa): the corresponding Master Curve impact reference temperature shifts have been compared with the shifts measured in terms of Charpy transition temperature and Master Curve quasi-static reference temperature.

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04-18

Mechanical Properties and Microstructure of Three Russian Ferritic/Martensitic Steels Irradiated in BN-350 to 50 dpa at 490°C

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Ferritic/martensitic (F/M) steels are widely used in various reactor facilities and are being considered as candidate for application in fusion reactors, intense neutron sources and accelerator-driven systems. The advantages of F/M steels over other materials are their relatively high resistance to void swelling, lower irradiation creep rates and often lower induced radioactivity. It is known, however, that one of the shortcomings of F/M steels is their inclination to low-temperature irradiation embrittlement. Insufficient data are currently available at all fusion-relevant temperatures and dose levels to predict the behavior of F/M alloys for fusion applications.

Traditionally, EP-450 has been used in the states of the former Soviet Union wherever F/M steels are employed. Two other Russian F/M steels, EP-823 and EI-852, were developed specially for reactor facilities employing liquid metal coolant. Compared to EP-450 these steels have silicon higher contents.

To elucidate the influence of silicon addition on microstructure and short-term mechanical properties EP-450, EP-823 and EI-852 were irradiated side-by-side in the BN-350 fast reactor. Ring specimens of the steels were irradiated in flowing sodium in a special experimental assembly to 50 dpa at 490°C. Ring-pull tensile tests were conducted at room temperature and elevated temperatures.

Measurements of mechanical properties show that EI-852 containing 1.9 wt% silicon undergoes very severe embrittlement at 490°C. Microstructural investigation reveals that formation of χ -phase precipitates on grain boundaries is the main cause of this embrittlement.

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04-19

Research and Development on the Chinese Low Activation Martensitic steel (CLAM)

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Low Activation Martensitic steels are the major candidate materials for the first wall and blanket structures for DEMO and future fusion reactors. Based on the progress of reduced activation martensitic steel research, including composition and structure, radiation embrittlement, helium effects, creep, fatigue, compatibility and their critical issues, Chinese Low Activation Martensitic steels (CLAM) have been designed to improve their composition and their performance, such as changing tungsten content to 1.5% to decrease the amount of Laves phase precipitation in weld metal and heat-affects zone at 550°C. Tantalum content increases to 0.14% to have smaller prior-austenite grain size and small spherical Ta-rich precipitate as unit cell of TaC to increase strength, as well as increasing the creep strength. Adding suitable amount of magnesium is to improve the compatibility of LiPb liquid and suitable Si is to improve the welding behavior. The performance of CLAM (9Cr1.5WVTa) has been determined: yield stress, 486.8 MPA; ultimate stress, 629.0 MPA; uniform elongation, 8.4%; total elongation 25.1%; the ductile-brittle transition temperature (DBTT_{41J}), -91°C. The tensile properties with temperature and creep performance have been determined, which are comparable to 9Cr2WVTa and EUROFER97 steels. The fracture surface of DBTT transition region has been observed and analyzed by Scanning Electron Microscope (SEM) to show the ductile-brittle transition along the fracture path. The metallurgic structure and microstructure have been studied and analyzed by Transmission Electron Microscope (TEM). The thermal physics properties, including thermal expansion; thermal conductivities and capacity are also measured and given in this paper. The data show that CLAM can be used as a leading candidate for the structural components for the Testing Modulus of ITER designed in China.

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04-20

Microstructural Inhomogeneity of Reduced-Activation Ferritic/Martensitic Steels

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In the current Japan Atomic Energy Research Institute (JAERI) – U.S. Dept. of Energy (DOE) fusion materials collaboration program, the ductile-brittle transition temperature (DBTT) of the reduced-activation ferritic/martensitic steels (RAFTs) is to be determined by the master curve (MC) method, in which the DBTT of ferritic steels is characterized in terms of a fracture toughness reference temperature, T_0 , as defined in the ASTM standard E1921. This method works when the transition fracture toughness values follow the MC, and once the value is scaled properly, the MC is usually independent of the type of steel or the type of test specimen. The problem with F82H-IEA steel, one of the RAFTs being examined in this collaboration, is that some fracture toughness values obtained by 1TCT specimens fell outside tolerance bands of the MC. There might be several reasons for the scatter and one of them would be the microstructural inhomogeneity of F82H-IEA. In this study, this possibility was investigated.

Plates examined in this study were obtained from F82H-IEA heat no. 9753, nominally Fe-7.5Cr-2W-0.15V-0.02Ta-0.1C, in wt%. The microstructures of plate numbers 2W-4 and 31W-3, obtained from the middle and the bottom-end section of the ingot, respectively, were examined. Scanning Electron Microscope (SEM) observations were performed on specimens that were etched by the Selective Potentiostatic Etching by Electrolytic Dissolution (SPEED) method, in which all precipitates, inclusions and unstable phases remain unetched. SEM observations on the fractured specimens with toughness values outside the tolerance bands of the MC were also performed. It turned out that Ta does not form MX precipitates, but instead, it forms complex $Al_2O_3 - Ta(V,Ti)O$ inclusions, or simple Ta(V)O inclusions. The complex inclusions are rather dominant in the plate obtained from the bottom of the ingot, but not in the plate from the middle of the ingot. SEM observations also revealed that broken complex inclusions tended to be observed at the crack-initiation site. These results suggest that the scatter of toughness values is corelated with this microstructural inhomogeneity, as the MC method assumes the material has a homogeneous microstructure.

Transmission Electron Microscopy (TEM) on the complex inclusion was also performed to analyze the detailed structure of the inclusions.

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Mechanical Properties of Irradiated 9Cr-2WVTa Steel With and Without Nickel

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In the past, the effect of helium on embrittlement of 9-12% Cr ferritic/martensitic steels has been studied by adding 2% Ni to the steels and irradiating in a mixed-spectrum reactor, where a two-step reaction of thermal neutrons with ^{58}Ni produces helium. Recent irradiation of nickel-doped steels below 300°C indicated that nickel-containing 9Cr steel hardened more than the same steel without nickel, casting doubt on the use of nickel-doping to study helium effects.

To determine the effect of nickel on these types of steels at temperatures above 300°C, tensile specimens of ORNL reduced-activation 9Cr-2WVTa and that steel with 2% Ni (9Cr-2WVTa-2Ni) were irradiated at 390-395°C in the Experimental Breeder Reactor (EBR-II) to 32-33 dpa. Charpy specimens of the steel were irradiated to 23-33 dpa at 376-404°C. The normalized-and-tempered steels were irradiated in two tempered conditions: 1 hr at 700°C and 1 h at 750°C. Since nickel reduces the temperature where ferrite transforms to austenite on heating to below 750°C, the 9Cr-2WVTa-2Ni steel tempered at 750°C contained untempered martensite.

In the unirradiated condition, the strengths of the two steels were similar after tempering at 700°C, but because of the untempered martensite in the 9Cr-2WVTa-2Ni, this steel was stronger after the 750°C temper. In both tempered conditions, Charpy properties of the 9Cr-2WVTa-2Ni were superior, despite the presence of untempered martensite in the steel tempered at 750°C.

After irradiation to 33 dpa, the strength of the two steels tempered at 700°C remained similar, with little indication of hardening. For the steels tempered at 750°C, there was again little change in strength of the 9Cr-2WVTa-2Ni, but the 9Cr-2WVTa showed a small strength increase. An increase in the ductile-brittle transition temperature (DBTT) was observed for both steels tempered at 700°C, with the 9Cr-2WVTa showing the largest increase. After the 750°C temper, the 9Cr-2WVTa showed no increase (a decrease was observed), but the nickel-containing steel showed a small increase, although it remained well below room temperature.

The results show none of the hardening effects attributed to nickel that were observed in experiments of nickel-containing steels irradiated at <300°C. The results have implications on how steels should be heat treated to minimize irradiation hardening effects.

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04-22

Effects of Heat Treatment and Irradiation on Mechanical Properties in F82H Steel doped with Boron and Nitrogen

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Fission neutron irradiation to steels doped with boron is frequently conducted to study effects of the helium production on microstructures and mechanical properties. The boron doping in F82H steels, however, may cause the ductility loss. Recently, we reported that co-doping with boron and nitrogen to F82H (F82H+B+N) and appropriate heat treatment improved the mechanical properties of F82H doped with boron. In order to evaluate the effects of helium atoms on mechanical properties of F82H, F82H+B+N is proposed for spectrally tailored irradiations of HFIR-15J. In this study, heat treatment effects of tempering on mechanical properties of F82H+B+N are examined and hardening behavior after irradiation is presented.

Specimens used in this study were standard F82H martensitic steels, F82H steels doped with 60 mass ppm ^{10}B and 200 ppm N (F82H+10B+N) and F82H steels doped with 60 mass ppm ^{11}B and 200 ppm N (F82H+11B+N). The specimens were firstly normalized at 1150 °C and tempered at 700 °C, secondly normalized at 1000 °C and tempered at 700, 750 and 780 °C. Tensile properties and ductile-brittle transition temperature (DBTT) were measured for the specimens. Single ion irradiations of 10.5 MeV Fe^{3+} and dual ion irradiations of 10.5 MeV Fe^{3+} and simultaneous 1.05 MeV He^+ of 10 appmHe/dpa were performed at 360 °C to 20 dpa and micro hardness was measured before/after the irradiation.

Yield stress of the F82H+10B+N steels increased about 240 MPa as tempering temperature decreasing from 780 to 700 °C and that of the F82H+11B+N changed similarly. When the F82H+10B+N was tempered at 750 and 780 °C, DBTT was about -100° C, which was close to that of F82H, but the DBTT increased about 50° C when the tempering was performed at 700° C. Radiation hardening of F82H+10B+N due to single and dual ion irradiations at 10 dpa and 10 appmHe/dpa was similar to that of F82H. Interstitial-type loops with the mean diameter of 20 nm were observed in the F82H+10B+N irradiated by single ion beam. Hardening behavior due to irradiation for the F82H+B+N was comparable with that for the F82H. Increment of radiation hardening, which caused by doping element for steels doped with boron, was not observed in the F82H+10B+N.

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04-23

Fracture toughness properties in the transition of the EUROFER97 tempered martensitic steel

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The tempered martensitic steel EUROFER97 is the European reference reduced activation steel for fusion applications. It has already been the subject of many investigations, characterization and irradiations. However, its fracture toughness properties in the transition between the ductile to the brittle regime have not been sufficiently studied and documented. This paper aims at providing a large set of fracture data obtained with compact tension specimen (0.2T and 0.4T) in the lower transition in order to precisely index the fracture toughness temperature curve and to investigate the scatter. More than 100 specimens have been tested in two different orientations, namely the LT and TL orientations. We focused our attention to five different temperatures below 170 K where the fracture toughness lies below 150 MPa $\text{m}^{1/2}$. Within this range of fracture toughness, using specimens as big as 0.4T C(T) ensures that enough constraint is maintained in the specimens ($M > 200$) so that the data may be considered as representative of small scale yielding condition. By applying the master-curve approach, it is found that T_0 is about 180 K, which is somewhat larger than previous published T_0 determined by other authors on a smaller data set. No excessive scatter has been found and all the data fall well between the upper and lower bound of the master curve. There is also no evidence of orientation effects between the TL and LT orientation on the data. Data obtained with the 0.2T C(T) specimens tested at 153 K clearly indicate that those specimens have lost constraint. A three dimensional finite element model has been developed to account for the constraint loss. The results of the 3D simulations were compared to those of the 2D simulations in plane strain conditions to assess the validity of the 2D model in the case of a fully constraint specimen. Finally, the parameters of the Weibull distribution describing the cumulative failure probability are reported for two temperatures, 153 and 173 K, for which more than 30 specimens were tested in each case.

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04-24

Effect of Heat Treatments on Tensile Properties and Microstructures of F82H Steel Irradiated by Neutrons

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The increase of yield strength and the shift of DBTT due to irradiation were reported that these were different in several martensitic steels, which had different concentrations in some elements and were also tempered at different temperatures. However, the mechanisms for the relation between the changes of yield strength and shift of DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment and impurities on them. The optimum heat treatment will be required to improve resistances to radiation hardening and embrittlement. The purpose of this study is to examine the mechanism of radiation hardening depending on tempering conditions and the recovering process of radiation hardening due to annealing after irradiation.

Heat treatments for F82H-std were firstly performed at 750°C after normalizing at 1040°C. Second normalizing for F82H-std was carried out at 1040°C, and the time of subsequent tempering at 750°C was varied from 0.5 to 10 h. The second tempering was performed at 780°C for 0.5 h. Irradiations of the specimens were performed at 150°C and 250°C in the JMTR to about 2 dpa. The heat treatment was also performed at 800, 860 and 920°C for 0.5 h and subsequently at 700°C for 10 h. The latter specimens were irradiated at 300°C in the HFIR to 5 dpa. After neutron irradiation, tensile tests of SS-3 were performed. The radiation hardening of F82H irradiated at 150°C was larger than that at 250°C and tended to increase with increasing tempering time and temperature. In the tensile tests at 400 and 500°C, the increment of yield stress in the specimens irradiated at 150°C was smaller than that at 250°C. In addition, the mechanisms of the radiation hardening depending on tempering conditions, recovering process and microstructural changes are examined.

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04-25

Comparison of radiation damage of the CLAM steel under irradiation in IFMIF and a fusion power reactor

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Low activation steel is one of the most promising candidate structural materials for fusion power plants in the future. The study on the CLAM (China Low Activation Martensitic) steel is carried out in China. In this work, radiation damages of CLAM relevant to displacement damage and gas production are evaluated and compared under the irradiation conditions of IFMIF (International Fusion Materials Irradiation Facility) and FDS-II (fusion power reactor designed by Institute for Plasma Physics of Chinese Academy). The Monte Carlo code MCNP is used for the transport calculations of FDS-II with a detailed three-dimensional (3-D) model. In order to simulate the Deuteron-Lithium neutron source, the Monte Carlo code McDeLicious, an extension to MCNP, is used for IFMIF with a detailed 3-D test cell model. The results are reported and discussed in this contribution.

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04-26

Ultra-high strength in nanocrystalline materials under shock loading

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Molecular dynamics simulations of nanocrystalline (nc) copper under shock loading show an unexpected ultra-high strength behind the shock front. The strength at high pressure can be up to twice the value at low pressure, for all grain sizes studied here (5-20 nm grains). Partial and perfect dislocations, twinning, and debris from dislocation interactions are found, while grain boundary sliding is reduced at high pressures. These simulations, together with new shock experiments on nc nickel, raise the possibility of achieving ultra-hard materials from shock loading. The implications of these results for the National Ignition Facility target design are twofold. In the beryllium "point design" for ignition, concerns about different shock transit times of the first shock through the different grains of the shell are significantly reduced by having very small grains. Secondly, the strength of nc materials could be beneficial for room temperature ignition designs that require a shell to hold over 0.7 GPa of DT gas fill.

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04-28

Implantation of D⁺ and He⁺ in Candidate Fusion First Wall Materials

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The effect of high temperature (700-1200 °C) implantation of deuterium and helium in candidate fusion first wall materials was studied in the University of Wisconsin Inertial Electrostatic Confinement (IEC) device¹. Tungsten "foam", single crystal tungsten, and a W-25%Re alloy were compared to previous tungsten powder metallurgy samples studied in the IEC device for the High Average Power Laser (HAPL) program^{2,3}. Scanning electron microscopy was performed to evaluate changes in surface morphology for various ion fluences at temperature ranges comparable to first wall temperatures. Preliminary results show that no deformations occur with deuterium implantation up to 1×10^{19} D⁺/cm² at 1200 °C polycrystalline tungsten samples. However, helium fluences in excess of 1×10^{18} He⁺/cm² show extensive pore formation at 800 °C. Helium retention profiles were evaluated using Elastic Recoil Detection (ERD) analysis with 8 MeV (4⁺) Oxygen. Initial ERD results indicate up to 40% atomic fraction within the tungsten matrix and helium saturation at fluences above 1×10^{18} He⁺/cm². These changes will have an impact on the lifetime of thin tungsten coatings on the first walls and divertors of inertial and magnetic confinement fusion reactors.

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04-29

Infrared Thermal Fatigue Test for IFE First Wall Materials

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First wall materials for laser Inertial Fusion Energy (IFE) chambers are subject to intense pulses of energy from target fuel implosions at frequencies of 5 to 10 hertz. This energy consists of x-rays, neutrons and ions that deposit in the first wall. Ions containing 30% of the total energy deposit in the first few microns of the chamber wall over a period of about 3 microseconds resulting in intense near-surface heating. This heat pulse dissipates rapidly between successive pulses. The reference first wall material for the HAPL laser IFE reactor is tungsten-clad low activation ferritic steel, F82H. In this case, the surface temperature of the tungsten can reach 2400°C to 2700°C. This cyclic heat pulse subjects the tungsten coating and tungsten-steel interface to high cycle thermal fatigue.

A pulsed plasma arc lamp has been employed to simulate the thermal fatigue condition at tungsten-steel interface. The infrared pulse amplitude was varied to achieve the desired temperature swing per cycle at the interface to emulate the laser IFE condition. Peak pulse width of 20 milliseconds and frequencies as high as 10 hertz were used. The tungsten coating thickness and nominal interface temperatures were varied. A finite element model of the infrared thermal fatigue test was developed to define the temperature profile through test specimens. Coating adhesion was assessed under thermal fatigue conditions for lifetimes approaching 10^6 cycles.

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04-30

Diffuse X-ray Scattering Measurements of Point Defects and Clusters in Iron

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An experiment has been carried out to investigate the residual damage from atomic displacement cascades in iron. Molecular dynamics simulations of primary damage formation indicate that many of the interstitials produced by these cascades are contained in small glissile clusters. Most interatomic potentials used indicate that the activation energy for cluster migration is ~ 0.1 eV, suggesting that most interstitials would be quickly lost from the material by rapid migration to and absorption by sinks. A single crystal single crystal iron sample obtained from Goodfellow Corporation was irradiated at $\sim 60^\circ\text{C}$ in the HFIR hydraulic tube to a fluence of 1×10^{23} n/m² ($E > 0.1$ MeV), or ~ 0.01 dpa. The irradiated sample was cut in half and one half was annealed for one hour at 450°C to partially recover the defect structure. Diffuse X-ray scattering measurements were then carried out at the Advanced Photon Source at the Argonne National Laboratory on three specimens: unirradiated, as-irradiated, and irradiated and annealed. The specimens were cooled to $\sim 40\text{K}$ to minimize thermal diffuse scattering. A reduction in the lattice parameter following irradiation indicates the presence of a substantial population of vacancy-type defects, with partial recovery induced by the one-hour anneal. The observed diffuse scattering is dominated by interstitial defect clusters exhibiting a tetragonal distortion consistent with a $\langle 100 \rangle$ type defect and a cluster radius of 0.65 nm. Substantial recovery of this defect component was also observed following the anneal at 450°C . The implications of these results for radiation damage evolution modeling will be discussed.

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04-31

The 1-D Reciprocating Motion of Vacancy-type Dislocation Loops in FCC Metals

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Clusters of self-interstitial atoms (SIAs) in the form of small perfect dislocation loops can be formed directly in high-energy displacement cascades in metals. Extensive studies by atomic-scale computer modeling have revealed that these defects exhibit fast thermally activated one-dimensional (1-D) reciprocating motion and contribute significantly to the evolution of irradiation damage microstructure via the so-called production bias. Molecular dynamic simulations have also suggested that vacancy loops can exhibit the 1-D reciprocating motion in bcc iron, but not in fcc copper*. This phenomenon may have an impact in cases when perfect vacancy loops are formed, but still has not been confirmed experimentally. In this paper we report *in situ* transmission electron microscopy (TEM) observation of vacancy clusters motion in deformed gold and aluminum.

In deformed gold, we observed a small dislocation loop ($\approx 2\text{nm}$), which was presumably a perfect loop, exhibited a few hundred cycles of 1-D reciprocating motion over a distance of 2nm for about four minutes at room temperature. This loop was then transformed into a stacking fault tetrahedron (SFT), which is a direct evidence of its vacancy nature. Taking into account that the stacking fault energy of gold is low and SFTs are the most stable vacancy clusters, this confirms that if perfect vacancy loops can be stabilized temporally it exhibits the

1-D thermally activated reciprocating motion. In deformed aluminum, we observed 1-D reciprocating motion of rather large perfect vacancy loops ($\approx 5\text{nm}$). And also, the loop motion was significantly enhanced by 200-keV-electron irradiation. The case in aluminum is different from gold in the following two ways: a) due to its high stacking fault energy perfect loops are rather stable, and b) due to its low threshold displacement energy 200-keV-electrons produce Frenkel pairs. We assume that the motion of perfect vacancy loops produced by deformation in aluminum is enhanced by a reaction with self-interstitial atoms. Annihilation of the self-interstitials at the loop perimeter inevitably results in releasing of its formation energy, i.e. a few electron volts per self-interstitial atom. These observations provide clear experimental evidence that small perfect dislocation loops can exhibit the 1-D reciprocating motion irrespective to their vacancy or interstitial nature, and the 1-D motion can be enhanced by reactions with freely migrating point defects.

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04-32

Void swelling behavior in electron irradiated Fe-Cr-Ni model alloys under temperature variation

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For developing materials for future nuclear applications, it is important to understand the effect of temperature variation under irradiation on defect structure evolution. Electron irradiation using high-voltage electron microscope (HVEM) yields the advantage of in-situ observation of the defect structure development, easy change of irradiation conditions such as irradiation temperature, and high damage rate (exceeds 10^{-3} dpa/s). The present study examines the effect of short pre-irradiation at lower or higher temperature on void swelling in austenitic stainless steel model alloys Fe-15Cr-xNi (x=15, 20, 25 or 30 wt.%).

The electron irradiation and in-situ observation of the defect structure were done with a JEM-ARM1250 high voltage electron microscope at Tohoku University, operating at an accelerating voltage of 1250 kV at an electron current density of about 3×10^{25} electrons $m^{-2} s^{-1}$. The typical damage level for each irradiation condition was about 6 dpa by the irradiation for 20 min.

In the constant temperature irradiation, voids were observed in the alloys of 15, 20, and 25 wt.% Ni (swelling peak was around 400°C), but in the alloy of 30 wt.% Ni. The result is in agreement with previous reports where high nickel contents suppress the void swelling.

In the temperature variation irradiation, the initial 0.6 dpa at the temperature range from 100°C to 600°C, was followed by the irradiation of 5.4 dpa at 400°C. No void was observed in Fe-15Cr-30Ni alloy by the temperature variation irradiation. In the other alloys, the effect of pre-irradiation at lower temperature was the slight increase in the void number density. For example, the pre-irradiation at 200°C increased the void density into twice that by the constant temperature irradiation at 400°C. On the other hand, the effect of pre-irradiation at higher temperature appears vice versa: the void number density was approximately an order of magnitude lower by the pre-irradiation at 500°C.

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04-33

Microstructures in F82H steel irradiated under alternating temperature

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Components of fusion reactors will undergo temperature variations during operation cycles. It has been pointed out that temperature variations during neutron irradiation may cause significant changes of microstructures through the changes of the nucleation and the growth of the point defect cluster depending on irradiation temperature. The microstructural evolution may affect the irradiation hardening, and result in the enhanced reduction of the ductility by irradiation. In order to evaluate the effects of temperature variations on the microstructural changes, irradiation experiment with changing the temperature alternately was performed.

By using an irradiation capsule with temperature control independent from reactor power (developed by JMTR operation at JAERI), a reduced-activation martensitic steel F82H was irradiated at temperatures of 250°C and 350°C. Irradiation was performed during 10 reactor operation cycles with the accumulated damage level of 1.5 dpa. For some of the specimens, temperature was changed during irradiation; for instance, temperature was elevated from 250°C to 350°C at the middle period of each reactor operation cycle.

The effect of the temperature change on the yield stress of F82H steel has been examined at 20°C after the irradiation and it was reported to be relatively large. In this study, the TEM observations of the microstructure were performed for the irradiated specimens. The size and number densities of dislocation loops were observed to be affected by changing temperature. The mechanism of the formation and growth of dislocation depending during temperature variations is discussed.

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04-34

Microstructure of Helium-implanted and Proton-irradiated T91 Ferritic/Martensitic Steel

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Ferritic-Martensitic alloys are proposed as candidate structural materials for fusion reactors, Generation IV reactors, and accelerator-driven transmutation systems. They have superior swelling resistance, improved irradiation stability and low activation compared to austenitic stainless steels. Understanding radiation effects in these alloys is critical for their success in advanced reactor and transmutation systems. In the present study, the ferritic/martensitic steel T91 was irradiated with 2.0 MeV protons to doses of 3, 7 and 10 dpa with and without pre-implanted helium at three different temperatures: 400°C, 450°C and 500°C. The He/dpa ratio was fixed at 180 appm/dpa. The irradiated microstructure consisting of dislocation loops, voids and precipitates were characterized by transmission electron microscopy (TEM). Radiation-induced segregation (RIS) of Cr and Fe along grain boundaries was examined by means of STEM-EDX. The microstructural and microchemical evolution in proton-irradiated T91, as a function of temperature, dose, and He pre-implantation will be reported and discussed.

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04-35

Hydrogen Retention Properties of Co-Deposition Layers Under High-Density Plasmas in TRIAM-1M

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Recycling and wall pumping properties are crucial issues for density control of a steady state plasma operation in thermonuclear fusion devices. In TRIAM-1M, large continuous wall pumping and temporal reduction of wall exhaust capability have been reported [1]. In our previous study, the large amount of retained hydrogen were detected in co-deposition layer formed under low density ($\bar{n}_e \sim 10^{18} \text{ m}^{-3}$) at a long pulse (4320 s) LHCD discharge in TRIAM-1M [2]. In ITER predictions, the expected density is $\sim 10^{20} \text{ m}^{-3}$ [3]. In such high density plasma, it will be expected that contribution of co-deposition to a wall pumping becomes more active. In the present work, hydrogen retention properties of co-deposition under high density plasmas in TRIAM-1M were studied.

In order to evaluate the hydrogen retention in the wall, material probe experiment was carried out in TRIAM-1M. Silicon, tungsten and pre-thinned SUS316L specimens were mounted on the probe head and inserted in the scrape-off layer (SOL) during the 186 shots of high density ($\bar{n}_e \sim 10^{19} \text{ m}^{-3}$) LHCD discharges (407 s in total) with limiter configuration. After the exposure, the quantitative analysis of deposition and hydrogen retention, and microstructure observations of specimens were carried out by means of Rutherford backscattering spectrometry (RBS) and Elastic recoil detection (ERD), and transmission electron microscopy (TEM), respectively.

Mo-deposits of various thicknesses (1~30 nm) were detected on the specimens. Mo was used for the limiter and divertor of TRIAM-1M. The deposit consisted of bcc polycrystalline with size of 10-20 nm. The Mo-deposition rate was about ten times higher than the low density discharge case [2]. While, the hydrogen concentrations (H/Mo) calculated from the hydrogen retained in the co-deposition layers was 0.04~0.15, which was much higher than bulk Mo and almost equal to the low density discharge case. Consequently, the wall pumping rate under high density discharges estimated from these results were $\sim 3.6 \times 10^{17} \text{ H/m}^2\text{s}$. The values were sufficient to explain the higher wall pumping under high density discharges. These results indicate that as long as the co-deposition layers are continuously formed, the wall pumping of TRIAM-1M is not saturated.

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04-36

The effect of Helium production on void growth in metals, during irradiation with 14MeV fusion neutrons

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This paper considers the irradiation of metals by fusion-neutrons, and treats theoretically how void growth will be affected by the helium that is produced by transmutation of nuclei. This is done within the approximation of treating the metal as an infinite crystal, neglecting the effect of impurities and grain boundaries, and taking the only effect of dislocations to be the separation of interstitials and vacancies.

Assuming that Helium is adsorbed into voids at a rate roughly proportional to the rate of increase of void volume, then the Helium pressure will on average remain approximately constant within voids, but the Laplace pressure will fall with time as the void volume increases. So in the long-time limit, voids will always become "over-pressurised", with a larger Helium pressure than Laplace pressure. This simplified picture is rigorously proven, with a proof that allows for a variety of void sizes and compositions, and void coalescence.

A bimodal void size distribution is predicted for an average void volume that is below that at which the Laplace and Helium pressures balance, but once the voids become over-pressurised, the larger voids in the distribution will grow at a lower rate, and can even initially reduce in size. Additionally, over-pressurised voids will not be able to Ostwald ripen, so in the absence of further irradiation, void growth will require either a flux of He between voids, or voids to coalesce.

Estimates predict that under fusion power-plant conditions, a bimodal distribution of voids will be expected in Austenitic steels, but that a reasonably single-sized void-size distribution is expected in weakly swelling Ferritic steels. It is suggested that the trapping of Helium within voids will slow the rate of helium embrittlement, improving the prospects for a sufficiently long-lived Ferritic steel.

The situation studied here is equivalent to forming a (stabilised) emulsion by continually adding a highly soluble and poorly soluble component to a continuous phase, and is similar to the growth of water droplets from a source of water vapour and soluble gas/particles (that can become trapped within droplets).

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04-37

Anisotropy Migration of Self-point Defects in Dislocation Stress Fields in BCC Fe and FCC Cu

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Spatial dependence of the elastic interaction energy between the self-point defects (vacancies and self interstitial atoms in stable and saddle point configurations) and the straight edge dislocations in slip systems $\langle 111 \rangle \{110\}$ and $\langle 100 \rangle \{100\}$ in Fe and $\langle 110 \rangle \{111\}$ in Cu was calculated by hybrid method. Elastic fields of dislocations and their interactions with point defects (elastic dipoles) were calculated in the framework of the anisotropic theory of elasticity. Characteristics of point defects (formation and migration energies, relaxation volumes and dipole moment tensor) in the absence of stress fields of dislocations were calculated by molecular statics method.

The most probable pathways of vacancies and SIA ($\langle 110 \rangle$ dumbbell in Fe and $\langle 100 \rangle$ dumbbell in Cu) along which the migration of the defects has the lowest energy barriers were defined in the presence of the dislocation stress fields.

These pathways lead directly to the dislocation core in case of SIA migration in the fields of $\langle 100 \rangle \{100\}$ dislocation in Fe and $\langle 110 \rangle \{111\}$ dislocation in Cu. In case of SIA migration in the fields of $\langle 111 \rangle \{110\}$ dislocation in Fe the pathways in the attraction region are parallel to the slip plane of the dislocation and do not lead to the dislocation core.

$\langle 110 \rangle$ dumbbell in Fe and $\langle 100 \rangle$ dumbbell in Cu have the lowest formation energy in the absence of stress fields of dislocations. In the neighbourhood of $\langle 111 \rangle \{110\}$ dislocation in Fe and $\langle 110 \rangle \{111\}$ dislocation in Cu the most energetically preferable SIA configuration are $\langle 111 \rangle$ dumbbell and $\langle 110 \rangle$ dumbbell correspondingly. In the neighbourhood of $\langle 100 \rangle \{100\}$ dislocation in Fe the region of stabilization of $\langle 111 \rangle$ dumbbell is absent.

Obtained results indicate severe differences in behaviour of SIA near dislocations of different Burgers vectors in materials with different crystallographic symmetry (bcc and fcc). These differences can exert influence on change of physical and mechanical properties of materials under irradiation (e.g. swelling, creep).

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04-38

Temperature Dependence of One Dimensional Motion of Interstitial Clusters in Fe and Ni

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The importance of one dimensional (1-D) motion of interstitial clusters for defect structure evolution has been well recognized in these years. Computer simulation showed that the migration energy of 1-D motion was very low, lower than that of the single interstitial when the cluster contained less than 100 interstitials in Fe and Ni. No temperature dependence is therefore expected above room temperature. Moreover, if clusters move with the frequency of lattice vibration and activation energy lower than 0.1eV, it is impossible to observe their movement using TEM. The movement has been, however, observed by many researchers. In this paper, the temperature dependence of 1-D motion of interstitial clusters and void growth in Fe and Ni was studied. We applied two criteria to detect the 1-D motion of interstitial clusters. One is the formation of interstitial type dislocation loops near the dilatational side of edge dislocations. The other is no formation of interstitial type dislocation loops near grain boundaries. They are typical defect structures formed by the 1-D motion interstitial clusters. The neutron irradiation was performed with the JMTR and the KUR.

In neutron irradiated Fe to a dose of 0.2 dpa, the 1-D motion of interstitial clusters occurred at 473 K and 623 K judging from the criteria. Nanovoids at 473 K and large voids at 623 K were observed using positron annihilation spectroscopy and TEM, respectively. A good correlation existed between the 1-D motion of interstitial clusters and void growth.

In Ni irradiated with a improved temperature control condition at 573 K, the 1-D motion of interstitial clusters existed. However the accumulation of interstitial clusters near grain boundaries, which is evidence of no 1-D motion, was observed in a conventional temperature control irradiation, in which the specimen temperature changed with a reactor power. The interstitial clusters must be formed at lower temperatures during the start-up of the reactor. This suggests to us that at temperatures lower than 573 K, say 473 K, the 1-D motion of interstitial clusters is not significant.

These results indicate that the actual long range migration of interstitial clusters is affected remarkably by impurities and stress fields near defects, which causes temperature dependence of 1-D motion above room temperature.

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04-40

Thermally Activated Transport of a Dislocation Loop within an Elastic Model

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It is usually observed that dislocation loops are nucleated in metals by irradiation with high-energy particles. The dislocation loops are composed of some self-interstitial atoms (SIAs) accumulating in a plane, that is, the dislocation loops correspond to the boundary of the SIA clusters. The dislocation loops (SIA clusters), particularly in BCC metals, have high mobility toward the closed packed directions and the one-dimensional motion contributes to the diffusion of the interstitial atoms. In the present work, we report the thermally activated transport of the dislocation loops within the framework of the conventional line tension model where the dislocation line is assumed to be a flexible string. We analytically estimate the activation energy for the thermal diffusion using a classical rate theory. According to the theory, the reaction path from a stable state to another passes through a saddle point in the phase space. The activation process is classified into two types according to the saddle point configuration of the dislocation loops. In fact, we find a transition size of the dislocation loop from point-defect-like to dislocation-like on the basis of the classification. The saddle point configuration affects not only the activation energy but also the temperature dependence of the jump frequency of the dislocation loops. If the dislocation loops are smaller than the transition size, the jump frequency is represented by the usual Arrhenius equation, $C \exp(-E/kT)$, where C is a constant. On the other hand, if the dislocation loops are larger than that, the factor C has a kind of temperature dependence.

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04-41

Defect Evolution in Silicon Carbide Irradiated with Ne and Xe Ions with energy of 2.3 MeV/u

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The understanding of defect production in silicon carbide irradiated with inert gas ions is important for the use of this material in harsh environments such as advanced nuclear reactors or in nuclear waste management technology. In the present work defect production and evolution in silicon carbide crystal irradiated with energetic heavy inert-gas ions (Ne and Xe) is studied. Specimens of mono-crystalline silicon carbide (4H polytype) were irradiated to three ion fluences ranging from 7.2×10^{14} to 6.0×10^{16} ions/cm² (to maximum damage levels of 1, 4 and 13 dpa, respectively) with Ne and Xe ions, respectively. The irradiated specimens were subsequently annealed at different temperatures up to 1273 K. Defect structure was investigated with transmission electron microscopy (TEM) using a cross-sectional preparation technique.

The microstructures of the annealed specimens Ne-ion or Xe-ion irradiated to the highest dose (13 dpa) are characterized by the formation of dislocation loops located in the basal plane in a rather wide depth range around the estimated damage peak, and gas bubbles in high density at deeper region around the the end of the estimated ion projective range. Only fine defects were observed in specimens irradiated to lower fluences. The results are in consistent with our previous work of helium-implanted silicon carbide that there is a rather high dose threshold for formation of gas bubbles [1]. The density and size of dislocation loops formed at the highest dose shows a strong dependence on dose and ion species. Larger dislocation loops with higher concentration were observed in Xe-ion irradiated specimens than in Ne-ion irradiated specimens at the same high dose level. A model concerning interaction of displacement damage with inert gas atoms was made to interpret the difference in dislocation loop structures.

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04-42

Effect of Impurities on the Reaction Kinetics of SIA Clusters and Damage Accumulation in Metals under Cascade Irradiation

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In pure metals exposed to cascade producing irradiation such as in a fusion environment, the damage accumulation at low doses and medium temperatures occurs in a spatially highly heterogeneous and segregated fashion: self-interstitial atoms (SIAs) segregate in the form of dislocation loops near dislocations or form rafts of loops while vacancies accumulate in the form of voids at very high rates in between.

During the last decade, these phenomena have been rationalized in terms of intra-cascade clustering of both vacancies and SIAs, and one-dimensional (1D) diffusion of SIA clusters. However, various features in damage accumulation, can not be understood without assuming that the 1D diffusion of SIA clusters is disturbed by changes between equivalent diffusion directions and/or diffusion transversal to the 1D direction, resulting in diffusion reaction kinetics (RK) between the 1D and 3D limiting cases. We have shown that such general RK of SIA clusters can be described by one analytical single-variable function ("master curve") interpolating between these limiting cases.

By their interaction with impurities, the diffusion of SIA clusters is reduced in rate and becomes more isotropic which results in a more homogeneous damage accumulation. We present a formal treatment of the effect of impurities on the 1D to 3D RK of SIA clusters. For estimating the interaction of SIA clusters with impurities we use elastic continuum theory. To illustrate the effect of impurities on damage accumulation via their effect on the RK of SIA clusters, we discuss the saturation void growth at high doses as a function of impurity concentration and temperature.

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04-43

Computer Simulation of Cascade Damage in Alpha-Iron

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MD simulation has been applied widely to study displacement cascades in metals. Since it is necessary to use ~1M atoms, the interatomic potentials have simple form and there may be inconsistency between different potentials for the same metal. Iron is of concern because numerous simulations have used potentials for which the <110> dumbbell interstitial is only marginally stable over the <111> crowdion, whereas recent *ab initio* calculations, e.g. Fu et al. (PRL 92 (2004)), show that the difference in energy of these defects is ~0.7eV. To investigate this, cascades have been simulated using a potential (Ackland et al., J. Phys.: Condens. Matter 16 (2004) S2629) for which the energy difference is close to the *ab initio* value. A large number (~200) of cascades of energy in the range 5-20keV have been simulated for temperature up to 600K to allow statistical treatment of the number of point defects created, the fraction of defects that form clusters during the cascade process and the geometry and other properties of these clusters. Comparisons have been made with previous results and the effects of temperature on the defect population emphasised.

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04-44

The Effects of Cascade Damages on the Dynamical Behavior of Helium Bubbles in Cu

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The understanding of effects of cascade damages, formed by high energy neutron, on fusion devices is one of the most critical issues for further nuclear fusion reactor. Furthermore the neutrons generate insoluble He due to nuclear transmutation, which make the reactor materials brittle. However there are a few reports on behavior of helium bubbles under the high energy ions irradiation, it is important to clarify the cascade effects on each bubble behavior, where no thermal effects. In the present study, it is therefore aimed to obtain the fundamental knowledge about the interaction of helium bubbles with the cascade damage by high energy self-ions by means of in-situ transmission electron microscopy (TEM).

Pre-thinned copper specimens (ϕ 3mm, 99.9999 at. % pure) were prepared by jet-polishing for TEM observation. The specimens were pre-irradiated with 10 keV-He⁺ at temperature between 573 and 773 K to introduce small helium bubbles, and then irradiated with 400 keV Cu⁺ at room temperature. To examine changes which occur rapidly, dynamical behavior of bubbles under the irradiation was monitored continuously and recorded by video system with time resolution of 1/30 sec. After irradiation, the processes of bubble motion were analysed as a function of time using individual video frames.

Although the thermal motion of helium bubbles is not expected in copper at room temperature, sporadic motions were observed under the self-ions irradiation with high energy. These motions occurred locally within a distance of several nm and the number of events was limited. As a result of the detailed analysis and comparison with simple computer simulation, it was suggested that the bubble motion was induced by the interaction with the collision sub-cascade.

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04-45

Energetic and Crystallographic Characteristics of Self-point Defects and Their Clusters of Different Geometry in Iron

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Study of mechanisms of formation and migration, energetic and crystallographic characteristics of various configurations of SIA (self interstitial atom), vacancy and their clusters was carried out by computer simulation methods for bcc Fe crystal.

The earlier proposed semi-empirical model of transition metal used for studying defects in Fe describes the ion core interactions in the framework of pair central-symmetric interaction potential and takes into account effects of electron density redistribution under crystal distortion or defect formation by introducing a volume dependent additive component into the crystal energy density. Parameters of the functional dependence of the interaction potential were determined by the iteration method during the process of adjustment of model results to experimentally known bulk properties of the crystal and characteristics of self-defects. The model results precisely meet the equilibrium elastic constants and agree with experimental measurements of the equation of state, dispersion curves, Debye frequency, Gruneisen constant and a number of other properties.

The most energetically stable configurations of clusters containing up to several tens of SIA or vacancies were determined. Their binding energies, relaxation volumes and migration characteristics were calculated. The most stable SIA clusters form prismatic dislocation loops $a/2\langle 111 \rangle$ able to slip along $\langle 111 \rangle$ directions. The formation of the edge dislocation vacancy loop $a/2\langle 111 \rangle$ was observed in the presence of more than 37 vacancies in the thin-plate cluster. Comparison with available in literature results of calculations and experimental data was made.

Structure and energy characteristics of edge ($a/2\langle 111 \rangle\{110\}$, $a/2\langle 111 \rangle\{112\}$, $a\langle 100 \rangle\{100\}$) and screw ($a/2\langle 111 \rangle$) dislocations were calculated by computer simulation methods. Obtained energy factors of the dislocations practically coincide with the results of calculations made in the framework of the anisotropic theory of elasticity. The core energies and the radii of the dislocation cores agree with calculations known from the literature.

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04-46

Kinetic Monte Carlo Studies of the Reaction Kinetics of Crystal Defects that Diffuse One-dimensionally with Occasional Transverse Migration

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The evolution of the microstructure in irradiated materials is governed by the diffusion reaction kinetics of the mobile defect species, and the reaction kinetics are crucially dependent on the dimensionality of the diffusion processes of the various defects. Energetic displacement cascades in metals produce clusters of self-interstitial defects (actually small glissile dislocation loops) that diffuse by one-dimensional (1D) glide along close packed directions and, under certain conditions, change the direction of their 1D migration to another close-packed direction. The limiting case of the increasing frequency of their direction changes is pure three-dimensional (3D) migration. The transition from 1D to 3D migration as a function of the frequency of direction changes can be described analytically and results of kinetic Monte Carlo (KMC) simulations fully confirm the analytical results. In the present paper we report on KMC simulations investigating the 1D to 3D transition in terms of a different kind of interruption of the 1D migration process: occasional migration within a plane transverse to the 1D glide direction of the defect clusters due to conservative climb by dislocation core diffusion. The transition from 1D to 3D kinetics appears to be quite different in this case. The KMC results are compared to the analytical description of this diffusion mode. In addition, we have used KMC simulations to further explore the “dimensionality” parameter space, examining the reaction kinetics when the defects diffuse under a variety of combinations of glide, climb and direction change.

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04-47

Irradiation Temperature Dependence of Self-Organized Nanostructure Generated on Au Surface Under Electron Irradiation

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In 1996, we have found a novel phenomenon in surface physics, viz. the formation of aligned nano-grooves and deep nano-holes on the exit surface of thin (001) gold foils irradiated with high doses of 360-1250 keV electrons at temperatures of about 100K [1]. The nano-grooves showed strong irradiation directional dependencies on their growth, which are along [100] and [010] for [001] irradiation, along [100] for [011] irradiation, whereas no marked grooves for [111] irradiation. The sizes of nano-grooves and holes are between about 1 and 2 nm, which are the smallest ones generated on metal surfaces so far. We have also irradiated Au(011) foils and found a surface orientational dependence on the growth of nano-grooves [2]. The final structures of the thin foils under electron irradiation are nanoparticles or nanowires. This method has been utilized to produce long gold nanowires for investigations of the interesting physics such as the electron transport properties and the multi-shell structure. Furthermore, the self-organized structure for silver, copper, nickel and iron thin foils are investigated [3-5].

Pure Au foils were used in the present study. After eliminating lattice defect on annealing, they were thinned by jet-polishing. The electron irradiations were performed in JEOL ARM1250 equipped with GATAN liquid nitrogen cooling stage. We chose grains with surface orientations near {100} and irradiated at temperatures between 95 K and 300 K mainly around [001].

In this study, we will show the temperature dependence of the nanostructure generated on Au surface under electron irradiation.

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04-48

Radiation damage in Fe-Cr alloys: computer modelling and modelling-oriented experiments

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High-chromium (9-12 wt %) ferritic/martensitic steels are candidate structural materials for first-wall and breeding blanket of future fusion reactors and accelerator driven systems. Their use for these applications requires a careful assessment of their mechanical stability under high energy neutron irradiation in aggressive environments. This assessment should rely on a satisfactory comprehension of the mechanisms leading to materials degradation, via the identification of the variables playing the most important role in determining the response of the material to irradiation. Evidence from experiments and recent computer modelling results show that the Cr concentration is a key parameter in order to determine the behaviour of ferritic/martensitic steels under irradiation, which needs to be optimised in order to guarantee the best corrosion and swelling resistance possible, together with the least embrittlement. These findings call for further experimental investigations, on Fe-Cr model alloys, aimed at complementing existing experimental evidence, against which computer simulation results should be contrasted. In this talk the research work performed at SCK•CEN, in collaboration with other European laboratories, on the problem of radiation damage in Fe-Cr alloys, both on the modelling and the experimental side, focussing on the effect of Cr concentration, is presented and the main results of the investigation are summarised. The knowledge so far accumulated and the issues that still remain to be tackled are thereby discussed.

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04-49

Monte Carlo simulations of radiation damage in hcp metals

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Radiation damage in hexagonal-close-packed (hcp) metals is different from that of the face-centred cubic (fcc) or body-centred (bcc) metals. In contrast with fcc and bcc cases, radiation damage in hcp metals varies from one hcp metal to another. The experimental study of point defect clustering in hcp metals is dominated by a consideration of the geometry of the hcp lattice and lattice parameters ratio (*c/a*). Because of this crystallographic anisotropy, defect anisotropic diffusion is expected (jump distances and jump rates depends on jump directions).

Hcp α -Zirconium is very similar to hcp α -Titanium (*c/a* 1.5873 for Ti and 1.593 for Zr and lower than ideal 1.633). It has been observed experimentally that damage produced in Zr is qualitatively similar to that in Ti. In this paper, using the input data obtained from molecular dynamics (MD) simulations on defect energetics and cascade damage, we present results obtained on irradiation of hcp α -Zirconium under different conditions with a kinetic Monte Carlo model. Using 25 keV cascades we have studied the evolution of the microstructure during irradiation under environment conditions of 600K, dose rate 10-6 dpa/s and final dose of 0.5 dpa. We have consider isotropic motion for vacancies and we have studied how the accumulation of damage is affected considering from one dimension to three dimension movement for interstitials. We present preliminary comparisons with experimental data. Finally, our previous and present results on α -Zr will be extrapolated to Titanium that has very useful applications in nuclear fusion reactors.

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04-51

Accumulation damage in Fe using kinetic MonteCarlo implemented with the last *ab-initio* parameters

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New diffusion parameters from last *ab-initio* calculations were implemented in the kinetic MonteCarlo BIGMAC code to study the differences between old parameters and those last implemented. 150 keV Fe⁺ ion irradiation in UHP-Fe were developed. Simulations were performed using the new activation energies for the diffusion of interstitial defects. Those energies implemented were 0.34 eV for SIA's, 0.42eV for di-SIA's and 0.45 eV for 3-4-5SIA's. These defects had a random 3D movement. New vacancy's migration energies for 3-4 vacancy clusters and last *ab-initio* diffusion parameters for impurities were also implemented. Results were compared to experiments carried out at CIEMAT, where TEM observations were developed to quantify the defect concentration and the defect type.

Large differences between old and new parameters and between simulations and experiments have been observed. The largest difference has been pointed out in the size of the defects, with a variation from 350 nm in experiments to 8 nm in simulations. New mechanisms have been proposed to try to explain those differences, such as new reaction probabilities and surface effects. Results including these mechanisms after inclusion in the model will be presented and discussed.

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04-52

Displacement cascades due to energetic recoils in amorphous silica using molecular dynamics simulations

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Amorphous silica is a candidate material for both final optics in inertial fusion reactors and analysis windows in magnetic fusion reactors. In operation, this material will be exposed to high neutron irradiation fluxes and it can result in point defects formation and changes in optical absorption, that is, degradation of the optical properties. In this paper we present molecular dynamics simulations of displacement cascades due to energetic recoils in amorphous silica.

We have performed statistical studies of the different types of defects produced for different energies of the primary knock-on atom (PKA). The range of PKA energies studied are from 13 eV to 10 keV, both for silicon and oxygen as the PKA. We measure how the concentration of different kinds of defects changes at different recoil energies and we catalogue the defect types depending on its final potential energy and its morphology, calculating the coordination and neighbor type.

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04-53

Molecular Dynamics Simulation of the Hardening of Fe by Irradiation Induced Defects

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Irradiation induced hardening of ferritic/martensitic materials envisaged for the future fusion reactor is hardly understood. In effect, transmission electron microscopy observations cannot account for sufficient defects to explain hardening using the classical dispersed barrier hardening model. In addition, He, produced in large quantities by the fusion neutrons of 14 MeV, is known to impact mechanical properties, but its effect at the microstructural level is still unclear. Molecular dynamics simulations of the interaction of a dislocation with defects in Fe were performed, with edge and screw dislocations. Various empirical potentials for Fe and He were tested. While early empirical potentials (e.g. Ackland et al. 1997) for Fe predict a strongly degenerated screw dislocation core, in the form of a three-fold symmetry core, dissociated onto {110} planes, ab-initio calculations show a weaker degeneration, in the form of a six-fold dissociated core. A recent empirical potential (Mendelev et al. 2003) reproducing this configuration was used. Our results show that the migration of the screw dislocation is only weakly affected by the difference in dislocation core configuration.

The defects of interest are glissile $\frac{1}{2} a_0 \langle 111 \rangle$ and sessile $a_0 \langle 100 \rangle$ dislocation loops, and cavities in the form of voids and He bubbles. Results show that voids and sessile dislocation loops present a similar and strong obstacle strength to edge dislocations. He bubbles present a lower obstacle strength than voids. Screw dislocations are only weakly affected by the interaction with such defects, when considering a direct migration in the twinning direction. However, their mobility is strongly affected by the defects when considering their migration by kink propagation.

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04-54

A Dislocation Dynamics Model for the Effects of Irradiation in the Brittle - Ductile Transition of F82H

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Discrete dislocation simulations have successfully been recently used to simulate crack-tip plasticity and the consequent brittle-ductile transition (BDT) in ferritic steels. The simulated crack system involves a micro-crack in the plastic zone of a macro-crack (elasto-plastic stress field). Crack tip plastic zones are represented as arrays of discrete dislocations emitted from crack-tip sources and equilibrated against lattice friction. The effects of blunting at the macro-crack-tip on modifying the crack-tip field are included. In addition, emitted dislocation arrays shield the elastic field of the crack, and the resulting field describes the elasto-plastic stress distribution around the crack. In this work, we build on our earlier model of the BDT by explicitly including the dependence of the yield stress on the irradiation fluence of F82H steels for fusion energy applications. The BDT curves are obtained by simulating the fracture toughness at various yield stresses and temperatures, and are compared with experiments. The experimentally observed shape of the BDT curve and the shift in the ductile-brittle transition temperature (DBTT) with irradiation dose are recovered. The effects of microstructure will also be presented. Blunting of the macrocrack as a result of dislocation emission is found to significantly affect the fracture toughness at and near the transition temperature. It is shown that increasing dislocation mobility at higher temperatures as a result of a decrease in the friction (or yield) stress enhance crack tip blunting and lead to a rapid increase in the fracture toughness in the transition regime.

04-55

Mechanisms of mobility of single-interstitial and interstitial clusters in Fe-Cr alloys: a computer simulation study

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In order to understand and rationalise the experimentally known behaviour under irradiation of both Fe-Cr alloys and high-Cr ferritic/martensitic steels versus Cr concentration, in particular concerning swelling, it is essential to understand how the presence of Cr may influence the mechanisms and rate of diffusion of point-defect and point-defect clusters in Fe-based alloys. In this work, the mobility of single-interstitial and interstitial clusters in Fe-Cr alloys of different concentrations is studied by molecular dynamics, using an Fe-Cr potential recently fitted to treat at best both the stability of interstitial configurations and the thermodynamic features of Fe-Cr alloys in the range of interest for application (up to 12%at Cr). It is observed that interstitial mobility changes in a non-monotonous way, depending on Cr concentration, in a similar way to how swelling rate in Fe-Cr alloys depends on Cr concentration. The diffusivity parameters deduced from the molecular dynamics simulations are then introduced in a simplified way into an object kinetic Monte Carlo code in order to evaluate the impact of the change of interstitial cluster mobility with Cr concentration on the evolution of radiation damage produced by cascade accumulation.

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04-56

Atomistic View of Dislocation – Obstacle Interactions in Ni via MD Simulations

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Interactions of edge and screw dislocations with various types of radiation obstacles, including nanometer voids, faulted ($a/3\langle 111 \rangle$) loops and He-bubbles have been studied in Ni by molecular dynamics simulations, using the MDCASK code and embedded atom method interatomic potentials. The simulations have been performed for a variety of temperatures and interaction geometries. The results provide insight into the atomic-level sequence of events controlling radiation strengthening and the conditions leading to plastic flow localization in the form of dislocation channels. As well, quantitative information about dislocation core structure modifications, the critical stress and bypass angle at the moment of dislocation detachment are determined. The results of the atomistic simulations are compared with available experimental data.

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04-57

Simulations of Elastic Electron Diffuse Scattering from Small Dislocation Loops

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We have recently shown that elastic diffuse scattering patterns obtained from single small dislocation loops carry information both on the morphology and nature of the loop (see Kirk *et al*, Phil. Mag. 85, 497, 2005). Potentially this is an important new method for characterising small clusters.

We have developed both kinematical and dynamical models of elastic electron diffuse scattering in order to calculate the distribution of diffuse scattered electrons in reciprocal space from small dislocation loops and other point defect clusters. Simulations were carried out for similar conditions used in the experiments of Kirk *et al* to investigate the influence of experimental parameters such as the type of loop, its size and its orientation and depth in the foil, the deviation parameter, and the foil thickness.

Both dynamical and kinematical models gave reasonably good agreement with the experimental results. Differences between the two methods and their relative advantages will be discussed. A database built from simulated results for different loop types is useful for optimizing the experiments.

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04-58

Lattice kinetic Monte-Carlo modelling of helium cluster formation in ferritic steel

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Irradiation of ferritic-martensitic steels in fusion environment is accompanied with accumulation of noticeable amount of helium. Helium concentrations exceeding hundreds of appm induce pronounced helium clustering at elevated temperatures. Moreover, small angle neutron scattering indicates formation of a high density of very small helium clusters invisible in transmission electron microscope. Clustering of helium is known to be of potential danger for mechanical properties of irradiated steels. Thus investigation of the details of helium clustering at high temperatures is important for the prediction of He effect on the evolution of mechanical properties of ferritic steels in fusion devices.

Clustering of helium atoms is driven by diffusion and at fusion related temperatures it occurs at time scales of the order of seconds and more. For this reason the most adequate technique for atomistic simulation of helium clustering is kinetic Monte-Carlo. Lattice kinetic Monte-Carlo (LKMC) approach was used to investigate the decomposition of an initially uniform solid solution of helium atoms into an ensemble of He clusters for different initial He concentrations and sample temperatures. Simulations are performed in the framework of two complimentary models: (a) The “fast” one, where He atoms are allowed to exchange their positions with neighboring iron atoms simulating actual diffusion jumps, and (b) The vacancy diffusion model, where the vacancies are explicitly included in the system. The resulting configurations are used for calculation of SANS signals, which are then compared with the experimental SANS results.

Our simulation results confirm that at high initial helium concentrations the decomposition of gas solid solution in the bulk leads to the formation of an array of very small He clusters. On the other hand, particular distributions of clusters over sizes depend very much on the external conditions (He concentration, temperature). Moreover, there is a systematic difference between the results of the “fast” and “vacancy diffusion” approaches, due to the effects of high mobility of small He-vacancy clusters.

04-59

Point Defect Interactions in Fe-Cr Alloys

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The progress of fusion technology as a viable future energy option necessitates the need for advanced high-performance materials for first wall and blanket structures. Fe-Cr alloys are a leading candidate material for operation up to about 550°C. Complete understanding of the response of Fe-Cr alloys to irradiation in high-temperature fusion environments requires knowledge point defect and point defect cluster interactions with Cr solute atoms and transmutant impurities, most notably He and H. In this work, we have used two different Finnis-Sinclair type potentials for Fe-Cr alloys, which predict different interactions between Cr and self-interstitial defects. The potentials for the pure elements were obtained from the literature and the two different Fe-Cr cross potentials were fit to experimental data on the mixing enthalpy and lattice constant of Fe-Cr alloys. Here we present the results of atomistic molecular dynamics and kinetic Monte Carlo simulations to investigate the point defect behavior in Fe-Cr alloys, as a function of Cr content and the character of Cr interactions with self-interstitial defects. The modeling results are compared to isochronal annealing recovery experiments performed by Maury and co-workers (F. Maury, P. Lucasson, A. Lucasson, F. Faudot and J. Bigot, *J. Phys. F: Met. Phys.* 17 (1987) 1143.) The KMC simulations were performed for similar conditions as the Maury work, namely electron irradiation of Fe-Cr alloys of varying concentration, followed by isochronal annealings. The input to the KMC simulations including the defect structure obtained by molecular dynamic simulations of 60 and 100 eV displacement cascades and the migration behavior of self-interstitials and small self-interstitial clusters, as a function of Cr content and Fe-Cr potential. The results provide insight into the effect of Cr atoms on self-interstitial cluster behavior and indicate which Fe-Cr cross-potential is more accurate for describing self-interstitial atom cluster – Cr interactions.

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04-60

PKA Energy Spectra and Primary Damage identification in Amorphous Silica under different neutron energy spectra

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Amorphous Silica is a key component in windows of heating systems in Magnetic Fusion and final focusing of lasers in Inertial Fusion. A significant irradiation from neutrons, charge particles and X-rays can appear in those components.

The energy spectra and fluxes will be updated for different magnetic (ITER, DEMO) and inertial (HYLIFE-II, SOMBRERO) fusion systems/conceptual reactors. Significant moderation is obtained through the liquid protection in the case of HYLIFE-II, and calculations will be performed using different materials such as PbLi and Flibe. The spectra and fluxes of other neutron sources actually used (HFR-Petten, BOR-60) or planned (IFMIF) to study radiation damage processes in this material will be considered also.

Primary Knock-on Atoms (PKA) energy spectra will be obtained using SPECTER code for Silica for each one of those neutron spectra. From those data a systematic analysis of primary damage will be obtained using TRIM and MARLOWE codes for high energy recoils, in order to get distribution of cascades and subcascades for different recoil energies.

A final conclusion of this work will be to describe the structure of damage to be included in Molecular Dynamics ulterior simulations, using MDCASK with Feuston-Garofalini interatomic potential, to get due account of the high-energy recoils, including spatial distribution of defects. The identification of defects will be discussed on the light of parallel work under development.

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04-62

Cracking at elevated temperature in CuCrZr alloys during Electron Beam Welding

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Copper alloys such as CuCrZr are extensively used in current fusion machines as heat sinks of actively cooled Plasma Facing Components (PFC) designed for steady state power exhaust. The precipitation hardened copper material has been selected for its good thermomechanical properties at high temperature associated with the possibility of industrial assembling by Electron Beam Welding (EBW). However, repeated occurrence of micro cracks in the EBW which may propagate and then reduce lifetime was observed, consequently, the qualification of a homogeneous crack-free EBW of CuCrZr alloys is a key issue and motivates further investigations on the assembling process.

For this, five batches of CuCrZr alloys with different manufacturing processes and chemical compositions were characterized. Preliminary welding tests were carried out on samples to identify the failure sensitivity to EBW parameters. Afterwards, EDX analysis was performed on cracked samples showing the presence of fine CuZr₂ chains precipitated along the grain boundaries. In addition, a dedicated experimental apparatus with a numerical camera has been set-up both to observe cracking initiation and to measure the local thermo mechanical behavior of the sample during EBW. The first EBW images pointed out crack initiation located near the liquidus limit while EBW tests and local thermal parameters were collected in order to model the crack propagation mechanism.

Experimental work operated in this study allowed a first description of the crack propagation mechanism with the help of metallurgical analysis, thermal and mechanical experimental results and EBW parameters effect. The final objective of this investigation will be the definition in 2007 of a "Weldability criterion of CuCrZr" via a simple commissioning test for a given welding geometry.

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The New Electron Beam Test Facility JUDITH-2 for Investigations on Plasma Facing Components – Design and First Operational Experience

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For more than 12 years, the electron beam facility JUDITH has been successfully used for high heat flux testing. Experiments performed so far cover the simulation of normal operational conditions (thermal fatigue) on actively cooled samples and abnormal events (disruptions, VDEs) on different plasma facing materials. Due to its location in a hot cell, neutron irradiated and unirradiated, as well as toxic (beryllium) samples can be tested.

In order to extend the testing capacity as well as the variety of testing techniques (medium scale mock-ups, simulation of ELMs), a new machine (named JUDITH-2) has been built.

Nominal power of JUDITH-2 is 200 kW, compared to 60 kW of JUDITH-1.

Due to a very flexible and individually programmable electron beam sweeping system, a very homogeneous surface heat distribution can be generated over an area of 0.5 x 1.0 m². Rather realistic simulations of the ITER relevant transient loads are achieved at power densities up to 10 GW/m² and event times from microseconds up to a few seconds. Furthermore it is possible to generate cyclic loading conditions with superimposed transient loads in one single experiment.

According to the relatively low acceleration voltage between 30 and 60 kV, the penetration depth of the electrons is rather low (<3 μm in W and <25 μm in CFC and Be). This reduces volumetric heating and achieves more plasma relevant surface loads.

A new digital IR-camera offers fast image grabbling and high resolution combined with a powerful software which offers a lot of new evaluation methods.

Especially brittle destruction will be addressed, using a spectrometer in the visible range, a photodiode array and acoustic emission. The first two methods serve to analyse the emitted particles while acoustic emission gives information on the onset of brittle destruction.

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Application of lock-in technique to CFC armoured plasma facing components inspection

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The Plasma Facing Components (PFC) commissioning is a key issue for actively cooled fusion machines. In particular, the thermal behaviour of the commissioned PFC must be assessed in order to guarantee the integrity of the components during its lifetime. For that, CEA/DRFC developed in Cadarache a qualification methodology based on Non Destructive Techniques (NDT) and High Heat Flux testing (HHF), the output of this methodology being an acceptance criteria. Due to the technological complexity of actively cooled PFC it is necessary to cross-check different inspection methods in order to increase the reliability of this acceptance criteria.

Therefore, a new inspection technique –so called lock-in – has been set-up in CEA/DRFC. This technique is based on an external sinusoidal thermal excitation generated by a set of modulated flash-lamps. The phase-shifting of the inspected component thermal response - which is recorded with the help of an infrared device - depends on the thermal diffusivity along the heat path, thus on the presence of cracks or failure into the component.

The lock-in technique was operated on CFC (Sepcarb® NB31) joined to Copper samples by “Direct joining” process from Politecnico de Torino [1], this paper deals with the experimental setting-up of the lock-in technique including a study on sensitive parameters and associated uncertainties. Finite element thermal calculations are performed to check quantitatively the CFC thermal properties and estimate the flaws at the CFC/Copper interface.

[1] Joining of C/C to Copper for ITER divertor, to be published in Proc. SOFT 23, Sept. 2004, Venezia

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04-65

Operational Conditions in a W-Clad Tokamak

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Economic fusion power production will critically depend on the ability to develop plasma-facing components (PFCs) which offer plasma compatibility, low erosion, controllable tritium inventory, thermo-mechanical stability, good heat transfer properties and stability under neutron irradiation. PFCs in present day devices are mostly designed to optimise fusion performance discarding the technical needs of a future fusion power plant. Even the next step device, ITER, takes a conservative ansatz using beryllium for the main chamber PFCs in order to minimise the risk of strong power loss through impurity radiation in the central plasma. However, in a reactor Be will not be a viable solution due to its high erosion yield and high-Z components may have to be used.

Experiments with tungsten PFCs are performed in the ASDEX Upgrade divertor tokamak and the fraction of W-PFCs has been increased steadily since 1999 reaching a fraction of 70% during the 2004/2005 campaign. Until 2007 a complete coverage with W PFCs is envisaged. The technical solution chosen, are W coatings on graphite and CFC. The different components exhibit different power loads and erosion yields. This is taken into account by different thicknesses in the W-coating produced either by physical vapor deposition (PVD) or vacuum plasma spray (VPS). Power loads in excess of 15 MW/m² can be handled in this way. Although the use of coatings will not be directly transferable to the power handling components of a reactor, it reduces considerably the efforts to be undertaken when transforming a low-Z into a high-Z device. A similar technique is envisaged to be used in JET when converting to a W divertor. Moreover, similar W-coatings - although on steel – would have sufficient erosion lifetime and may be used in reactor as main chamber PFCs.

The experiments on ASDEX Upgrade indicate that plasma operation is feasible with walls and divertor surfaces mostly covered with tungsten, but also reveal critical issues: Fast particles from plasma heating can play a crucial role in W erosion and central particle transport must be kept high enough to overcome impurity accumulation.

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04-66

Accumulation of Helium in Tungsten Irradiated by Helium and Neutron

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Plasma-facing and high heat flux materials in a fusion reactor suffer two types of damage: displacement damage caused mainly by high-energy neutrons and surface damage, such as erosion, sputtering and blistering, caused by hydrogen and helium plasma. Usually, these two types of damage are investigated separately. In the present study, cooperative effects of helium plasma and neutron irradiation on microstructural evolution in tungsten were investigated using computer simulations based on a rate theory.

Both damages produced by neutron irradiation and helium plasma with 10 keV and the effect of helium induced by plasma were considered simultaneously in the present simulations. In contrast to the uniform damage produced by neutron irradiation, the damage and the distribution of helium had a rectangular shape with its center at 15 nm from the plasma facing surface and a width of 10 nm. In order to elucidate the main defect reactions dominating the microstructural evolution, several assumptions were made as follows: (1) only helium atom, interstitials and vacancies were mobile; (2) maximal number of helium atoms absorbed by a vacancy was six; (3) thermal dissociation was only considered in helium-interstitial clusters. The tungsten matrix with thickness of 0.067 mm was divided into fifty parts in the present simulations, where concentration of helium, point defects and their clusters were uniform in each part, and numerical solutions of differential equations in each part were carried out simultaneously using Gear method.

Helium diffusion in tungsten 0.067 mm thick only needed 0.01 s to reach saturation at 873 K. The concentration of helium-vacancy clusters of the type 6He-V was uniform in the matrix and increased up to 10⁻⁶ even after 1 s of irradiation. The formation of helium-vacancy clusters, such as 6He-V, was mainly determined by the vacancy concentration, which depended on the irradiation time and temperature, defect production rate, and helium concentration. The helium concentration depended on helium diffusion during the initial stage of irradiation, and subsequently, on the emission of helium by the interaction of interstitials and helium-vacancy clusters.

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04-67

Behavior of Deuterium in Boron Films Covered by Oxygen-containing Layer

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Boron is an attractive candidate of the first wall coating materials in fusion reactors. The behavior of deuterium implanted into boron films has been studied. This paper studies the effect of thin oxygen-containing boron layer on the behavior of deuterium implanted into boron film by X-ray Photoelectron Spectroscopy (XPS) and Thermal Desorption Spectroscopy (TDS).

Three kinds of samples were used in the experiment. Boron films (sample 1[#]) used as contrast were deposited by PCVD with B₁₀H₁₄+He, with a thickness of 150 nm, and oxygen of 0.6 %, carbon of 0.6 %. Sample 2[#] was deposited by PCVD with B₁₀H₁₄+He+O₂ after prepared by the same processes of sample 1[#]. The thickness of the oxygen-containing layer was about 10 nm. The average oxygen concentration within the layer was 1.6 % and carbon of 1.5 %. Sample 3[#] was prepared by oxidation of Sample 1[#] in O₂/He plasma. The thickness of the oxidized layer was about 2 nm, in which B₂O₃ was formed. The average oxygen concentration within the thickness of 2 nm was 6.5 % and carbon, 1.7 %. After degassed at 993 K for 10min, the samples were irradiated by D₂⁺ ion with an energy of 3 keV at a flux of 1×10¹⁸ D⁺ s⁻¹ m⁻² up to a fluence of 1×10²² D⁺ m⁻² at room temperature.

After the D₂⁺ implantation, the peak energy of B1s in the near surface and bulk of all the samples were shifted to higher energy side about 0.3 eV due to deuterium trapping; the oxygen concentration in the oxidized layers of the sample 2[#], 3[#] obviously decreased. In the TDS of all samples, there were three desorption peaks at about 400, 580 and 760 K corresponding to desorption processes for adsorption on the surface, deuterium bound to boron as B-D-B and B-D bond, respectively. The deuterium retention in the sample 2[#], 3[#] decreased by 14% and 16% compared with the sample 1[#]. These results imply that the oxygen is preferentially sputtered by deuterium ions due to chemical reaction between deuterium and oxygen during the D₂⁺ implantation.

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04-68

Silicon Doped Carbon/Cu Joints Based on Amorphous Alloy Brazing for First Wall Application

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The limitation on using silver-based alloys for brazing in-vessel components of fusion experimental reactor created a development problem for new brazing materials. Amorphous ribbon-type filler metals represent a promising selection for joining heterogeneous materials together the advantage results from the homogeneity of elements and phase compositions and the strictly specified geometrical dimensions of such fillers.

In this paper, rapidly solidified ribbon-type Ti based amorphous filler with a melting temperature of 850°C and a thickness up to 20 μm was used to join silicon doped carbon to copper. According to finite element analysis, very thin Mo foil and Cu foil were selected as middle layer to mitigate the thermal stress between carbon and copper. SEM examinations demonstrated the high quality of brazed joints. The brazed seam has a uniform structure along its entire length. The shear strength test shown that the shear strength of this carbon-copper joint is more than 25Mpa, and the rupture was mainly occurred on the carbon side. The thermal shock resistance is tested and the prospects of these joints for fusion reactor applications are discussed.

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04-69

Properties of co-deposits on graphite high heat flux components

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High heat flux components in most of present-day tokamaks are made with graphite or/and carbon fibre composites. The major drawback of carbon is its reactivity with hydrogen isotopes leading eventually to significant fuel inventory in co-deposited layers. However, excellent power handling capabilities and non-melting of carbon make it a candidate material for target plates in the strike point region in a next-step machine, i.e. ITER. Therefore, as long as carbon is considered for future applications as plasma facing material all aspects of its behaviour in presently operating controlled fusion devices must be studied in detail.

The objective of this work was to examine morphology (structure and composition including the fuel content) and properties of co-deposited flaking films and dust from the TEXTOR tokamak. Hydrogenated films formed on the toroidal belt pump limiter (ALT-II), on the neutralizer plates of that limiter and on the rf antenna grill were studied using a set of surface analysis techniques.

The essential results may be summarized by the following: (i) deuterium content determined with nuclear reaction analysis is up to 12 atomic % in the deposition zone on the ALT-II plates and only 0.5% in brittle and dust forming deposits on the neutralizers; (ii) the structure of deposits shows significant diversity dependent on the location, stratified and columnar forms have been observed; (iii) distribution of co-deposited plasma impurity species is non-uniform; (iv) mechanical properties of deposits depend on the substrate on which they were formed. The results will be discussed in terms of process governing the material erosion and migration. Their impact on the material lifetime will also be addressed.

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04-70

Comparison of Erosion Processes of RAF and Pure Fe by Hydrogen and Carbon Mixed Ion Beam Irradiation

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Accurate estimation of sputtering erosion for low activation materials and studying feasibility to use them as PFM's are important issues in fusion reactor development. Since several materials may be used for PFM's (Be, W, and C for ITER), materials mixing effects on sputtering erosion are very important. In this study, hydrogen and carbon mixed ion beam were irradiated to RAF and pure Fe to study carbon impurity effects on sputtering. The ion beam consists of 1keV H₃⁺ (dominant species), H₂⁺, and H⁺. Carbon impurity of 0.05% to about 0.8% was deliberately introduced into the ion beam to examine the carbon impurity effects on erosion.

For RAF, sputtering erosion by hydrogen and carbon (~0.8%) mixed ion beam increased by about 20% as temperatures went over 700 K, which is attributable to the decrease in carbon concentration at the surface at elevated temperatures due to carbon diffusion and chemical sputtering. When the surface was partly covered by carbon atoms, sputtering erosion of RAF was reduced. For pure Fe, however, sputtering erosion significantly decreased as temperatures went over 700 K. The erosion yield of pure Fe at 873 K was only 20-30% of that at 423 K. At temperatures over 700 K, recrystallization of pure Fe was observed and the erosion depth of each grain showed quite a large difference. This recrystallization phenomena and dependence of erosion yield on crystal structures could be related to the decrease in sputtering erosion for pure Fe at elevated temperatures. In addition, for low carbon concentration in hydrogen ion beam (C: 0.05%), this decrease in the sputtering erosion was not observed. These results indicated that a small amount of carbon impurity in hydrogen ion beams has some effects on sputtering erosion, which was not observed in the case for RAF.

04-72

In-reactor Creep-Fatigue Tests of a CuCrZr Alloy at About 333K

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At present it seems almost certain that the precipitation hardened CuCrZr alloy will be used both in the first wall and divertor components of ITER. In the reactor vessel, this alloy will be exposed to a flux of 14 MeV neutrons and will experience thermomechanical stresses as a result of the cyclic nature of the plasma burn operations expected in ITER. This kind of cyclic loading would induce not only fatigue damage but also may make the material creep during the extended hold periods foreseen in the plasma burn cycle. To our knowledge, nothing is known about the impact of this kind of complicated mode of deformation on the mechanical performance and lifetime of metals and alloys in the environment on intense neutron irradiation. The limited amount of experimental results of creep-fatigue experiments carried out on unirradiated CuCrZr alloy (and in the absence of neutron irradiation) have shown, on the other hand, that hold times are damaging and shorten the number of cycles to failure.

In order to throw some light on this problem we have initiated a programme with the objective of carrying out creep-fatigue experiments directly in the environment of neutrons in a fission reactor at Mol (Belgium). A test module appropriate for carrying out low cycle fatigue tests with fully reversible (i.e. R = -1) tension-compression cycle with the provision of implementing a holdtime of 100 seconds both in the tension and compression sides of the cycle was designed. A number of such modules have been fabricated, calibrated and tested in air as well as in water. During experiments the strain will be measured and controlled by LVDT sensors and temperature will be measured by thermocouples. These tests will be carried out in the strain controlled mode. The first series of experiments are expected to be carried out at about 333K. Reference tests on specimens identical to the ones to be used in the in-reactor tests and using the same test modules that will be used for the in-reactor tests have already been carried out. The in-reactor creep-fatigue tests are scheduled to be started on 19 April 2005 and are expected to continue for four weeks. The results of the reference tests on the unirradiated specimens and of the in-reactor creep-fatigue tests will be presented and discussed.

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04-73

Production and Characterization of Titanium Beryllides for HIDOBE Irradiation

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Preliminary investigations revealed that beryllides like Be₁₂Ti may be much more suitable for neutron multipliers in future fusion power plants as pure beryllium, as these intermetallics promise faster tritium release, much smaller swelling and less reactivity with stainless steel. A high dose irradiation programme has been started at HFR Petten which is aimed to reach 6000 appm He (30% of the expected DEMO end-of-life) at blanket relevant temperatures in various Beryllium products and in some titanium based beryllides. However, the lack of knowledge on methods for a reliable production of beryllides still exists.

In this study two processes of fabrication of Be₁₂Ti samples, corresponding to atomic ratio Be-7.7at.%Ti, have been carried out by cold pressing of powders followed by arc-melting in oxygen-free atmosphere. One process is based on Be and Ti powders having relatively coarse particles (Be – 150-200 µm, Ti – <44 µm). The second process provides on the basis of a wet milling process a powder mixture of Be and Ti particles ranging from 1-3 µm. According to the requirements of the HIDOBE rigs, cylindrical samples with 3 millimetre diameter and about three millimetre height have been cut from the arc melted ingots. X-Ray, scanning and transmission electron microscopy measurements revealed that the vast majority of the dense cylinders consist of Be₁₂Ti phase. In addition, a few samples of Be-3.3at.%Ti composition have been fabricated to achieve a two-phase structure (α-Be + Be₁₂Ti) which might have improved plastic properties. Besides the microstructural results, instrumented compression tests and tritium release experiments will be presented and discussed.

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04-74

Progress of Research on Plasma Facing Materials in USTB

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On ICFRM-9(1999), we have made a presentation titled "Development of Functionally Graded Plasma facing materials" (published in J. Nuclear Materials, 283-287(2000) 1116-1120). Since then, we have made some progress in research on plasma facing materials in the following aspect.

FGMs have been designed with the plasma erosion resistant materials (SiC, B₄C, SiC-B₄C, W) facing plasma and high heat conductive materials facing active cooling medium. Analysis of residual thermal stress in FGM was conducted with

finite element method. The composite distribution exponent, the thickness of surface layers and the numbers of FGM layers have been optimized through minimization of maximum residual stresses.

Several fabrication methods have been developed to fabricate FGM. Taking W/Cu FGM as example. Apart from coating W/Cu FGM with plasma spray, process of Cu infiltration into porous W skeleton, SPS process, a newly developed method named resistance sintering under ultra-high pressure (RSUHP) have been developed in RCFM, this RSUHP process can give strong bonding not only on W/Cu, but also for those material systems of constituents with great differences in melting point and sintering temperature.

Besides the above-mentioned process, graded joining of W-Cu and C-Cu with Ti based amorphous filler has been achieved.

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04-75

Effects of microstructure on exfoliation and blistering behavior of various W by He implantation at about 550C

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Blistering and exfoliation have been studied to evaluate surface erosion behavior of plasma facing components of fusion reactors. The recent assessment of energetic particle deposition to the wall in DEMO reactor design suggested that the lost particle flux to the wall could be as high as $2 \times 10^{18} \text{ m}^{-2} \text{ s}^{-1}$ for alpha-particles. The fluence during the operation cycle can reach about 10^{26} m^{-2} , a few orders magnitude larger than the critical fluence for exfoliation. The energy spectrum of the alpha particles has distributions near the 3.5MeV and also below 100keV. We have been studying the blistering and exfoliation behavior by He-ion bombardment with such energy spectrum at around 550C. From the view point of material development of tungsten as a plasma facing material, microstructural control by fabrication technique has been applied to suppress irradiation or recrystallization embrittlement of W. Grain size control by K dope, La oxide dispersion or Carbide particle dispersion W by mechanical alloying(MA) have been studying by many researchers. The purpose of this study is to examine blistering and exfoliation behavior and evaluate resistance to exfoliation of these tungsten samples.

Irradiation experiments were performed using powder metallurgical processed various Tungsten samples. K dope W and La oxide dispersion W fabricated by powder metallurgical process, TiC dispersion W processed by MA, and pure W by standard powder metallurgical process were investigated. Recrystallized or stress released specimen were examined. The implanted specimen was polished mechanically with diamond paste and electro-polished to obtain flat and smooth surfaces. 3MeV He ion beam irradiation was carried out by a Dynamitron accelerator of Tohoku University with/without a rotating energy degrader system in order to study the effect of implanted He distribution. The irradiation temperature was controlled at around 550C. Surface morphology change after the irradiation was examined using a SEM and a confocal laser microscope.

The critical fluence of exfoliation of recrystallized specimens at 550C due to 3MeV He ion without energy degrader was about 2 to $3 \times 10^{22} \text{ He/m}^2$ and the critical fluence of exfoliation of stress released specimen tended to be larger than that of the recrystallized ones. Exfoliation expanded through grain boundaries on the irradiated surface. The quantitative analysis of the irradiated area and comparison to that of tungsten specimens will be presented also.

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04-76

Distribution of hydrogen isotope retained in the divertor tiles used in JT-60U

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It is one of the critical safety issues to evaluate tritium inventory in the plasma facing materials of a fusion reactor. We have been investigating the retention properties of hydrogen and deuterium in the plasma facing divertor tiles used in JT-60 and JT-60U. In this study, we try to evaluate the total retention of all hydrogen isotopes in the divertor area of JT-60U with both side pumping geometry by thermal desorption spectroscopy (TDS). The analyzed tiles were taken not only from the plasma-facing surface but also from the shadow area from the plasma of the divertor tiles used in JT-60U.

The most of the plasma facing surfaces of the inner divertor tiles were covered by thick deposited layers with different thicknesses. The deposition was also found on the plasma facing surface and the bottom of the outer dome wing tile. All other divertor areas were covered by little deposition or even eroded. The hydrogen retention in the deposited layers varied from 2 to $16.5 \times 10^{22}/\text{m}^2$. Hydrogen concentration in the deposited layers depends on the position of the divertor area and clearly affected the frequency of strike-point-hits, or history of temperature rise. The largest retention of hydrogen isotopes, 1.65×10^{23} atoms/ m^2 , was observed on the bottom of the outer wing tile facing to the pumping slot where the plasma did not hit directly and was covered by the deposition layers of $\sim 35 \mu\text{m}$. Assuming homogeneous retention in the re-deposited layers and the density of the deposition layers to be 0.91 g/cm^3 , the hydrogen concentration in this re-deposited layer was approximately 0.07. (H+D)/C ratio and those in the deposition layers on other areas was less than around 0.03.

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04-77

Analysis on Damage to TF Coils of A Compact Reversed Shear Tokamak CREST

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The compact reversed shear tokamak CREST is a conceptual tokamak reactor design with high β plasma, high thermal efficiency, competitive cost and water-cooled ferritic steel components. Some of its parameters are similar to those of the ITER advanced mode plasma. A lot of work has been done on CREST design such as for the design and optimization of the core plasma parameters, estimation on cost of electricity (COE), hydrodynamics and safety etc. In this manuscript, the specific neutronics issues and analysis on damage to TF coils of CREST were carried out based on the three-dimensional (3D) model of the CREST with the worldwide used code MCNP/4C and the IAEA latest released Fusion Evaluated Nuclear Data Library FENDL/2.0. Damage to some specific regions of the TF coils are calculated and analyzed. These regions locate at the mid-plane region or near the large openings such as the neutral beam injecting (NBI) port and the divertor cassette, and they will face and stand strong neutron irradiation for being at the mid-plane or near the large openings. Distributions of nuclear heat density, fast neutron flux ($E > 0.1 \text{ MeV}$), dose rate to insulator, peak DPA (displacement per atom) to Cu etc. for the TF coils of CREST at these regions were done and analyzed. The shield thicknesses at these regions are optimized. The results were compared with the criterions of the ITER case to make sure that they are under the limitations and that the TF coils are well shielded and the device is safe during operation.

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04-78

Experimental and numerical analyses on LiSO_4 Pebble beds used in a ITER Test Module Blanket

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This paper deals with a research activity on the breeding blankets of nuclear fusion reactors. In particular, an experimental set up for the determination of the granular material conductivity in presence of an interstitial pressurized gas is described. This research is performed in the frame of the studies connected with the ITER plant. The ITER operation phase foresees to test Breeding Blanket Modules (Test Blanket Module, TBM). One of the possible configurations of TBM, that will be tested, has the neutron multiplier and the breeder made up of pebble bed. The knowledge of the effective conductivity of the pebble bed versus the temperature and the bed deformation is of fundamental importance for designing and optimizing these types of TBMs. The experimental set uses the so called 'guarded hot plate' method for the conductivity determination. The tests were performed with a simultaneous compression of the bed (uniaxial oedometric deformation) which permitted to obtain the effective bed conductivity versus the axial deformation at different values of temperature. The effective conductivity of LiSiO_4 and Li_2TiO_3 pebble bed were determined considering several packing factors and several pressures of interstitial gas.

Moreover, a theoretical model and a discrete FEM model to simulate the thermal-mechanical behaviour of the ceramic pebble bed are being developed by the authors. The results of the theoretical models have been compared with the experimental ones, obtaining a good agreement in terms of bed conductivity and stiffness.

04-79

Characteristics of surface water on Li_4SiO_4

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It has been considered that bred tritium in a solid breeder blanket can be recovered in the chemical form of HT when purge gas with hydrogen is used. However, it has been pointed out by the present authors that no small amount of bred tritium is released in the chemical form of HTO when hydrogen is added to the blanket purge gas because of the occurrence of the competitive reaction including adsorption/desorption reaction, isotope exchange reaction with hydrogen, isotope exchange reaction with water vapor and water formation reaction on the surface of grains of a solid breeder material. For this reason, it is required to establish the way to predict the chemical form of bred tritium released to the purge gas for the design of a reliable recovery system from the solid breeder blanket. It has been also shown by the present authors that the amount of water on the grain surface plays an important role in decision of the chemical form of bred tritium.

The way to quantify the amount of surface water and the water generation capacity of solid breeder materials at various conditions was discussed in this study. The amount of water from a Li_4SiO_4 (made by FzK) packed bed to the purge gas was measured using the thermal release method where the temperature of the sample bed was changed linearly from room temperature to 1073K with the rising rate of 5K/min. Although the water release curve to dry purge gas from the Li_4SiO_4 sample stored in the room atmosphere gave 4 peaks at room temperature (second largest peak), 400K, 500K (largest peak) and 673K, respectively, the release curve to dry purge gas mixed with hydrogen gave the additional fifth peak at 900K. The water generation capacity estimated from this work was compared to that estimated previously using tritium by the present authors. In addition, the temperature dependence of the isotope exchange capacity estimated from the integration of the water release curve in this study gave good agreement with the empirical equation that the present authors reported elsewhere.

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04-80

Pre-irradiation characterization of beryllium for High Fluence Irradiation

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A high fluence irradiation of beryllium in the High Flux Reactor in Petten will be performed in the frame of the European Programme for the development of the Helium Cooled Pebble Bed (HCPB). The objective of the irradiation programme is to assess the irradiation behavior of beryllium and beryllium pebbles with respect to swelling (dimensional stability) and tritium retention. The thermo-mechanical behavior of beryllium pebble beds under irradiation, hence the interaction of swelling and creep is also investigated. The test-matrix include a large variety of beryllium grades and pebbles to study effects of microstructure, chemical composition and geometrical aspects (bulk to surface ratio, density) on tritium release.

This paper is focused on the pre-irradiation characterization of the beryllium grades and pebbles that are irradiated in the High Fluence Irradiation in the HFR Petten. The pebble size distribution of each pebble bed and pebble stack has been determined in order to study the interaction of swelling and creep at the irradiation temperatures. Moreover, the measurement of the size-distribution enables modeling of small size pebble beds using discrete element models. The size of the pebble beds is too small to be fully representative for large pebble beds and to support the development of continuum models. Furthermore, thermal diffusivity of beryllide and beryllium discs have been measured using the laser flash technique. The (immersion) density of all grades has been determined to enable PIE swelling measurements.

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04-81

Use of Refractory Metal Alloys in Permeator and Heat Exchanger Applications in Dual-Coolant PbLi Breeding Blankets

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The dual coolant lead-lithium (DCLL) blanket concept is under active investigation in the U.S. because it potentially offers much higher performance than designs based on solid breeding blankets. Helium cooled ferritic steel serves as the structural material in the DCLL concept. The breeder is self-cooled lead-lithium eutectic operating at temperatures up to 700°C. Silicon carbide flow channel inserts thermally and electrically insulate the ferritic steel from the lead-lithium since the ferritic steel is not compatible with lead-lithium at 700°C. A critical feasibility issue for the DCLL blanket concept is identifying suitable materials for the tritium extraction and heat exchanger portions of the power conversion system. Group V refractory metal alloys are being considered as potential candidates for such applications because of their high tritium permeability, good high temperature mechanical properties, and anticipated compatibility with lead-lithium at 700°C. A study was performed to determine the limiting operational conditions for successful application of refractory metals in the DCLL environment. The stress limits to avoid creep deformation, the rate of reaction with gaseous impurities and tolerable levels of such impurities to avoid serious mechanical property degradation, and general compatibility with liquid metals were all considered in the evaluation.

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Experimental Determination of Creep Properties of Beryllium Irradiated to Relevant Fusion Power Reactor Doses

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Beryllium has been selected for the Helium Cooled Pebble Bed blanket (HCPB) in the European fusion technology long term program. Beryllium acts as a neutron multiplier that will allow tritium production in the lithium ceramic breeder. Before using the HCPB concept in a power reactor (e.g. DEMO) it has to be fully qualify and tested in experimental programs and in fusion reactor (e.g. ITER).

Tritium production and retention is a potential safety and waste issue. Another important issue is the dimensional stability of the HCPB. Indeed, due to helium produced by irradiation and migration into bubble, swelling will occurs. It will induce large compressive stresses in the pebble bed, undesirable loads on structural components and will modify heat transfer coefficient. Thermal creep can play an important role to reduce and redistribute stresses in the pebble bed. Most of the creep data available in the literature has been produced in the sixties and are not relevant to end of life of a fusion power reactor. Therefore, an important experimental program has been executed.

A dead weight machine has developed to produce creep experimental results on beryllium irradiated to $4.6 \cdot 10^{22}$ n/cm². Due to the external compressive load the material creep and the specimen shrink. However, the specimen also swells due to the combine effect of internal pressure in the helium bubble and creep. One of the major challenges is to unmask swelling and derive intrinsic creep properties. This has been achieved through adequate modeling oriented experiments. Creep has been measured and its temperature dependence characterized.

In addition, experimental results are supported by a dedicated code ANFIBE aiming at describing swelling, helium and tritium production, gas precipitation, migration and re-dissolution.

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Investigation of Phase Transition in Li₂TiO₃ by High-Temperature X-ray Diffraction

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So far, various Li containing oxides have been proposed as candidate materials for tritium breeder for thermonuclear fusion reactor. Among them, Li₂TiO₃ has been expected as most promising because it has good tritium release property. Since the tritium breeder is used under severe conditions such as high temperatures and reductive atmospheres, information on thermal and mechanical stability of employed materials at high temperatures, phase transition for example, is essential for application. One of the authors proposed existence of phase transition in Li₂TiO₃ at around 700 K with measurement of thermal conductivity. However, existence of the phase transition has not been definite due to lack of analysis of crystal structure at high temperatures. In this study, crystal structure and thermal expansion property of Li₂TiO₃ have been investigated by using high temperature X-ray diffraction.

Li₂TiO₃ polycrystalline powder was prepared with solid state reaction method. Nominal composition of Li₂CO₃ and TiO₂ powder was mixed in ethanol with alumina mortar, followed by pressing into pellets. The pellets were heated at 1273 K for 8 h in air. The X-ray diffraction measurement revealed that the prepared Li₂TiO₃ was single phase with monoclinic structure with $a=504.5$ pm, $b=878.4$ pm, $c=973.5$ pm and $\beta=99.87^\circ$. X-ray diffraction measurement at high temperatures from 303 K to 923 K was carried out in air by using RINT-2500. (CuK α : 50 kV, 250 mA: Rigaku Co., Ltd.) The lattice constants at high temperatures were calculated by using calibrated Bragg angles of diffraction peaks. The calibration angle was estimated by comparing Bragg angles of the peaks at 303 K for high temperature X-ray diffraction apparatus and those obtained by room temperature X-ray diffraction apparatus.

For 303-923 K, the crystal structure of Li₂TiO₃ maintained monoclinic with increase of a -, b - and c -axis and decrease of β with increase of temperature, indicating that absence of the first order phase transition. In temperature dependence of molar volume calculated from lattice constants, however, slight variation of volume thermal expansion coefficient was observed at around 700 K, suggesting existence of higher-order phase transition. These results showed agreement with thermal conduction and dilatometric measurements.

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04-84

Experiments on Eurofer steel corrosion by Pb-17Li

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Corrosion samples of the EUROFER 97 steel were exposed to Pb-16Li in the LIFUS II loop at 480°C and 550°C, with a liquid metal velocity of about 0,01 m/s in agreement with the foreseen operative conditions of the HCLL (Helium Cooled Lithium Lead) blanket concept.

The specimens were extracted after 1500, 3000 and 4500 hours exposure at the lower temperature, while a further time step of 6000 hours was added at the higher temperature.

After extraction, weight change measurements and metallurgical analysis were performed on the corrosion specimens. The experimental results demonstrated a linear corrosion mechanism, together with no preferential elemental depletion of the steel in contact with the liquid alloy.

In this paper the results are reported and discussed with reference to previous similar works.

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04-86

Beryllium Interactions in Molten Flibe

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Molten Flibe (2LiF·BeF₂) is a candidate as a cooling and tritium breeding media for future fusion power plants. Neutron interactions with this salt will produce tritium and release excess free fluorine ions. Beryllium metal has been demonstrated as an effective REDOX control agent to prevent the free fluorine from reacting with structural metal components. The extent and rate of beryllium solubility in a pot design experiment to suppress continuously supplied hydrogen fluoride gas has been measured and modeled. This paper presents evidence of observations and measurements of beryllium metal in the salt from post test examinations. Potential influences of bi-metal exposures, some with direct coupling, e.g., beryllium to nickel and beryllium to iron are examined. The impact of the bi-metal contacts upon Be dissolution in the Flibe and the potential alloying with the transition metals are examined using scanning electron microscopy, Auger electron spectroscopy and x-ray photoelectron spectroscopy.

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04-87

Study on reaction of titanium Beryllide with water vapor

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Beryllium is one of the candidate materials of the neutron multiplier in the tritium-breeding blanket, which would be placed in the high neutron flux and high temperature environment. Thus, there are some problems such as the compatibility with structural materials, the tritium inventory and the reactivity of beryllium with water vapor and oxygen. Titanium beryllides such as Be₁₂Ti are known to have advantages over beryllium from the perspectives of higher melting point, lower chemical reactivity, lower swelling and so forth. Thus, these materials are thought to become promising alternatives of beryllium. However,

few experimental data related to the subjects described above are available for these materials. Therefore, in this work, the authors investigated the reaction of titanium beryllides with water vapor. In the experiments, the sample disks of Be₁₂Ti were exposed to an argon gas with 10,000 ppm of water vapor, and the sample temperature was raised up to 1000 °C. However, the chaotic breakaway reaction, which is known to take place on the surface of beryllium, was not observed. The analysis of the result reveals that the amount of water, which reacts with Be₁₂Ti, is much smaller than the case of beryllium. Surface analysis of the samples was conducted by means of SEM, XRD and ESCA, which suggest that thin oxide layers were present on the surface of the samples exposed to water vapor. The kinetics of oxidation on the surface of Be₁₂Ti by water vapor was investigated using a model differential equation, and the reaction constant was quantified. Furthermore, to know the electron state in Be₁₂Ti, ab-initio calculations of quantum chemistry were performed using CRYSTAL 98. In the presentation, more details of these subjects will be discussed.

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04-88

Effect Of Neutron Irradiation On Radiation Hardening And Electrical Resistivity Of A Number Of Refractory Metals And Alloys

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Refractory alloys are currently the most promising materials for application in PFC in such units as the ITER divertor and limiter, which, when in operation, will experience high thermomechanical loads and intensive neutron irradiation. Yet, the information on the radiation lifetime of refractory materials is rather limited.

Under neutron irradiation the thermal conductivity of refractory alloys is reduced. A drop in the thermal conductivity results in an increase in thermal stresses on PFC materials, therefore there is the need to have data on the effect of irradiation dose on a change in thermal conductivity of these materials.

This paper presents the results of the investigation into the effect the low-temperature neutron irradiation has on the mechanical properties and electric conductivity of a number of refractory materials.

Samples of pure Mo and Nb and Ta-4W, W-27Re alloys were irradiated at $T_{irr}=80^{\circ}\text{C}$ in the SM-2 reactor in the dose range of $5 \cdot 10^{-4} - 5 \cdot 10^{-2}$ dpa. The samples were tested for tension, and a change in the electric conductivity of irradiated materials was measured.

The dose dependencies of radiation hardening of refractory alloys show that the hardening rate is maximal in W-27Re alloy (~ 400 MPa at a dose of $5 \cdot 10^{-2}$ dpa). Ta-4W alloys and pure Mo demonstrate a hardening of ~ 300 MPa. The radiation hardening is the lowest in pure Nb (~ 120 MPa at the maximal dose).

The dose dependencies of a gain in the electrical resistivity of irradiated metals and alloys were constructed. This gain correlates with radiation hardening, i.e. it is maximal for W-27Re alloy and minimal for pure Nb.

On the basis of the data obtained, the authors calculated a change, when under irradiation, in the thermal conductivity and thermal strength of refractory alloys. It is shown that due to a high thermal conductivity pure Mo offers considerable advantage over other materials, as far as thermal strength is concerned.

The contribution of radiation defect clusters and transmutation processes to hardening and gain in electric resistivity has been analyzed. The transmutation processes are shown to play an essential role in changes in the properties of W-27Re alloy.

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04-89

Molybdenum-Rhenium Alloys for High Heat Flux and High Neutron Flux Applications

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Various refractory alloys, including Mo-Re alloys have been suggested for use in high heat flux applications in fusion reactors because of their high melting point, high thermal conductivity, and high-temperature mechanical properties. Molybdenum alloys with rhenium contents of 41% to 47.5% (wt%), in particular, have good ductility and strength at high temperatures. However, irradiation-induced embrittlement is one of the key issues for these alloys, and could ultimately determine their suitability in fusion reactor applications. Embrittlement may be driven by radiation-induced transmutation, segregation, and/or precipitation of the brittle sigma and chi phases. The formation of Os (via transmutation of Re) could produce other precipitates which may also enhance embrittlement. This paper reviews the current database for irradiation effects in Mo-Re alloys, with particular attention given to the influence of radiation-induced precipitation on mechanical properties. Results from testing of Mo-Re alloys within the last 2-3 years will also be assessed and the potential for Mo-Re alloys in fusion reactors will be evaluated.

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04-90

Evaluation of the mechanical properties of W and W-1%La₂O₃ in view of divertor applications

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Within an estimated operating temperature window of 800 °C to 1200 °C pure tungsten and W-1%La₂O₃ (WL10) are currently envisaged for use as structural materials in recent DEMO divertor designs. Here the upper limit is mainly defined by recrystallization and loss of strength while the lower temperature is limited due to embrittlement. For a qualified assessment of these materials' applicability, long-term creep and brittle-to-ductile transition data – determined by standard tests – would be necessary. But up to now such data have been unavailable.

Therefore, creep rupture (up to 3000 hours) and standard impact tests have been performed with specimens fabricated from commercial tungsten and WL10 rods. The specimens were oriented parallel to rod axis for optimum mechanical properties and all tests have been accompanied by detailed microstructure analysis with regard to recrystallization and fracture mechanisms. Even so, ductile-to-brittle transition temperatures of just 800±50 °C for tungsten and approximately 950±50 °C for WL10 have been determined. After 3000 hours at 1300 °C both materials show also relatively low creep rupture strength (40 MPa for W and about 70 MPa for WL10). While lower strength values could possibly be compensated by according design modifications, embrittlement certainly can not. Furthermore, under fusion specific neutron irradiation, an according alloy should exhibit transition temperatures of 200-400 °C, at least, if utilized for divertor components. Hence, the impact of these results on the use of dispersion strengthened tungsten for divertor structures is discussed and alternative materials as well as possible routes of alloy development are outlined.

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04-91

Effect of In-Cascade Defect Cluster Formation on Radiation Hardening of Molybdenum

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The purpose of this study is to understand in-cascade defect cluster formation and their influence on the hardening and deformation behavior of molybdenum (Mo) under fast neutron irradiations at low temperature. Low carbon arc cast (LCAC) molybdenum was irradiated in the hydraulic tube facility of the high flux isotope reactor (HFIR) at the reactor coolant temperature (~80°C) to five nominal fluences ranging from 0.0002 to 2×10^{25} n/m² (E > 0.1 MeV). Tensile tests and hardness measurements were performed to provide experimental evidence of radiation hardening caused by in-cascade defect clusters. Electrical resistivity measurements before and after irradiation combined with electron microscopy examinations were carried out to determine the size, density and structure of defect clusters as a function of irradiation dose. Radiation hardening models are applied to explain the experimental observations and provide insight into the role of immobile defect clusters produced directly in displacement cascade in irradiation-induced hardening and deformation behavior. Body centered cubic (bcc) iron (α -Fe) is used as a reference and is compared with molybdenum to show the similarities and differences in in-cascade clustering and hardening behavior between the two metals that have the same crystal structure but different atomic mass. The experimental results are also compared with published molecular dynamics simulations to understand the fundamental aspects of defect production and accumulation in displacement cascades in bcc metals.

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04-92

Anodic polarization properties of V-Cr-Ti type alloys for fusion applications

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Vanadium-based alloys nominally containing 4wt% Cr and 4wt% Ti have been identified as the reference material for fusion reactor applications. One of the main issues of using V-based alloys at elevated temperatures is their high affinity with gaseous elements. It is necessary to improve oxidation and corrosion resistance of V-based alloys, in order to use them in various environments, such as vacuum leak and reaction with water. It is important to evaluate the corrosion mechanism of V-based alloys for improvement of corrosion resistance. In this paper, V-based high Cr and Ti alloys were prepared and corrosion tests by electrical method were conducted to evaluate the effect of Cr or Ti content on the anodic polarization properties of V-based alloys.

V-(4, 7, 10, 12, 15, 20)Cr-(4, 10, 15)Ti alloy were prepared. Electrical corrosion tests with linear sweep voltammetry were carried out at 30°C in 0.5mol/l H₂SO₄. The sweep rate was 0.4mV/sec from -2 to 2V. Counter and reference electrode were graphite and saturated calomel, respectively. After corrosion tests, observations of surface microstructures were conducted by optical and electrical microscope. Characterization of surface oxide layer by SEM-EDS were also carried out.

As consequences of anode polarization, corrosion-started voltages were shifted to anodic direction as Cr content increased. The current of V-4Cr-15Ti alloy decreased at the area of passive-oxide formation rather than that of V-4Cr-4Ti alloy. It is supposed that to increase Cr content is effective to improve corrosion property and to increase Ti content is effective to stabilize the passivity of the alloy.

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04-93

Deformation Properties of V-4Cr-4Ti in Compression

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Vanadium alloys are of interest as potential first wall structural materials because of their low induced activation characteristics coupled with generally good high-temperature strength and thermal stress factor. The irradiation conditions for the first wall structural material may include temperatures in the range of ~400°C where very high hardening can occur due to production of a high density of small defect clusters that are easily sheared by dislocations. Accompanying the high hardening is rapid onset of necking in a tensile test and very low uniform elongation. Microstructurally all of the dislocation activity occurs in a few channels that have been cleared of defect clusters. To understand the effect of stress state on the propensity for localized deformation in vanadium alloys compression specimens fabricated from Heat 832665 and NIFS-1 heats of V-4Cr-4Ti are currently under irradiation in the High Flux Isotope Reactor at 425°C and 600°C. Control samples were also fabricated. Presented here are results of room temperature, 425°C, and 600°C compression tests on the unirradiated controls. Results are compared with uniaxial tensile data from the literature.

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04-94

Evaluation of Interfacial Strength between Yttrium Oxides and Vanadium

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Since self-cooled blanket design with liquid lithium is primarily considered when vanadium alloys would be used as structure materials for fusion reactor, it is vital issue to reduce pressure drop due to MHD force in magnetic field. Insulator coating inside of the lithium channel is major option to solve the issue. Several candidate materials for the coating such as oxide and nitride ceramics have been assessed from viewpoints of its bulk properties. From successful assessments, Y₂O₃ and Er₂O₃ resulted in the prime candidates. Selection of coating method that can be applied to the complex structures like tubing channel and elbow are now under consideration. A good knowledge of the mechanical properties of the interface between the ceramics and vanadium base metal is of essential for reliable design of the coating including their preparation methods. First-principles calculation became a powerful tool to explore characteristic of the interfaces.

In this report, yttrium oxide as prime candidate ceramics was selected for fundamental study of the metal and ceramics bonds in terms of their strength. Solid-state diffusion bonding at elevated temperature, DC-sputter coating of yttrium in oxidation atmosphere followed by heat treatment and low-vacuum plasma-spray were utilized to make Y₂O₃/V bonds. The bonding behaviors were discussed compared with their ideal interfacial strength evaluated by the first-principles calculation.

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04-95

Impurity behavior in V-4Cr-4Ti-Y alloys produced by levitation melting

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Reduction of oxygen level and control of Ti-C, N, O precipitate distribution are critical to improve workability, weldability and mechanical properties of low activation vanadium alloys. The present authors have found that yttrium (Y) addition is effective for reduction of oxygen level, and have been developing a levitation melting as a new process for high purity and large scale V-4Cr-4Ti-Y alloy ingot. In the present paper, V-4Cr-4Ti-Y alloy was fabricated by 2.5 kg-scale and 10 kg-scale levitation melting.

In the 2.5 kg-scale melting, oxygen level of the V-4Cr-4Ti alloy without Y addition was 496 wppm, while the one with 0.1 wt% Y were 108 wppm. The oxygen level was decreased with increasing Y amount. 0.5 wt% Y addition resulted in a lower oxygen level as 51 wppm. Y₂O₃ precipitates were identified in the ingot surface slug by X-ray analyses. These results indicated that Y is effective agent to sweep oxygen out to the ingot surface by the formation of Y₂O₃. On the other hand, increase of Y lead to degradation of workability and impact property, which is attributed to large Y-containing inclusions observed in the matrix of the alloy. Considering a trade-off between oxygen level and mechanical properties, Y addition amount was set at 0.15 wt% for the large-scale-10 kg levitation melting. Impurity behavior in the melting and breakdown processes, and Y effect on impurity precipitation and mechanical properties were discussed.

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04-96

Effects of Si, Al and Y Additions on Neutron Irradiation Behavior of V-Cr-Ti type Alloys

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It is known that irradiation behavior of vanadium-base alloys depends on the concentration of its interstitial impurity levels. It has been shown that the impurity levels of the alloys can be controlled by small additions of silicon, aluminum and yttrium by means of their scavenging effect. Initial impurity levels were successfully reduced by modification of melting process as demonstrated by NIFS-heats. In this paper, mechanical properties of the series of V-4Cr-4Ti-Si-Al-Y type alloys were examined after neutron irradiation to see the effects of the small additives.

Neutron irradiation was carried out at Japan Materials Test Reactor (JMTR) to the fluence of $1 \times 10^{20} \text{ n/m}^2 (>1 \text{ MeV})$ at 290C. Tensile tests and Charpy impact tests were carried out using miniaturized specimens. Results from tensile tests before irradiation at temperature of liquid nitrogen with initial strain rate of 6.7×10^{-4} to $10^{-2} / \text{s}$ showed that yttrium addition up to 0.5wt.% affected neither flow stress nor elongation of the V-4Cr-4Ti-Y alloy but reduced their reduction in area depending on both of the amounts of yttrium additions and the strain rates. Dimple pattern in the fracture surface of the specimens indicated that yttrium precipitates might be starting points of fracture. The effects of the small additives, especially yttrium, will be discussed in terms of their mechanical properties including microstructure evolution after irradiation.

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Helium Gas Permeability of SiC/SiC Composite After Heat Cycles

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SiC/SiC composite is candidate for blanket structure material. The operation temperature is high, approximately 1100 K, so that a high efficiency of energy conversion is expected. In the SiC/SiC blanket, helium gas will be employed as coolant. There is a concern with respect to leakage of helium gas into a fusion plasma, since SiC/SiC is believed to be porous. Alpha heating power is reduced by fuel dilution if the leakage rate of helium gas exceeds helium production rate due to fusion reactions. Numerous SiC/SiC composites recently have been developed at Kyoto University and the helium gas permeability was measured using a permeability measurement device at Hokkaido University. The permeability of SiC/SiC composites developed by several NITE processes showed a very low permeability with a range from 10^{-11} to 10^{-8} m²/s. The blanket module can be made using only SiC/SiC composite if the permeability is kept low, though a vacuum pumping is required for the blanket module.

The blanket receives heat cycles owing to startup and shutdown of a fusion reactor. Then the concern is an increase of permeability due to the heat cycles. In the present study, heat cycles were applied to SiC/SiC composite made by NITE process. The maximum temperature was adjusted in the range from 1100 to 1300 K, and the heating speed in the range from 6 to 10 K/s. The cycle number was taken up to 120. This condition covers heat cycles for at least 10 years in blanket module. The permeability remained the same when the maximum temperature was lower than 1200 K or the heating speed was lower than 8 K/s. The increase of permeability was observed only when the maximum temperature was 1300 K, the heating speed was 10 K/s and the cycle number exceeded 60. The surface morphology of the SiC/SiC composite with an increased permeability was observed. The boundary region between SiC fiber bundle and SiC matrix became porous, and the matrix particles with a size of several microns were lost at the surface. The maximum temperature - heating speed diagram for operation regime of SiC/SiC blanket module was obtained using the above results.

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04-98

Evaluation of Ion Irradiation Effect on Mechanical Properties of High Purity SiC by Flexural Test

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To understand the mechanical property changes of high purity SiC/SiC composites by neutron irradiation is critical issues for realizing advanced nuclear energy systems. It has been reported that significant degradation of mechanical properties was not seen in high purity SiC/SiC composites even after high dose and high temperature irradiation. To understand irradiation effect of the SiC/SiC composites precisely, it is required to understand irradiation effect on high purity SiC which is the constituent of the composites. There have been very limited reports on the irradiation effect on mechanical properties of high purity SiC. Strength of dense ceramics is basically determined by the defects near surface, while the damage introduced by the ion irradiation is limited to the surface. The purpose of this study is to establish the simple evaluation method of mechanical property of β -SiC after ion irradiation by mean of flexural test.

The samples used in this study were CVD (chemical vapor deposition) SiC. Dimension of specimens was 1x1x24mm. Ion irradiation experiment was conducted at DuET Facility at Kyoto University, using 5.1MeV Si²⁺ ions.

Irradiation temperature and total dose at irradiated surface was up to 1873K and 2.1dpa, respectively. Same specimens were neutron irradiated in JMTR, and mechanical properties were compared with those of ion-irradiated specimens. Irradiation temperature and damage were 673-1023K and up to 0.6dpa, respectively. At least 20 samples were irradiated at each condition, and three point flexural test, where tensile stress was applied to the irradiated surface, and nano-indentation hardness test were carried out. The values of flexural strength were analyzed statistically by Weibull analysis. Fracture surfaces and indented surfaces were observed by field emission scanning electron microscopy (FESEM).

Flexural strength of CVD-SiC following ion irradiation increased in all condition, although the strength and weibull modulus decreased with increasing irradiation temperature up to 1273K. This tendency was also observed in fracture toughness by indentation test. It was revealed by SEM observation that the initial cracks propagation region of unirradiated sample almost corresponded to the damage area of the irradiated sample, and less cracks were observed in the irradiated sample than unirradiated sample. From these results, flexural test seems reasonable and consistent to evaluate mechanical property of β -SiC after ion irradiation. The details of temperature dependency of flexural strength will be discussed with the results of the neutron irradiated specimens.

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04-100

Electrical Conductivity of SiC/SiC

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The design of a Dual Coolant Blanket Module (DCBM) is being considered as one element of the U. S. "ITER Mission." Within the flow channels of a DCBM, a key component is a Flow Channel Insert (FCI) that provides electrical and thermal insulation between the flowing, hot liquid Pb-Li metal and the helium-cooled ferritic steel channel walls. A ceramic, fiber-reinforced silicon carbide composite (SiC/SiC) has been proposed as a promising high temperature, radiation and corrosion resistant material for construction of FCIs.

To carry out the required FCI-functions, the SiC/SiC material must have a relatively low transverse electrical (EC) and thermal conductivity (according to preliminary models, about 1-10 S/m and 1 W/mK, respectively) in the envisioned 500-800°C temperature operating range. To reliably measure the transverse EC as a function of temperature up to 800°C, 2-probe AC and 4-probe AC and DC methods were compared. A 2D-SiC/SiC composite made with advanced Hi-Nicalon™ type S woven fabric and a chemical vapor infiltrated (CVI) matrix and with a thin CVD-carbon interphase was used as a reference material. To estimate the influence of the carbon interphase on the transverse EC, conductivity measurements were made in the transverse and in-plane directions for as-received composite and for composite with the carbon interphase removed by oxidation. Also, the EC of a bare type S fiber bundle was measured. Results will be presented and analyzed using an appropriate conductivity model based on constituent properties and composite architecture.

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04-101

Bubbles in Neutron Irradiated SiC Fibers

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A fusion environment will induce large quantities of helium by transmutation in SiC composite materials. However, experiments intended to understand the evolution of bubble formation in SiC for a fusion environment are difficult to perform. We propose to examine Sylramic SiC fibers following irradiation at 300 and 800°C in the HFIR 14J experiment to ~10 dpa. Sylramic fibers contain 2.3 wt% B in the form of 50 nm TiB particles. Transmutation of the boron to helium and lithium will produce 100 appm He non-uniformly distributed around TiB particles, larger particles developing haloes containing higher concentrations of helium. Therefore, examinations are expected to provide quantitative information on helium bubble formation in SiC fibers over a range of helium levels. Results of the examination will be reported.

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04-102

Effect of Al and Be as Transmutation Products on Formation and Growth of Helium Bubbles in SiC/SiC Composites

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Silicon carbide fiber reinforced silicon carbide matrix (SiC/SiC) composites are considered as functional and structural materials to be used for first wall components in fusion reactors. In the fusion environment, helium and hydrogen are produced and helium bubbles can be formed in the SiC by irradiation of 14-MeV neutrons. The formation of helium bubbles might reduce the tensile strength and thermal conductivity. Wakai et al. reported that the formation and growth of helium bubbles in ferritic/martensitic steel was suppressed by carbon ions pre-implantation. Recently, Tyranno SA SiC fiber, which is high crystalline and near-stoichiometric SiC fiber, has been developed and it includes a slight amount of Al as an impurity. Al and Be atoms as transmutation products are produced in SiC/SiC composites by irradiation of 14-MeV neutrons. The effect of Al or Be atoms on the microstructural change in SiC/SiC composites has not been investigated. In this study, the effect of Al and Be ions on the formation and growth of He bubbles and microstructural change in SiC/SiC composite was investigated.

Al or Be ions were implanted uniformly from 1.0 to 1.7 μm in SiC/SiC composite by changing the implanted energies at 1000 °C. Two kinds of ions implanted specimens were prepared; the concentrations of Al or Be were approximately 100 and 1000 appm. The microstructural change of the matrix and fiber in Al or Be ions pre-implanted SiC/SiC composite up to 1000 appm was not observed. The un-implanted and Al or Be pre-implanted SiC/SiC composites were irradiated by simultaneous triple ion beams (6.0 MeV Si^{2+} , 1.0 MeV He^+ and 340 keV H^+) to 10 dpa at 1000 °C. Helium bubbles were formed in both matrix of the un-implanted and Al or Be pre-implanted SiC/SiC composites irradiated by triple ion beams. The difference of the formation and growth of He bubbles between the un-implanted and Al or Be pre-implanted SiC/SiC composite was observed. The size of He bubbles in Al pre-implanted SiC/SiC composite was slightly larger than that in un-implanted SiC/SiC composite. The effect of Al or Be ion as transmutation products and impurity on the formation and growth of He bubbles will be discussed.

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04-103

Swelling and Time-Dependent Crack Growth in SiC/SiC Composites

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SiC-based continuous-fiber composites are considered for nuclear applications but concern has centered on the differential materials response of the fiber, fiber/matrix interphase (fiber coating), and matrix. When a coated fiber composite is subjected to thermo-mechanical loading, high stresses often occur in and around the fiber. In our study, a continuous fiber composite was simulated by four concentric circular cylinders to explore the magnitude of these stresses when irradiation swelling of the various components is added to the thermo-mechanical loads. The model was used to calculate the radial, axial, and hoop stresses in the composite components as a function of dose. Two fiber types were studied, SiC Type-S and Hi-Nicalon. Three types of transversely isotropic carbons were studied for the fiber coating: high-density isotropic carbon, low-density isotropic carbon, and high-density slightly anisotropic carbon. The outputs of this model were then input into a time-dependent crack-bridging model to estimate component life in a hypothetical fusion irradiation environment where thermal and irradiation creep of SiC-based fibers is considered. The fusion environment consists of a neutron flux of about 0.44 dpa/yr together with a temperature range of 800 to 1400°C and a low, but significant, oxygen concentration in otherwise high-purity He. The synergistic effects of neutron irradiation on the local stresses together with the environmental effects will be explored using the crack growth model. The results of the crack growth model together with the swelling model will be used to generate a crack growth mechanism map to detail the synergistic effects on cracking in SiC/SiC due to environmental effects, neutron-induced swelling, and temperature. The mechanism map will highlight areas of concern for SiC-based materials in Fusion energy systems.

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04-104

Microstructural Evolution Analysis on NITE SiC/SiC Composite Using TEM Examination and Dual-Ion Experiments

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Nano-powder Infiltration and Transient Eutectic Phase (NITE) process is able to fabricate SiC/SiC composites having high purity, dense, and stoichiometric matrix. Another progress of the research is the development of Tyranno-SA fiber which is crystallized, stoichiometric SiC fiber. The NITE SiC/SiC composite is expected to have a great potential as a fusion material, but the precise microstructure and the irradiation effects for NITE SiC/SiC composites are not well known. In present research, precise microstructural investigation and chemical analysis of NITE SiC/SiC composites were performed using an advanced transmission electron microscope. A dual-beam ion irradiation research was also used study the irradiation effects focusing on the migration of displacement damages, helium gas and impurities.

A JEOL JEM2200FS omega filter TEM was used for the analysis of microstructural construction of NITE SiC. NITE SiC had dense, isotropic grains having several hundreds nm diameter. Few secondary phases and pores were observed. Precise chemical analysis detected very thin alumina-rich layers on grain boundaries and on the surface of carbon coating of fiber reinforcements. A 1.7MeV tandem and a 1 MeV singleend accelerators at DuET facility, Kyoto University were used for the dual-beam experiments. The dose rates and irradiation temperature were over 50 dpa and 1200 °C, respectively. From 800 °C to 1200 °C, the NITE SiC/SiC composite after single-ion irradiation at 10dpa kept the microstructural stability. The SiC grains and secondary phases did not change significantly after the irradiation. The synergistic effects of heavy irradiation and helium gas for NITE SiC/SiC composites at elevated temperature are discussed in present research.

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04-105

The Microstructural Evolution of SiC/SiC composites under Multiple-beam Ion Irradiation at Elevated Temperature

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Silicon carbide fiber-reinforced Silicon carbide composites are the major candidates as the advanced structural materials for the future fusion power reactor due to its low induced radioactivity, high specific strength, and high temperature strength. One of the main concerns to use these materials in the fusion environment is the radiation stability of the microstructures in advanced SiC/SiC composites which in turn will affect their high temperature mechanical strength. In this study, we irradiated two types SiC/SiC composites made with advanced SiC fibers namely, Tyranno-SA and Hi-Nicalon Type-S, respectively, by using dual-ion beam (6 MeV Si³⁺ and 1.13 MeV He⁺) at a dose rate of 4×10^{-6} dpa/s and 150 appm He/dpa. The experiments were performed at elevated temperatures (600°C~1000°C) and under high vacuum (1×10^{-7} Torr). The microstructure analysis is done by transmission electron microscopy.

In the experiment of 10dpa at 600°C, there was no cavity or bubble found in both materials: Tyranno-SA/SiC and Hi-Nicalon Type-S/SiC. In the experiment of 10dpa at 800°C, we found bubbles in the matrix of both SiC composites, (fig. 1) but not in the fiber of both SiC composites. The average size of bubbles in the matrix of both SiC composites is about 2~3nm. In the experiment of 100dpa at 800°C, bubbles were found in the matrix of both SiC composites (fig. 2), and also in the fiber of Tyranno-SA/SiC, (fig. 3) but not in the fiber of Hi-Nicalon Type-S/SiC. It is believed that due to the grain size of Hi-Nicalon Type-S is much smaller than that of Tyranno-SA. The size of bubble in the SiC matrixes of both composites is about 8~10nm, and about 2~3nm in fiber of Tyranno-SA/SiC. In the experiment of 100dpa at 1000°C, bubbles in the matrix of Tyranno-SA/SiC and Hi-Nicalon Type-S/SiC, (fig. 4) and the fiber of Tyranno-SA and Hi-Nicalon Type-S (fig. 4) were found. The size of bubbles in the both matrixes of SiC composites is about 30~40nm. The bubbles that were found in the fiber of Tyranno-SA/SiC is about 10~15nm, and 1~2nm in the fiber of Hi-Nicalon Type-S/SiC.

The triple beam irradiation system have completed recently. The experiment of dual-beam irradiation (15000appm He⁺ and 6000appm H⁺ ions) at elevated temperatures (800°C~1000°C) and triple-beam irradiation (150appm He/dpa and 60appm H/dpa) at 800°C are underway and we will report more results in the symposium.

04-107

Slip-Infiltration of SiC-Fiber Preforms for Production of SiC/SiC Composites

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Fibre-toughened SiC/SiC composites are considered as a possible candidate material for the first-wall blanket of the future fusion reactor because of their low activation behaviour and high-temperature capability. However, current materials made by chemical vapour infiltration (CVI) are characterised by a certain amount of internal porosity and high processing cost. As an alternative method, a slip-infiltration technique was used to prepare a dense composite.

The main problems to be solved were the high sintering temperature of SiC, the high neutron activation of commonly used sintering additives and the poor adhesion of the matrix phase to the SiC fibre preform. The main stress in the present investigation was therefore to find a sintering additive from a limited number of low-activation elements, that would enable sintering of SiC to high density at moderate temperatures. Candidate sintering aids, alternative to conventionally used, was selected on the base of contact gamma dose data and on the survey of relevant phase diagrams.

Using adapted slips containing mixtures of surface-modified micro- and nano-powders of SiC and sintering aids, the SiC-fibre preforms were infiltrated under vacuum. The infiltrated samples were then carefully dried and sintered in an inert atmosphere at temperatures from 1400 to 2000°C. After firing, the specimens were characterised by X-ray diffraction, optical and analytical electron microscopy. High-resolution transmission electron microscopy, EDXS, electron energy-loss spectroscopy (EELS), high-angle annular dark-field detector imaging and electron nano-diffraction were used for the characterization of the fibre-matrix interfaces, the chemical composition of the phases present and the crystal structure.

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04-108

Irradiation Creep of Chemically Vapor Deposited Silicon Carbide as Estimated by Bend Stress Relaxation Method

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Creep deformation and rupture are major lifetime-limiting mechanisms for high temperature materials for structural applications. In the prospective fusion reactor environments, irradiation-induced/enhanced creep will be added to thermally activated creep deformation. In many cases, irradiation creep is caused by preferred absorption of supersaturated point defects at edge dislocations with certain Burger's vectors and hence generally the deformation rates are insensitive to temperature. Irradiation creep data for silicon carbide are extremely limited because of inadequacy of the pressurized tube method that is commonly used for metals. Bend stress relaxation (BSR) method, which estimates the creep deformation rate by measuring the extent of stress recovery in an elastically constrained sample, is a potential technique that helps understanding creep behavior of ceramics in nuclear environments.

In this work, BSR creep experiment for the study on irradiation creep of chemically vapor deposited silicon carbide was developed and performed. Samples machined into thin strips were held within a curved gap in the silicon carbide fixture, and irradiated in High Flux Isotope Reactor at Oak Ridge National Laboratory to neutron fluences of $0.1 \sim 4 \times 10^{25} \text{ n/m}^2$ ($E > 0.1\text{MeV}$) at 873 – 1573K. It was demonstrated that the bend stress retention ratio, m , could be determined to the accuracy of <1% by the developed experimental configuration. Derivation of the irradiation creep coefficients will be discussed based on the obtained m values at the different fluence levels.

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Plenary Session 05 – Materials Challenges for Next Step Fusion Devices

05.1

Materials Challenges for ITER – Current Status and Future Activities

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Approaching the construction of ITER, the materials activity is entering a new stage. During previous ITER phases the materials for various components have been selected based on a comprehensive assessment, summarized in several ITER documents. Results from the ITER materials R&D program have indicated the feasibility of the selected materials and joining technologies to provide the required operational lifetime and structural integrity.

In the current stage, the main materials activities are focused on the following:

- preparing the specifications for material procurements, taking into account the specific ITER requirements;
- further consolidation of the data and providing reliable recommendations on materials properties and performance analysis;
- in some areas R&D is still on-going with the goal of optimizing the design, reducing manufacturing costs and providing additional data needed for the design assessment;
- preparing of the testing program in ITER for the tritium breeding blankets and materials for the next step reactor DEMO.

The status of the ITER Materials Properties Database (MPDB) and the ITER Materials Properties Handbook (MPH) are reviewed. The ITER MPDB includes fully traceable detailed information about the test results from ITER R&D and other open sources. The ITER MPH recommendations are being prepared by an Expert Group based on the harmonized and validated database. These recommendations are being produced in accordance with internationally accepted code procedures and specified for the different components. The key materials issues are reviewed in the paper with emphasis on materials for the ITER vacuum vessel and in-vessel components. Further materials activity during the ITER construction phase and urgent needs are briefly discussed.

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05.2

The role of ITER and associated facilities on the pathway towards fusion energy*M.Seki*¹Japan Atomic Energy Agency, Naka, Japan

Fusion energy realization gives us many tough challenges including burning plasma control and long time sustainment, development of structural materials, development of high heat flux materials and components, development high field superconducting magnets, and development of tritium-breeding high temperature blankets. We have to perform intense R&Ds to tackle and solve these challenges, which requires quite a few experimental facilities. ITER is of prime importance to realization of the extended burn of DT plasmas. The objective of ITER also includes the testing of high-heat flux and nuclear components required to utilize fusion energy for practical purposes.

It is widely accepted that we need to construct Fusion Neutron Sources for development and characterization of neutron-resistant materials. IFMIF is the most realistic facility, which has been designed under international cooperation. To test components under fusion neutron environment, Component Test Facilities have been discussed and designed, which provide a large test space to accommodate full-scale components of breeding blankets for Fusion Power Demonstration Facilities, DEMO.

In the course of ITER site selection, intense discussions were made on the Broader Approach Projects, which will be performed in parallel with ITER to facilitate the realization of fusion energy. EU hosts ITER and Japan Broader Approach Projects. The contents of the Broader Approach Projects are still under discussion between Japan and EU. The projects are likely to include IFMIF-EVEDA (Engineering Validation and Engineering Design Activities), JT-60 Satellite Tokamak, DEMO Design and R&D Coordination Center, and so on.

The DEMO Design and R&D Coordination Center is expected to perform DEMO designs, to identify critical R&D subjects with their targets. In general, the need and level of R&D towards DEMO depends largely on the demand of DEMO. In case we are required to develop economically-competitive highly-reliable DEMO facilities, we have to do a lot of R&Ds with quite a few sophisticated experimental facilities.

The energy extraction system from DEMO is more complicated compared to that of fission reactors. The need to demonstrate blanket cooling system and overall heat transport system is high enough to require well-designed demonstration facilities.

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Parallel Oral Session 06A – Test Blanket Modules for ITER

06A.1

Test Blanket Modules in ITER: an overview on proposed designs and required DEMO-relevant materials

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Within the framework of the ITER Test Blanket Working Group (TBWG), which includes representatives of the six ITER Parties and of the ITER Team, the ITER Parties have made several proposals of Test Blanket Modules (TBMs) to be tested in ITER since the first day of the H-H operation (day_{one}).

The proposed TBMs for installation on ITER day_{one} are the following: 1) He-cooled ceramic/Be TBMs, using Li-based ceramics pebbles and Be pebbles (or porous Be), 2) He-cooled or Dual-coolant (He) Lithium-Lead TBMs, 3) Water-cooled pebble-beds ceramic/Be TBM, 4) He-cooled Li TBM, and 5) Self-cooled Li TBM. The first four TBM types use Ferritic/Martensitic Steel (FMS) structures, the last one Vanadium-alloy structures. Other types of TBMs have been also proposed at a later stage.

This paper shortly describes the proposed TBMs designs, the ITER boundary conditions and the expected TBM operating conditions which have to simulate the corresponding DEMO blanket operating conditions by using engineering scaling. Operating conditions will vary throughout the various ITER phases, starting from the initial H-H phase where no neutron and, therefore, no nuclear volume heating will be present, to the later D-T phase where pulses of up to 3000 s length may be expected.

The paper will be focused, in particular, on the design requirements for structural materials and for functional materials that will be used in each TBM, both from the performance point of view and fabrication point of view.

The required time schedule of the TBMs manufacturing and out-of-pile testing prior to installation in ITER will be addressed in order to establish the main R&D priorities and deadlines.

06A.2

Structural Materials for TBMs in ITER

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The International Structural Materials R&D road map toward ITER TBMs (test blanket modules) and DEMO breeding blankets concepts is reviewed. The reference structural materials are RAFM Fe-(8-12)Cr-W-V-Ta steels and low activation vanadium alloys V-4Ti-4Cr. Application of these materials in the ITER TBM is an important stage to provide an adequate database for the design of DEMO blankets. The current status in the EU, Japan, PRC, RF and USA of the development of the RAFM steels, including nano-particle dispersion strengthened modifications and the status of the vanadium alloys development for TBM projects are outlined.

The main properties such as strength, fracture toughness, creep-fatigue of structural materials, and corrosion and coatings issues are discussed. The major issues of concern are well within our knowledge and process technologies are mostly ready for TBMs applications. Material database is practically ready and further progress is anticipated for the design of ITER TBMs. The materials and their semi-finished product forms can be manufactured on industrial scale with sufficient quality and their welding is also established.

The next important steps include further studies of irradiation effects on mechanical properties of base material and joints. Relevant near term neutron sources therefore are fission reactors and dedicated material test reactors. Intense fusion neutron sources, such as IFMIF, are mandatory to provide an adequate database for the design of DEMO relevant blankets in time. Coordinated international collaboration and cooperation (ITER, IEA, bilateral) is strongly recommended also in future for R&D program on structural materials for TBMs, DEMO and beyond for fusion power plants.

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06A.3

The Irradiation Performance of TBM Welds in Relation to Their Heat Treatment

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In a Test Blanket Module, with neutron radiation and temperatures at selected places of below 325°C, the resistance of a fusion weld to irradiation embrittlement is essential. The Post-Weld Heat Treatment (PWHT) must be specifically aimed at the prevention of excessive embrittlement. The critical parameters for integrity are ductility and fracture toughness, which are strongly related. A frequently used indicator of fracture toughness is the Charpy impact test.

Hot Isostatic Pressing (HIP) is used for producing intricate structures in (near-) net shape in TBMs, starting from solid parts (diffusion welding). HIP is performed at temperatures above the austenisation temperature. Therefore, the entire structure needs tempering. The temperature control during the Post-HIP Heat Treatment (PHHT) must ensure that the entire structure is tempered sufficiently to soften the martensitic lattice. The critical parameter for 'HIPed' structures may not be fracture toughness but the bonding strength, particularly in fatigue (crack growth).

In this paper, the irradiation response is discussed of TIG and EB welds in 2 plate heats 9741 and 9753 of F82H-mod. and TIG and EB welds in the European reference steel, Eurofer97. The welds have been irradiated in the EU High Flux Reactor at temperatures of 275-300°C up to dose levels of 2.5 dpa nominal, and in some cases 8.5-10 dpa nominal. In addition, specimens of HIPed Eurofer97 plate with subsequent PHHT have been incorporated in the same irradiation experiment series in the HFR.

EB welds in F82H had mostly not received any PWHT prior to irradiation. Their DBTT shift was unacceptable. The F82H TIG welds had received a PWHT. The results for unirradiated, and irradiated 15 mm and 25 mm TIG weld are promising. The irradiated Eurofer97 welds show an entire range of shifts of the DBTT, depending on the pre-irradiation hardness. The irradiation response of HIPed Eurofer97 is comparable to plate material with regard to impact properties. Fatigue crack growth experiments show preferential cracking tendency along the diffusion weld.

It is concluded that in principle good properties can be obtained in technological structural materials and welds. The implications for the design and manufacturing of TBMs are discussed.

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Status and Perspective of the R&D on Solid Breeder Materials for Testing in ITER TBMs

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The main line of ceramic breeder materials research and development is based on the use of the breeder material in form of pebble beds; the pebbles are quasi spheroid with small dimension ($d < 1\text{mm}$); because they have a better margin against thermal cracking, can easily fit into complex blanket geometries, and better accommodate volumetric swelling and expansion. EU and Japan have developed in the past several years three materials that are the candidates for the respective home DEMO reactors, which will be used for testing in ITER. They include the Li_4SiO_4 pebbles produced by melt-spraying (EU-FZK), the Li_2TiO_3 pebbles produced by extrusion-spherodization-sintering (EU-CEA), and Li_2TiO_3 pebbles produced by wet process (JAEA). The production of these ceramic breeder pebble materials has already attained a semi-industrial scale and the yielding is sufficient (with few improvements of the production) to fabricate enough pebbles for the respective ITER TBMs.

These three fabricated materials have been largely investigated including microstructure, mechanical stability under thermal cycles, compatibility with the RAFM steel used in the design, thermomechanical properties of pebble beds, tritium release and irradiation behavior under DEMO relevant Li-burn-up.

According to the results of these investigations no killing issues have been identified for any of these materials, however, some important investigations have been completed before taking a final selection of these materials for DEMO application. It is commonly agreed among the experts that the most important criteria for this selection will be the demonstration of their resistance under relevant irradiations. In the EU programme a dedicated one-year-irradiation (HICU) is starting in Petten; in the frame of an IEA EU-JA collaboration where the three mentioned materials will be tested at temperatures and ratio of dpa to % Li burn-up relevant to DEMO conditions.

Another important issue is the mechanic resistance of these materials under loading, assembly and operational conditions. Some out-of-pile tests have been already performed and other are under planning before the construction of the TBM will be started.

The purpose of this paper is to review the status of as-fabricated pebble materials against the blanket performance requirements and to make recommendations on where improvements of pebble characteristics can be made. In addition it will be looked at parallel developments that have recently been proposed as alternative to the mentioned materials (like porous ceramics or heat treatment of glassy Li_4SiO_4 pebbles).

06A.5

ITER Test Blanket Module Functional Materials

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Solid breeder and liquid breeder blanket concepts are being developed to be tested in ITER. In addition to the use of reduced activation ferritic/ferritic-

martensitic steels (RAFS) as the structural material, there are two classes of functional materials being considered. In the class of functional materials, SiC_f/SiC composite with low thermal conductivity is proposed as the flow channel insert (FCI) to perform the functions of MHD and thermal insulation for the dual-coolant Pb-17Li concept. Er₂O₃ and Y₂O₃ coatings and metallic sandwiched inserts are proposed as mandatory MHD insulation options for the self-cooled Li breeder concept. When used with the Pb-17Li breeder self-cooled design and in the dual coolant approach, high thermal performance of > 44% can be projected. Application of these functional materials to respective blanket concepts will be described in this paper. At the same time required properties, development status, and development requirements will also be presented.

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Parallel Oral Session 06B – Ferritic Steels - II

06B.1

Recent Progress in US-Japan Collaborative Research on Ferritic Steels R&D

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Recent progress in reduced activation ferritic steels (RAFS) R&D in the US/Japan collaborative research is overviewed, which includes the issues of irradiation effects on mechanical properties, helium related property changes, some processing issues for fabrication of test blanket modules (TBM) and issues towards DEMO and beyond with higher thermal efficiency.

Irradiation database is accumulating under the US/Japan collaborative research utilizing HFIR and ion accelerators. Neutron irradiation effects on fracture toughness were investigated for a few RAFS. Comparing to the previous data of impact properties, the role of the irradiation hardening on the embrittlement is

discussed with focusing on deformation mechanism and microstructure. Dual-beam and triple-beam irradiation facilities were used to investigate the effects of transmutation He and/or H.

Mechanical properties database, which is a measure of the feasibility of processing blanket components, such as curved thin pipes and partition walls, is compiled from the previous database and recent results with taking rather precise irradiation conditions, test method and specimen size and geometry into account. Recent results of surveillance tests of fatigue properties, fracture toughness and corrosion resistance under the relevant environment are summarized.

In order to examine the feasibility of oxide dispersion strengthening ferritic steels (ODSS) as fusion structural material, recent progress in the ODSS R&D is also overviewed, which includes high-temperature strength, anisotropy in the mechanical properties, corrosion resistance in super-critical pressurized water (SCPW), thermal aging effects and irradiation effects. Raw-powder particles characterization studies are also introduced.

Finally, the strategy of ferritic steels R&D towards DEMO and beyond is shown on the basis of "coupling" utilization of RAFS and ODSS as structural materials of the blankets.

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06B.2

Fracture Toughness and Charpy Impact Properties of Several RAFS Before and After Irradiation in HFIR

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As part of the development of candidate reduced-activation ferritic steel for fusion applications, several steels, namely F82H, F82H with 2%Ni, 9Cr-2WVTa steels and F82H weld metal, are being investigated in the joint DOE-JAERI collaboration program. Within this program, two capsules containing a variety of specimen designs were irradiated at two different temperatures in the ORNL High Flux Isotope Reactor (HFIR). These capsules were irradiated in the HFIR removable beryllium positions with europium oxide (Eu₂O₃) thermal neutron shields in place. Specimens were irradiated up to 5 dpa at target temperatures of 300°C and 500°C. Precracked third-sized V-notch Charpy (3.3x3.3x25.4 mm) and 0.18T DC(T) specimens were tested to determine transition and ductile shelf fracture toughness before and after irradiation. The master curve methodology was applied to evaluate the fracture toughness transition temperature, T₀. Irradiation induced shifts of T₀ and reductions of J_Q were compared with previously reported Charpy properties. Fracture toughness and Charpy shifts were also compared to hardening data reported previously.

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06B.3

Implications of the Temperature Dependence of the Arrest Toughness of Ferrite to an Invariant Master Curve Shape

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The toughness temperature K_{Jc}(T-T₀) curves of ferritic-martensitic steels may have an remarkably invariant master curve shape, where the reference (T₀) depends on the material-condition, and ranges from about -150 to > 200°C. For unirradiated alloys, with T₀ < 0°C, an invariant MC shape is predicted by simple micromechanical models assuming cleavage fracture occurs when an approximately temperature independent critical stress (σ*) contour in front of a crack, encompasses a critical local volume (V*) of material. Finite element (FE) methods are used to determine the loading (K_{Jc}) at cleavage as mediated by alloy's constitutive properties as well as their local fracture properties σ* and V*. Typical σ* are about 2000 MPa or more. Thus cleavage fracture requires both a high yield stress (σ_y) and tri-axial crack tip constraint, elevating maximum local near crack tip tensile stresses to peak values of 3-4σ_y. At low T₀ the shape of K_{Jc}(T) is governed almost entirely by the temperature dependence of σ_y(T). Irradiation hardening increases T₀ (ΔT₀). When T₀ is in the athermal σ_{ys}(T) regime, assuming a temperature independent σ* predicts changes (layovers) in the shape of K_{Jc}(T). This apparent contradiction with observation is resolved by assuming a mildly temperature dependent σ*(T) at higher temperatures. We hypothesize that σ*(T) is due to a corresponding increase in the microcrack arrest toughness [K_{fa}(T)] of the ferrite matrix. We report here the results of an independent study of the temperature dependent initiation and arrest toughness in (100)[010] and (100)[011] Fe single crystals oriented for cleavage, using specially designed composite specimen test techniques. The K_{fa}(T) is weakly temperature dependent below about -100°C increasing from a minimum of about 3 to 5 MPa√m, but rises more rapidly at higher temperatures. Static initiation toughness curves are shifted to lower temperatures, with dynamic loading initiation toughness curves fall in between, consistent with the strain rate sensitivity of σ_y. However, K_{fa}(T) shows a stronger temperature dependence than for σ*(T). Thus we further hypothesize that solute (Si, Cr,...) lattice strengthening shifts the K_{fa}(T) to higher temperatures, consistent with the MC shape.

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06B.4

Thermal Creep Behavior of Japanese Reduced Activation Martensitic Steels

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In Japan, Reduced Activation Martensitic Steels (RAMs) have been researched and developed by Universities and Japan Atomic Energy Research Institute (JAERI) as a first candidate material for the structural material of the DEMO fusion reactor blanket module. Japanese Universities and JAERI have been researched Japanese RAMs, JLF-1 (9Cr-2W-0.2V-0.08Ta) and F82H (8Cr-2W-0.2V-0.04Ta) as a reference material, respectively. Extensive property database of these RAMs including irradiated materials have been established. Creep property is one of the most important properties to determine the component lifetime. However, it is not regarded as the most important issue, because the operation temperature of blanket structural material is around 450°C in the

current design. High performance blanket module is also designed to improve the efficiency of fusion reactor system. In this high performance blanket module, structural material will be operated at higher temperature, so that creep property could control the module lifetime.

Creep property data on JLF-1 and F82H have been obtained at wide test condition range, and characteristics of creep properties of these steels have been almost finished. Additionally, creep behavior of modified F82H (F82H mod3), which contains 0.1%Ta, 0.0014%N, and <0.001%Ti, was also researched. F82H mod3 is a toughness-improved type of F82H to provide enough margin or resistance against the irradiation hardening, because water-cooled blanket system includes the part to be irradiated at low temperature. This paper reports the creep behavior of these RAMs and discusses the differences between them. Irradiation creep behavior of F82H is also introduced in the presentation.

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06B.5

Structure Features of the Heat Resistant RAFMS RUSFER-EK-181.

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The structure features and their influence on the physical and mechanical properties of the heat resistant reduced activation ferritic-martensitic steel (RAFMS) RUSFER-EK-181 (Fe-12Cr-2W-V-Ta) in the initial and after reactor irradiation (BOR-60, 6-8 dpa, the irradiation temperature 320-340°C) are investigated.

The electron microscopy was used to investigate the microstructure of the initial and irradiated states of the steel.

The non-destructive acoustic testing technique for the evaluating embrittlement of the steel was used and the correlation between ultrasonic characteristics and

embrittlement (DBTT temperature) was found from the results of the ultrasonic measurements and the Charpy impact tests of specimens. The elastic module, microplasticity and internal friction (decrement) in the temperature interval from 100 to 1000 K were investigated by the acoustic method.

The modification of the steel chemical composition from the nuclear reactions under the high dose irradiations in neutron spectra of fusion (DEMO-RF), the fast breeder power reactor (BN-600) and neutron source IFMIF were calculated and their influence on the functional properties of the steel are discussed also.

The functional properties are governed by uniformity of crystal structure and by physical nature of phase transformations at all structural-phase levels of such steel. Peculiarities of the polymorphic $\gamma \leftrightarrow \alpha$ and carbide transformations determine differences in levels of short-term and long-term mechanical properties of the steel.

Heat resistance level of the steel (650-700 °C) is ensured by the matrix microstructure, composition, distribution and morphology of the phases (carbide, carbonitride and intermetallide) and the state of grain boundaries. Regimes of the heat treatment for optimization of structure-phase state are defined for the chosen composition of RUSFER-EK-181 steel.

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Parallel Oral Session 07A – IFMIF & Specialized Test Techniques

07A.1

The Role of IFMIF in the Roadmap Toward Fusion Power System

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The world fusion program is now entering a new phase to construct, operate and exploit the ITER with a programmatic objective to demonstrate the scientific and technological feasibility of fusion energy for peaceful purposes. In anticipation that the ITER will be made operational in a decade and the programmatic objective can be met in the succeeding seven or eight years, the roadmap toward the DEMO can be revisited and R&D elements indispensable for fusion energy utilization can be aligned in the horizon of the ITER schedule.

A minimum set of R&D elements essential for fusion energy utilization can be categorized in the following: 1) demonstration of technologies essential to a reactor in an integrated system under fusion environments through ITER construction and operation; 2) integrated testing of the high-heat-flux and nuclear components required to utilize fusion energy through ITER exploitation; and 3) development of structural materials with high irradiation resistance and low neutron-induced activations.

Development of radiation-resistant and low-activation materials is a central R&D issue to realize fusion energy utilization. The world fusion community can now identify candidate materials for the DEMO and extensive R&D efforts have been devoted worldwide to data accumulation and evaluation of the candidates by means of irradiation testing as well as modeling and simulation. In addition, the world fusion community stresses the necessity of an appropriate irradiation test facility, which can adequately simulate the fusion environment and provide qualified data under fusion-relevant neutron irradiation. To this end, the IFMIF Project has been implemented under the framework of IEA Implementing Agreement of Fusion Materials, and conceptual design and key elements R&Ds have been successfully completed during the period of so-called 'Key Element Technology Phase (KEP)'. Discussions are in progress on a possible framework and content of technical activities for the succeeding phase, Engineering Validation and Engineering Design Phase (EVEDA), on the basis of the Joint Paper attached to the Joint Declaration by the Negotiators of the six ITER Parties, 28 June 2005. The EVEDA is planned to focus on the detailed engineering design and the associated prototypical component tests with an objective to providing engineering database necessary for making a decision of IFMIF construction.

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07A.2

Evaluation and Validation of D-Li Cross-Section Data for the IFMIF Neutron Source Term Simulation

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In the IFMIF lithium target, neutrons are generated through the d-Li stripping reaction and various other nuclear Li(d,xn) reactions. The neutron source generation must be represented accordingly in the neutron transport calculation. The McDeLicious Monte Carlo code was developed along this guideline to simulate in the transport calculation the neutron generation on the basis of evaluated d + ^{6,7}Li cross-sections. A first set of d + ^{6,7}Li cross-section data was evaluated previously in a collaboration of Forschungszentrum Karlsruhe and INPE Obninsk.

Tests against thick lithium target experiments showed good agreement for the total and forward neutron yields. Recent measurements of the neutron angle-energy spectra indicated, however, a significant overestimation of d-Li neutrons populating the low energy range. New measurements of double-differential d+^{6,7}Li cross-sections showed severe deficiencies of the neutron angular distributions and the inability to properly represent the population of residual nucleus excited levels.

These results initiated an effort to re-evaluate the d + ^{6,7}Li cross-section data applying a new methodology which takes into account compound nucleus reactions, pre-equilibrium processes, stripping and direct interactions. While the first two reaction processes are represented with the GNASH nuclear model code, the nuclear stripping is described with the semi-empirical Serber model and the direct reactions by the DWUCK4 code. A microscopic optical model potential was used for incident deuterons and a modified Koning global optical model potential for neutrons and protons.

The re-evaluated d + ^{6,7}Li cross-section data were prepared in the laboratory frame, stored in standard ENDF-6 data format and processed by the NJOY code system. A series of benchmark calculations was performed with McDeLicious to test the new data evaluations against experimental thick and thin Lithium target neutron yield data up to 40 MeV deuteron energy.

In the paper, the new evaluation methodology is outlined, data are presented for specific d + ^{6,7}Li reaction cross-sections, detailed results are given for the benchmark calculations performed with McDeLicious on thick and thin lithium target experiments, and, eventually, the impact of the updated data evaluations on the neutron source term simulation for IFMIF is discussed.

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07A.3

Preliminary Assessment of the Safety of IFMIF

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The International Fusion Materials Irradiation Facility (IFMIF) will have an important role to play on the path to fusion power, through the testing and qualification of materials under high energy neutron irradiation. Results from IFMIF tests will be key requirements in the design and licensing of a fusion DEMO power plant. Once a decision to proceed with the construction of IFMIF is reached, therefore, delays must be avoided, and in particular the licensing of the IFMIF facility must proceed smoothly. It is not known, of course, what will be the regulatory framework within which IFMIF will be constructed and operated, but it can be anticipated that authorities will need a demonstration of satisfactory safety performance.

With this background, a preliminary safety analysis report has been prepared for the current design of IFMIF, based on the various analyses that have been performed in previous phases of the IFMIF project. The major systems of the IFMIF facility will include dual high-current deuteron accelerators, a lithium target and associated liquid lithium metal loop, and the test cell in which samples are exposed to the intense high energy neutron fluence. Each of these systems introduces potential hazards.

The safety report presents an assessment of these hazards and the measures taken to mitigate them. It covers topics including safety approach and design, radiological and energy source terms, effects of normal operation, radioactive materials arising from operation and decommissioning, occupational safety and public safety analysis. The outcomes of this assessment are summarized in this paper.

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07A.4

Thermo-Structural Design of the Replaceable Backwall in IFMIF Liquid Lithium Target

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The International Fusion Material Irradiation Facility (IFMIF) is an accelerator-based intense neutron source for testing candidate materials of fusion reactor. The major function of the Li target assembly is to provide a stable Li jet with a wave amplitude less than 1 mm up to a speed of 20 m/s under irradiation of a 10 MW deuterium beam. For maintaining the required stability of the high-speed liquid Li flow, thermo-structural design of the backwall is one of critical issues. Among the target assembly, the backwall (stainless steel 316 or RAFM) is located in the most severe region of neutron irradiation (50 dpa/year). In a reference design, minimum thickness of the backwall is 1.8 mm. In case of the stainless steel 316 backwall, thermal stress and deformation calculated by ABAQUS code with a maximum nuclear heating rate of 25 W/cm³ at the center are 458 MPa and 2 mm with a thermal contact of 15.8 W/mK and 267 MPa and 0.3 mm with a thermal contact of 150 W/mK, respectively. Although the thermal stress in the latter case is near a 3Sm value (250 MPa), a contact pressure of 1 MPa is needed to realize a thermal contact of 150 W/mK. To mitigate a design requirement on the contact pressure, dependence of minimum thickness of the backwall in a range of 1.8 mm to 5 mm on thermal stress and deformation is evaluated in case of a thermal contact of 15.8 W/mK. Although reduction of the thermal stress and the deformation were obtained as increase of the backwall thickness, the thermal stress in case of 5 mm backwall is 405 MPa higher than 3Sm value.

To optimize the backwall design, thermo-structural analysis with a thermal contact between 15.8 W/mK and 150 W/mK is in progress. In addition, evaluation of material data base (stainless steel 316 and RAFM) and design modification of the backwall structure for reliable design of the backwall will be also presented.

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07A.5

The Role of Small Specimen Test Technology in Fusion Materials Development

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Small specimen test technology (SSTT) has long been an integral part of fusion materials development. Past work has led to an array of techniques that have not only provided a means of efficiently using available irradiation volumes but have led to improving insights and understanding of the role of specimen size in deformation and fracture phenomena. A significant driving force to advance this technology has arisen from the expectation of a high-energy neutron source for

development and verification of an engineering database for materials for fusion power reactors, and hence many of the techniques have reached maturity as evidenced by the frequency of and reliance upon their application in irradiation experiments. The need to use small specimens has also fostered a science-based approach to their development, leading to new approaches to fracture assessment, and to improved opportunities to explore the effect of stress state on deformation phenomena and constitutive laws in irradiated materials. This in turn has enabled large matrix single variable experiments and highly controlled basic mechanism studies, and has demonstrated the advantages of combining small specimen testing with finite element modeling of deformation and fracture phenomena. As the fusion community increases its focus on design and construction of next generation fusion machines, the role of creep, fatigue and high temperature constitutive behavior in high-energy neutron irradiated materials will require increased efforts on the part of the SSTT community to address these issues. This paper will provide a context of the past and present work on SSTT and explore the opportunities to build on past successes to address future needs.

Parallel Oral Session 07B – Development of ODS Steels

07B.1

Current Status and Future Prospects of ODS Steel Development for Fusion

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For specific blanket and divertor applications in future fusion power reactors a replacement of presently considered Reduced Activation Ferritic Martensitic (RAFM) steels as structural material by suitable oxide dispersion strengthened (ODS) ferritic martensitic steels would allow a substantial increase of the operating temperature from ~550°C to about 650°C or even more.

The work in Japan on 9Cr ODS steels concentrated on the development of cladding materials for fast reactors. The control of extrusion temperature, excess oxygen and titanium content allows the control of the nano-mesoscopic structure and thus the mechanical short and long-term behavior. The fabrication process for cladding tubes as well as the pressurized resistance welding technology to weld claddings and end-plugs is well developed and fuel pins have been fabricated and are being irradiated in the Russian BOR-60 reactor.

The work in Europe concentrated on the development of RAFM-ODS steels basing on the 9CrWVTa EU reference steel Eurofer. While these RAFM-ODS steels of the first generation showed good tensile, creep and low cycle fatigue properties, they revealed poor impact behavior and high temperature ductility. Selecting a specific production route which included rolling and appropriate thermal treatments, DBTT could be shifted from values between +60 and +100°C for hiped Eurofer-ODS of the first generation to values between -40 and -80°C.

12-14Cr ferritic ODS steels that are investigated in Japan and USA would allow a further increase of the operating temperature to 750°C and more. The control of the consolidation temperature and Ti content led to a new class of material, the nanostructured ferritic alloys (NFA) that contain a high number density of Y, Ti, and O enriched nano-clusters resulting in high strength and creep strength.

The excellent mechanical behavior, high irradiation resistance, as well as the availability of qualified fabrication and joining technologies, make this class of materials suitable for the application as structural material in advanced breeding blanket and divertor concepts.

The good corrosion resistance in aqueous environments and good compatibility to liquid metal expand the field of application also to advanced fission reactors and accelerator driven actinide conversion systems.

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07B.2

Kinetic Monte Carlo Simulations of Nanocluster Formation and Structure in Nanostructured Ferritic Alloys

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Lattice-based kinetic Monte Carlo simulations have been performed to better understand the formation and stability, strengthening contributions and radiation damage management capabilities of nm-scale clusters, or nanoclusters (NCs), in nanostructured ferritic alloys (NFAs). A key fundamental issue is the local atomic arrangement (structure) and chemistry of the Y-Ti-O NCs in the matrix. Bond energies are obtained from calculations of the mixing enthalpies within a regular-solution thermodynamics model, making the simplification that the calculations are performed in pure Fe (as opposed to Fe-14Cr) and omit the impurity elements. The mixing enthalpies are obtained from the free energies for Y, Ti and O solute atoms. The simulations are performed on a body-centered cubic lattice, with oxygen diffusion on an octahedral interstitial sub-lattice. NC evolution is simulated, starting from super-saturated solution. The resulting structures are compared to experimental observations by transmission electron microscopy, small angle neutron scattering, atom probe tomography and positron annihilation spectroscopy. The results provide atomic-level insight into the NC structure and chemistry and provide a basis for understanding the thermal and radiation stability of the NCs in NFAs. Future work will focus on including the effect of high, non-equilibrium vacancy concentrations on the precipitation kinetics.

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07B.3

Nano-Mesoscopic Structural Control of 9Cr ODS Martensitic Steel for Improving Creep Strength

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9Cr-oxide dispersion strengthened martensitic steel (9Cr ODS) has been identified as a promising candidate for fusion reactor blanket material as well as advanced fast reactor (FR) fuel cladding material because of its excellent radiation resistance and improved creep strength. We reported that creep strength of 9Cr ODS can be improved by appropriately controlling hot-extrusion temperature and chemical compositions (oxygen and titanium concentrations) and that this creep strength improvement should be correlated with number density of oxide particles and volume fraction of elongated grain containing ultra-fine oxide particles. The elongated grain is parallel to extrusion direction and would be so-called δ -ferrite phase. However, we designate this phase residual- α phase because this phase should remain untransformed from ferrite (α) to austenite (γ) during hot-extrusion. For the purpose of clarifying the mechanism of the creep strength improvement, we will quantitatively evaluate oxide particle distribution and volume fraction of residual- α phase in 9CrODS.

The 9Cr ODS bars containing different concentrations of oxygen and titanium were produced by mechanical alloying (MA) and hot-extrusion process. The hot-extrusion temperature was set at 1150°C and 1200°C. Oxygen and titanium concentrations were controlled to 0.10~0.24 wt% and 0.20~0.46 wt%, respectively. Microstructures of these bars were characterized using field emission type transmission electron microscope (FE-TEM) and electron microprobe analyzer (EPMA). The volume fraction of residual- α phase was evaluated by measuring the concentrated area of ferrite former elements (chromium, tungsten) in the EPMA mapping image.

In the presentation, we will report the qualitatively evaluated results of the oxide particle distribution (particle diameter, number density) in the 9CrODS bars hot-extruded at the different temperatures. We will also report the volume fraction of residual- α phase in the 9Cr ODS bars containing different concentrations of oxygen and titanium. Based on the experimental results, the mechanism of the creep strength improvement by controlling hot-extrusion temperature and the chemical compositions will be discussed.

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07B.4

Influence of Particle Dispersions on the High-Temperature Strength of Ferritic Alloys

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The dispersion of oxide particles in ferritic alloys by mechanical alloying (MA) is a well known process for improving their high-temperature strength and creep properties. In recent years, advances in understanding the mechanical alloying (MA) process have resulted in creating a new type of material, namely, nanostructured ferritic alloys (NFA) that contain a high number density of Y, Ti, and O enriched nano-size clusters, or nanoclusters (NC). Investigations have shown that the NC have structural characteristics that are different from that of oxide particles. In this study, four ferritic alloys based on the composition Fe-14Cr-3W-0.4Ti (nominal wt.%) were developed with a predominant dispersion of either oxide particles or NC. The tensile tests performed on sheet specimens of these alloys showed that the high-temperature strength of NFAs was significantly better than that of the ferritic alloys that were strengthened with oxide particles, which are more commonly known as oxide-dispersion strengthened (ODS) ferritic alloys.

The four ferritic alloys that were investigated in this study were prepared in two pairs by the mechanical alloying (MA) method. In one pair of samples, the

pre-alloyed powders were ball milled with 0.3wt.%Y₂O₃ and extruded at different temperatures, which produced either a low number density of Y- and Ti-based oxide particles with sizes ranging from ~10 to 50 nm or a high number density of ~2-5 nm size NC. In the second pair of samples, the pre-alloyed powders were ball milled with and without 0.3wt.%Y₂O₃ and extruded at the same temperature. This processing condition produced a low number density of Ti-oxides in the alloy that was ball milled without Y₂O₃ and a high number density of NC in the alloy that was ball milled with Y₂O₃. Sheet tensile specimens of these alloys were tested at room temperature and at temperatures ranging from ~360°C to 800°C using a strain rate of 10⁻³ s⁻¹. The room temperature yield strengths of the two alloys containing the NC were 1464 MPa and 1256 MPa while the yield strengths of the two oxide dispersion alloys were 801 MPa (Y-Ti-oxides) and 594 MPa (Ti-oxides). However, the latter two alloys had better ductility than the former two alloys. Details regarding the processing and microstructural characterization of the ferritic alloys will be presented and the tensile test data will be used to compare the effect of variations in particle dispersion and grain size on the high-temperature mechanical properties of these ferritic alloys

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07B.5

Model Experiment for Minor Alloying Element Effect of Dispersing Nano-Particles in ODS Steel

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From the irradiation resistance and high-temperature strength, oxide dispersion strengthened (ODS) ferritic steels are candidate materials for advanced and fusion reactors. For the development of advanced steels the key issue is to homogenize nano-particles into matrix. Recent studies have indicated that Ti addition can homogenize Y-Ti complex particles into ferrite matrix, but the reason of the effect of additional elements has not been clarified. In this model study, we focus on the effect of additional elements, such as IV and V families and other oxide formers, which can control potentially the distribution of the oxide particles.

The materials used in this study were based on Fe-9Cr-Y₂O₃ alloys which were mechanical alloyed (MA) from the powder of Fe, Cr and Y₂O₃, which was added

systematically with the element of Ti, Zr, Ta, V, Nb, Hf, Al, Si and others. Usually ODS fabrication process is required for hot extrusion, but we annealed up to 1150 C for simplify the microstructure. To evaluate the distribution of ODS particles; we used TEM equipped with EDS after electro-polishing or FIB techniques.

(1) In the case of Si or Al addition, oxides were disappeared after MA process, which means Y₂O₃ and other elements should be in solution at non-equilibrium condition. Two types of oxides of Y₂O₃ and Al₂O₃ or SiO₂ developed after the annealing at 850 C, but only complex oxides were developed after the annealing at 1150 C. This result suggests that the oxide formation is independent process for Y and Si or Al.

(2) In the case of Ti addition, oxides also were disappeared after MA process, but developed after annealing at 1150 C. This means that Ti can stabilize complex oxides of Y and Ti, and enhance the fine distribution of the oxides comparing with simple Fe-9Cr- Y₂O₃ alloy. In IV and V elements, Zr had similar effect for homogenization of the particles.

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Poster Session 08

08-2

Clustering Behavior of Ni, Mn and Si in Irradiated and Thermally Aged RPV Steels

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Formation of solute clusters causes embrittlement of the reactor pressure vessel (RPV) materials of light water reactors. Impurity copper is the most enriched element in the clusters, and the bulk concentration of copper is the most important parameter to predict the amount of embrittlement. The copper enriched clusters are also known to contain such elements as nickel, manganese and silicon, which are all added intentionally to control the properties of the RPV materials. These elements basically have high solubility in the RPV materials, and the impurity copper has been known to play a key role for the formation of the agglomerations of these elements.

On the other hand, however, Odette has suggested the formation of Ni/Mn enriched clusters in the materials with no copper and high nickel irradiated to high doses. Recent 3DAP (3 Dimensional Atom Probe) results obtained by JNES, Japan, also show formation of Ni/Mn/Si enriched clusters in the RPV materials irradiated to a very high dose of about $1 \times 10^{20} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$). The behavior of the solute atoms of nickel, manganese and silicon with and without copper is thus now an important issue to understand the mechanism of embrittlement at the high dose region.

In this paper, we perform 3DAP and CDB (Coincidence Doppler Broadening of positron annihilation) experiments on the highly irradiated and thermally aged RPV materials and model alloys with high and low copper concentrations to characterize the behavior especially of nickel, manganese and silicon. We also perform Kinetic Monte Carlo computer simulation of thermal ageing to look at the interactions of these elements in terms of thermodynamics, and then we discuss the effect of irradiation on the solute clustering.

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08-3

Wigner Energy Predicted by Dislocation Accumulation Model

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Graphite has been extensively used in nuclear reactors because of its useful nuclear and thermal properties. In 1942, Wigner predicted significant changes in the physical properties of graphite irradiated by fission neutrons and in 1957 the Windscale reactor fire happened by the effect known as Wigner-energy storage and its release. In spite of a lot of works done so far on irradiation damage of graphite, Wigner-energy is still not clear on the temperature and the dose dependence.

Recent progress in experimental and theoretical analyses by Raman spectroscopy has allowed us to investigate the irradiation-induced phenomena from the viewpoint of in-plane defects of vacancies and their clusters. Based on the change in the Raman spectra during irradiation, a model of irradiation-induced amorphization has been proposed [1]. The model has successfully predicted the temperature dependence of the critical dose for amorphization. However, it cannot predict the change of the defect concentration under irradiation nor give information on the structure of the disordered region. Then, we have modified the disordered region model to the dislocation accumulation model by assigning the disordered region to the dislocation dipole [2]. The model well simulates the experimental results of the square-root dose dependence of the Raman intensity ratio [3] and the dose dependence of the c-axis expansion [4] under irradiation. We have also proposed the growth process of the dislocation dipoles [5].

In this work, we will simulate the dose and temperature dependencies of Wigner energy using the dislocation accumulation model and compare with experimental results.

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08-4

Effects of Cold Work on Degree of Sensitization of Type 316L Stainless Steel

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According to the recent reports on the BWR shroud crackings, the intergranular stress corrosion cracking (IGSCC) was accelerated in the weld bond region that was considerably hardened. Although low carbon stainless steels are considered to be resistant to thermal sensitization, the effect of a severe cold work on the sensitization behavior of the steels is not known. The objective of this research is to make clear the effect of a severe cold work on the degree of sensitization of type 316L stainless steel.

The susceptibility to sensitization of type 316L stainless steel was determined by means of the double loop-electrochemical potentiokinetic reactivation (DL-EPR) tests in a 0.5M H₂SO₄-0.01M KSCN liquid solution. Specimens were thermally aged at 700 °C for different periods up to 100h, and then cold worked (CW) at ambient temperature to reductions in thickness ranging from 25% to 75%.

For the annealed condition (without cold working), the sensitization was increased with increasing aging time at 700 °C as expected. As for the effect of cold work, the degree of sensitization was decreased with increasing degree of cold work. Desensitization occurred in the highly cold worked specimen. Especially the 75% CW specimen exhibited a high resistance to sensitization even after the aging for 100 hr. Metallographic observation revealed that the grain boundary corrosion was remarkably suppressed by the cold work to 75% reduction. It is considered that the excessively increased dislocation density may be the cause of the suppressed sensitization in cold worked type 316L stainless steel.

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Creep mechanism of V-4Cr-4Ti alloys after thermal creep in a vacuum

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Vanadium alloys are candidate materials for fusion reactor blanket structural materials because of their potentially high operation temperatures. However the knowledge about the mechanical property of vanadium alloys at high temperatures is limited and there are uncertainties that may have influenced the results such as the interstitial impurity content of specimens. The objective of this study is to investigate the creep properties and microstructural changes of the high-purified V-4Cr-4Ti alloys, NIFS-HEAT2 [1] by using pressurized creep tubes (PCTs), in order to prepare for in-pile creep tests.

The V-4Cr-4Ti alloy used in this study was produced by NIFS and Taiyo Koko Co. and designated as the NIFS-HEAT2 [1]. The detailed tubing process and fabrication process of pressurized creep tubes have been reported in the refs [2]. The PCTs wrapped with Ta and Zr foils were enclosed in a quartz tube in vacuum. Thermal creep tests were done using the sealed quartz tubes in Muffle furnace at 600, 700, 750, 800 and 850°C. Dimensional changes of PCTs were

measured with a precision laser profilometer to an accuracy of 1µm for the outer diameter measurement. After a creep strain exceeded 20% in a measurement, the creep test was finished and the TEM observation was performed for the specimen pieces cut out from PCTs.

From the results of dimensional changes, the activation energy of creep deformation in the NIFS-Heat2 alloys was about 180kJ/mol. This amount of creep activation energy of PCTs is very similar to that of uniaxial creep specimens of NIFS-Heat1 alloys in the previous study [3]. The creep strain rate of PCTs is several times larger than that of uniaxial specimens. Tentative microstructural observation shows that the grain refining and the formation of fine dislocation-cell structures occurred under creep deformation at 850°C with stress levels of 100 and 150 MPa. The size of grains changed from 25µm to 5µm in average after creep deformation and the length of cell structures was about 0.5µm. The character of Burgers vector of dislocation in dislocation cells was most of $a/2\langle 111 \rangle$ type. The correlation between creep behavior and microstructural changes will be presented in the conference.

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08-7

Oxidation Behavior of a V-4Cr-4Ti Alloy During the Commercial Processing of Thin-Wall Tubing

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Commercial-scale fabrication of thin-walled creep tubing from the V-4Cr-4Ti alloy was investigated using three levels of vacuum during intermediate annealing at 1000-1050C. Annealing in a low vacuum environment (10^{-4} torr range) during each of the ten drawing/annealing cycles resulted in acceptable levels of oxygen pick-up with the average oxygen concentration approximately doubling from the initial level of ~300wppm; however unacceptably high levels of surface cracking developed. With an improved vacuum in the 10^{-5} torr range, it was found that the oxygen pick-up rate accelerated and resulted in a six-fold increase in the average oxygen concentration. Although oxygen reached unacceptably high levels (~1700wppm), the incidence of surface cracking was greatly reduced. Increasing the vacuum into the 10^{-7} torr range, coupled with careful cleaning and gettering procedures resulted in both a minimal rate of oxygen pick-up and a low incidence of surface cracking.

The acceleration in oxidation rate that occurred at the intermediate vacuum level is discussed in terms of a transition from a linear-parabolic oxidation regime in the low vacuum environment, in which the diffusion of oxygen is limited by the formation of a visible oxide film, to a regime of linear oxidation kinetics. In the linear-parabolic regime, oxidation is restricted to a thin surface layer which is subsequently removed by the acid cleaning procedure which follows the subsequent draw cycle. The average oxygen increase per cycle is therefore reduced to acceptable levels; however, the presence of the surface oxide during tube drawing is thought to promote the development of the observed surface cracking. The mechanism of the rapid internal oxidation at the intermediate vacuum level involves the formation of a globular form of the Ti(CON) phase; scanning Auger was used to provide detailed information of the distribution of oxygen between the particles and the matrix. Based on the understanding of the vacuum oxidation behavior of V-4Cr-4Ti, a procedure was developed that yielded high quality thin wall creep tubing with minimal interstitial pick-up.

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08-8

Dynamic and static hydrogen effects on mechanical properties in Vanadium alloys

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Vanadium-based alloys are potential structural materials for first wall and tritium blanket of fusion reactors. A major concern is their high affinity and diffusivity of hydrogen and its isotopes, which can lead to embrittlement at low temperature. The dynamic and static hydrogen effects on the mechanical properties of V and V-4Cr-4Ti will be studied in this paper.

Two kinds of tensile tests were performed for hydrogen-charged miniature specimens: One is with hydrogen charging prior to testing (static charging), and the other is with hydrogen charging during straining (dynamic charging). A part of specimens were irradiated to 10^{20} n/cm² in JMRT at 300 C. The hydride formation, microstructural evolution and fracture were studied by means of TEM and SEM.

Static hydrogen effect: From the result of gas emission and microstructure, it was shown that hydrogen can be trapped by lattice defects, dislocations, vacancies and voids, which are effective up to 500 C. The static charging prior to the straining test generally resulted in hydrogen-induced hardening, which was caused by the hydride formation and stabilized lattice defects. However, the hydrogen-induced softening was observed in the case of low hydrogen concentration.

Dynamic hydrogen effect: In the case of V, the deformation stress dropped obviously just after the dynamic charging, and returned to the original level after stopping of charging; this is the hydrogen-induced softening. In the case of V-4Cr-4Ti, the level of the softening was restricted. The hydrogen-induced softening can be attributed to both of the fast diffusion and interactions of hydrogen with mobile dislocations. The activation volume measured by different strain rates test was decreased with hydrogen charging, which means a small amount of hydrogen can reduce Piers potential for mobile dislocations.

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08-9

Studies of Reactor Irradiation Effect on Hydrogen Isotopes Release from Vanadium Alloy V4Cr4Ti

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Vanadium alloys are most promising materials being considered for lithium blanket-breeder in future fusion reactors. The primary reason for these stems from good combination of physical-mechanical and radiation properties of vanadium alloys. In operational conditions of fusion reactors the very important issue is behavior of vanadium alloy with respect to hydrogen isotopes under neutron and gamma irradiation.

Given paper shows results of first stage of experimental studies of reactor irradiation influence on parameters of hydrogen release from vanadium alloys. This paper specifically describes gas release parameters and parameters of phase transformations of vanadium alloy V4Cr4Ti under various conditions of reactor irradiation.

The vanadium alloy V4Cr4Ti samples were saturated with gas phase hydrogen isotopes at room temperature. All reactor studies of gas release were conducted using two variants of thermodesorption method: differential and integral. Irradiation was carried out at IVG1.M reactor of National Nuclear Center of the Republic of Kazakhstan. Experiments were carried out for various levels of reactor irradiation and showed the effect of irradiation on parameters of hydrogen release from vanadium alloy V4Cr4Ti.

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08-10

Effects of 2.1%Ti Addition on Tensile Properties and Microstructures of an Ultra-Fine Grained V-1.7%Y Alloy with Nano-Sized Y₂O₃ and YN

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Vanadium alloys have many favorable properties for fusion structural applications, such as inherently induced low-activation and high ductility and fracture toughness, however, they exhibit environmental embrittlement and radiation embrittlement. In order to relieve these types of embrittlement, ultra-fine grained V-(1.6-2.6)wt%Y alloys with nano-sized Y₂O₃ and YN are reported to be very effective. The alloys, however, show lower strength above 1100K than a V-4Cr-4Ti alloy due to grain boundary sliding and less solution hardening. Ti is known to be a solution hardening element. In this study, therefore, effects of Ti addition on tensile properties and microstructures are examined for V-1.7wt%Y and V-1.7%Y-2.1%Ti alloys fabricated by advanced powder metallurgical techniques including mechanical alloying and HIP. Tensile tests were conducted at temperatures from 77 to 1273K.

It is found that 2.1%Ti addition significantly suppresses the reduction in high temperature strength and the suppression effect tends to increase with increasing temperature. In addition, the Ti addition increases the ductility at and below 288K though it decreases the yield and tensile strengths. The ductility increase becomes prominent with decreasing temperature: The total elongation of V-1.7%Y-2.1%Ti is 21% at 288K and 27% at 77K, whereas that of V-1.7%Y is 18% at 288K and 9% at 77K. It is also shown that the Ti addition decreases the number density of dispersoids and increases their average size, resulting in an increase in grain size from 340 to 520 nm. The causes of the observed tensile properties and microstructures are discussed.

08-11

Effect of Ti(CON) Plates on Dynamic Strain Aging in V-Cr-Ti Alloys

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The mechanical behavior of the V-4Cr-4Ti alloy and of BCC alloys in general is strongly influenced by interstitial C, O, and N solutes. The deformation behavior of the V-4Cr-4Ti alloy has been shown to exhibit dynamic strain aging (DSA) during tensile testing, typically at temperatures ranging from 300°C to 750°C and strain rates ranging from 10^{-1} s^{-1} to 10^{-5} s^{-1} . The DSA phenomenon is manifested as serrations (continuous) and discontinuous yielding in the Lüders strain and work hardening regimes of stress strain curves and a concomitant negative value in strain rate sensitivity (SRS) for flow stresses. The interstitial impurities O, C, and N, which occur typically at a concentration of 200 to 500 appm in V-4Cr-4Ti alloys, react strongly with Ti to form a series of complexes and precipitate phases. The nature and extent of these reactions significantly affect the mechanical properties of these alloys. Typical thermomechanical processing (TMP) results in a nonuniform distribution and low number density of globular-shaped Ti-oxycarbonitride, Ti(CON) particles in V-4Cr-4Ti. The globular Ti(CON) forms at high temperatures during processing and frequently occurs in a coarse non-uniform dispersion that does not contribute to alloy strength and often leads to a banded microstructure.

A new TMP method was developed that incorporates a solutionization treatment at 1300°C, which causes the dissolution of the globular Ti(CON) and re-precipitation of a high-number density of nano-size Ti(CON) plates with $\{100\}_{\text{bcc}}$ habit at lower temperatures. Sheet tensile specimens that were treated with the new TMP, were tested at temperatures ranging from 400 to 600°C at strain rates ranging from 10^{-1} s^{-1} to 10^{-5} s^{-1} . The decrease in precipitate size and increase in number density was found to increase the high temperature strength properties of the V-4Cr-4Ti alloy. In addition, the DSA behavior was significantly modified by the nano-size Ti(CON) plates; a region of smooth flow preceded the on-set of DSA oscillations in the stress-strain curve (critical strain phenomenon). When only the globular form of Ti(CON) is present, DSA-related serrations occur immediately on yielding. The detailed morphology and distribution of the nano-sized plates are described and possible mechanisms involved in the appearance of a critical strain are discussed.

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08-12

Effect of Internal Oxidation on Microstructure and Mechanical Properties of Vanadium Alloys

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The features of the structure-phase transformations that occur in low-activation (V-Zr, V-Cr-Zr) and model (V-Mo-Zr) vanadium alloys during their internal oxidation at temperatures below the recrystallization temperature were studied. It was investigated how the multiphase structure that is formed in this case affects the thermal stability of the microstructure and the mechanical properties of the alloys. Methods and modes of chemical-thermal treatment have been developed which make it possible to produce multiphase structures with the oxide phase showing practically unlimited dispersibility (the particle size may be as small as a few nanometers). The possibilities for production of multiphase structures which would be stable up to temperatures close to the melting temperature have been substantiated. For high-defect-density structures subject to internal oxidation, it has been demonstrated that the methods developed may efficiently increase the recrystallization temperature of the alloys to $T \approx 0.8 T_{\text{melt}}$. The combined dispersion and substructural hardening occurring in internally oxidized alloys results in a severalfold (2–4 times) increase in their low-temperature and high-temperature strength with the margin of plasticity remaining rather high.

A comparison of internally oxidized (V-ZrO₂, V-Me(Cr,Mo)-ZrO₂) alloys and V-Ti-Cr-system alloys in thermal stability of the microstructure and in mechanical properties suggests that the methods of internal oxidation hold promise for increasing (by 100° – 200°) the operating temperatures of vanadium alloys. It is also expected that the high oxygen content in the solid solution of internally oxidized alloys will promote the recovery of the elemental composition and lengthen the useful life of self-healing oxide-based protective coatings. Furthermore, the fine oxide particles and the boundaries of the microcrystalline structure in vanadium alloys, being efficient sinks for point defects, may substantially increase their radiation stability.

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Irradiation induced precipitates in vanadium alloys studied by atom probe microanalysis

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Vanadium alloy is one of the excellent candidate materials for fusion reactors. It has been reported that significant radiation hardening and embrittlement occurred in V-4Cr-4Ti. According to the previously reported TEM studies, radiation-induced titanium precipitates are the major cause of irradiation hardening, while the microstructural information is still far from comprehensive. Especially, the nature of black-dot clusters which were observed under irradiation at low temperatures is still not clear while the large size of Ti-precipitates with platelet shapes are relatively well characterized. For the analysis of fine precipitates, 3-dimensional atom probe microanalysis (3DAP) is a very powerful tool with its atomic scaled spatial resolution. In the present paper, experimental results using 3DAP combined with TEM observation on fine Ti-precipitates in vanadium alloys are presented.

V-4Cr-4Ti, V-4Cr-4Ti-0.1Si, V-4Cr-(0.1,0.3,1,3wt.%)Ti were prepared. Neutron irradiation was conducted in Japan Material Testing Reactor (JMTR). The damage level was 0.2dpa and the irradiation temperature was 350°C. TEM observation was performed using JOEL-2010 equipped with an energy

dispersive X-ray spectrometry (EDX) detector. A needle-shaped tip of 3DAP specimens were prepared by the electro-polishing technique followed by the focused ion beam (FIB) processing with gallium ions of 30 keV. The elemental distributions in vanadium alloys were investigated using an energy compensated 3DAP.

In TEM observations, platelet precipitates on {100} planes were observed in V-4Cr-4Ti-0.1Si, while a high density of small defect clusters was observed as black dotted contrast in V-4Cr-1Ti. Enrichment of titanium as well as oxygen and titanium oxide in the matrix were observed by 3DAP microanalysis in V-4Cr-3Ti, V-4Cr-1Ti, V-4Cr-0.1Ti, which may corresponds to the precipitates and black dotted defect clusters observed by TEM. The size of precipitates increased with the titanium concentration. Small cluster with a sphere shape were observed in V-4Cr-0.1Ti, while the platelet shape in other alloys. It could be deduced that the spherical cluster was on the nucleation stage of precipitates. In other words, the nucleation of titanium-rich precipitates might be started with the segregation of titanium and oxygen to dislocation loop. Nitrogen and carbon were also contained in the large precipitates of platelet shape in V-4Cr-3Ti, which will be explained by the difference in the extent of strain field depending on the coherency of precipitates.

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08-14

Retention and Desorption Behavior of Helium in Oxidized V-4Cr-4Ti Alloy

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V-4Cr-4Ti alloy is a candidate for blanket first wall and structure material in a fusion reactor. The vanadium alloy first wall receives helium fluxes. It is known that vanadium alloy is well oxidized, so that the retention and desorption behaviors of helium may depend on the oxidized state at the surface. In the present study, the surface thermal oxidation was conducted for V-4Cr-4Ti alloy (NIFS-HEAT-2) after pretreatment of heating at 873 K for 30 min in a vacuum. Oxidation temperature was kept at 873 K for 15 min. The thickness of oxidized surface was approximately 120 nm, which is larger than projected range of helium ion. The helium ion irradiation was conducted for the oxidized and non-oxidized samples in ECR ion irradiation apparatus at room temperature. The energy of helium ion was 5 keV and the fluence was taken in the range from 5×10^{20} to 10^{22} He/m² (4.5-90 dpa). After the irradiation, the desorption behavior and the amount of retained helium were examined using a thermal desorption spectroscopy, TDS.

Sample was heated from room temperature to 1673 K. The helium desorption peaks of the oxidized sample shifted to lower temperature compared with those of non-oxidized sample. The amount of retained helium in both samples increased with the fluence, and then saturated at the fluence of 5×10^{21} He/m². The saturation level of the oxidized sample was 1.9×10^{21} He/m² which was about 75% of that of non-oxidized one. The amount of retained helium at higher temperature was smaller than that of non-oxidized sample. The amount at lower temperature was comparable with that of non-oxidized sample. This means that the helium trapping behavior might be changed by the oxidation. Saturation values were comparable with those of the other plasma facing materials such as carbon and tungsten.

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08-15

Creep of V-4Cr-4Ti Pressurized Tube Specimens

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Pressurized thermal creep tubes of V-4Cr-4Ti have been examined following testing in the range 650 to 800°C and 40 to 250 MPa effective midwall stress for tests lasting $\sim 10^4$ h to provide comparison with tests on similar tubes following irradiation. It is found in all cases that creep is controlled by dislocation motion. But the mechanism changes with increasing temperature from one controlled by the climb and interaction of individual dislocations, to one controlled by sub-grain boundary structure that is created by relaxation of the interacting dislocations to a lower energy planar array. At the lower temperatures, the dislocation structure consists of individual dislocation tangles, whereas at the highest temperatures and lowest stresses, low angle sub-grain boundary structures are found. Additionally, a change in mechanism is indicated from power law creep to Newtonian creep such that the stress exponent drops from ~ 4 to ~ 1 . Although it is possible to explain the Newtonian response as Nabarro-Herring or Coble creep, it appears more likely that behavior is due to Harper-Dorn creep, in which case the change in response occurs at the Peierls stress. We predict that response in-reactor will be similar due to the similar natures of Harper-Dorn and irradiation creep at low stress.

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Diffusional behavior of tritium in V-4Ti-4Cr alloy

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Low activation vanadium alloys have been developed as good candidate structural materials of a nuclear fusion reactor, and their material properties have been examined from various aspects. Tritium retention and permeation in the alloys are important concerns and the diffusion coefficient of tritium is one of key parameters to be evaluated. For pure vanadium, diffusion coefficients of hydrogen isotopes and trapping effect of some alloying elements have been examined well. However, only limited data are available for the newly developed alloys.

In the present study, we have examined hydrogen diffusion behavior in a V-4Ti-4Cr (NIFS-Heat-2) alloy applying a tritium tracer technique, in which a small amount of tritium (T) together with hydrogen (H) was firstly implanted into specimen surface, the specimen was diffusion-annealed at a temperature from 373 K to 523 K, and then the diffusion depth profile of T in the specimen was measured to determine the diffusion coefficient. The tritium imaging plate (IP) technique was used to obtain the diffusion profile of T. In this technique, the diffusion-annealed specimen was exposed to IP for a certain time duration, and finally processed by an imaging plate reader to get intensity profiles of photo-stimulated luminescence (PSL) corresponding to the diffusion profile of T. The obtained diffusion profiles of T have indicated that there are two diffusion paths, one is fast diffusion like hydrogen diffusion in pure vanadium and the other slow diffusion caused by tritium trapping of alloy elements.

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08-17

The Diffusion Behaviors of Interstitial Impurities in V-4Cr-4Ti Alloys under Ion Irradiation.

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Vanadium alloys are recognized as candidate materials for structural components of fusion reactors, because of their low activation and high strength at elevated temperatures. However, change of mechanical properties by formation of the precipitates under irradiation at elevated temperatures is a key issue. Recent studies have shown that the precipitation behaviors are affected by long range diffusion of oxygen. Therefore, the effect of diffusion of interstitial impurities such as oxygen and nitrogen on the precipitation behavior of Ti(O,N,C) is studied quantitatively in this paper.

Nine types of the V-4Cr-4Ti alloys with different oxygen and nitrogen contents, which were produced by NIFS, were used in this study. The interstitial impurity levels in these alloys were controlled. Oxygen concentrations were 44-921 wppm and nitrogen concentrations were 11-1070 wppm, respectively.

Irradiation was performed with 2.4MeV Cu²⁺ ions at 973K up to dose of 10dpa using a tandem type accelerator at Kyushu University. The hardness of irradiated specimen was measured by an ultra micro-indenter. Microstructure was also observed by using transmission electron microscopy (TEM). The concentration of impurities are estimated from number densities and size of the Ti(O,N,C), which were measured by TEM observations.

The total amounts of oxygen in the precipitates for 0.75, 3 and 10 dpa, which were estimated from the density and size of the precipitates, were higher than that of the matrix level before irradiation. In this irradiation condition, the flux of oxygen through the incident surface are estimated, and oxygen levels in specimens are calculated by finite difference method. In the cases of high purity specimens, oxygen diffused from incident surface into irradiated region. Moreover, oxygen diffuses from the back of the irradiated surface into irradiated region and enhanced the precipitations at the irradiated region. In the case of low purity specimens, which contained more than 1000wppm impurities, the precipitation is enhanced, and many impurities are trapped at Ti(O,N,C) precipitates. Therefore, the concentration of impurity in matrix decreased with increasing irradiation dose. These results suggest that the precipitation rates under ion irradiation are determined by initial impurity level and the flux of oxygen, which invades from the surfaces under irradiation.

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08-18

Dissolution of Hydrogen Isotopes into V-4Cr-4Ti Alloy

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The present authors have investigated the influence of heat treatment on hydrogen ingress into a V-4Cr-4Ti alloy and reported that the heat treatment at 1273 K led to significant reduction in the absorption rate of H₂. In the present study, surface analysis by X-ray photoelectron spectroscopy and additional absorption experiments for H₂ or D₂ were carried out to understand the mechanism underlying the reduction in the ingress rate and isotope effect.

The heat treatment of a V-4Cr-4Ti alloy in vacuum above 800 K resulted in the surface segregation of Ti. No significant redistribution of Cr was observed. The surface concentration of Ti reached to 10 and 20 at% at 973 and 1273 K, respectively. Absorption experiments for H₂ after heat treatments at 673, 973 and 1273 K showed the systematic reduction in the ingress rate with increasing surface Ti concentration. The detailed analysis of photoelectron spectra indicated that Ti was preferentially oxidized by impurity oxygen. The reduction in ingress rate of hydrogen was ascribed to the increase in surface oxygen coverage by preferential oxidation of segregated Ti. Results of ab initio calculations for systems comprising a small cluster of metal and oxygen atoms and a hydrogen molecule agreed with the experimental observations. The isotope effect between H₂ and D₂ was small and not observable under the present conditions. The absorption experiments for tritium were also carried out, and the influence of heat treatment on tritium distribution in the bulk of the alloy would be also presented.

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08-19

Helium in Irradiated Iron: a Multi-Scale Study

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Helium is commonly formed in structural materials exposed to neutron irradiation due to (n, α) reactions. Helium is quite mobile, but is trapped by vacancy-type defects in the bulk. This strongly affects radiation-induced microstructural evolution, e. g. giving rise to gas-bubble formation, swelling and degradation of mechanical properties. Predictive modeling requires detailed information on the properties and mechanisms in which He and radiation induced defects, such as vacancies, interstitial atoms and their clusters, are involved. Such information is unlikely to be obtained from purely experimental or theoretical methods and, we therefore suggest a multiscale modeling approach. Properties of small defect clusters, up to 3 He atoms associated with point defects, have been determined using ab initio methods. These results were used to create empirical many body interatomic potentials for the Fe-He system. The empirical potentials have then been used to study larger defects, containing tens of He atoms and point defects, via large-scale classical molecular dynamics (MD) simulation. The MD method permits the energy and dynamic properties of the defects to be studied on the scale of ~100 nm and 100 ns. The information obtained can be then used in cluster dynamics models and rate theory simulations of microstructural evolution under irradiation. Some of the results obtained are directly compared with experimental data on irradiated and He-implanted and annealed Fe.

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08-20

Thermal Activated Helium Induced Swelling of Beryllium Irradiated up to Fusion Reactor Relevant Doses

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The European Helium Cooled Pebble Bed (HCPB) blanket, selected as test module for ITER, incorporates beryllium as neutron multiplier to optimize tritium production in the lithium-based ceramic breeder. A key issue herewith is the dimensional stability of the HCPB blanket design in view of the expected helium induced swelling of the beryllium. Although swelling phenomena are widely recognized in materials under irradiation, the extent to what they act in beryllium at fusion reactor relevant conditions, i.e. in the wide range of blanket relevant temperatures lasting a considerable long time, is poorly known and not yet assessed experimentally.

Beryllium irradiated up to a high fusion reactor relevant dose (22,000 appm He) at low temperature (50 °C – hence with all generated helium still incorporated) has been investigated for its swelling behaviour upon long-term annealing at fusion reactor relevant temperatures (500 – 900 °C for up to 9 months). Particular attention has been paid in identifying saturation phenomena.

The helium was measured by a hot vacuum extraction technique and found to be released exponentially above 500 °C, total release being observed after annealing for 200 hours at 900 °C.

The induced swelling, measured by mercury pycnometry, was found to be characterized by a similar exponential increase at higher annealing temperatures, giving rise to a maximum saturation swelling of 37%. The empirical exponential correlation of the swelling with the temperature as derived formerly from restricted time and temperature experiments was confirmed.

Optical microscopic examinations revealed helium bubble formation to be the driving force for swelling: bubble parameters (mean radius and concentration) are assessed as a function of the annealing time and temperature and related to swelling.

The results of this study enlarge the available database for the improvement and validation of models to predict beryllium swelling up to the end of life conditions of blanket modules in a fusion power reactor.

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08-21

Radiation Enhanced Diffusion of Hydrogen in Insulating Materials Under Reactor Irradiation

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Oxide ceramics have potential for use as electrical insulator materials for burning-plasma diagnostics components as well as tritium breeding materials of blanket in nuclear fusion reactors. So far, it has been reported that the electrical properties of the insulating materials are dynamically changed by several radiation induced phenomena such as radiation induced conductivity (RIC), radiation induced electromotive force (RIEMF) and radiation induced electrical degradation (RIED). It will be predicted that the radiation induced phenomena are further enhanced by behavior of hydrogen isotopes trapped in the insulating materials during long term D-T discharge. Therefore it is extremely important to understand how hydrogen isotopes are transported in the insulating materials under irradiation.

In the present study, electronic and protonic conductions of typical proton conducting oxide ceramics ($\text{BaCe}_{0.9}\text{Y}_{0.1}\text{O}_{3-\delta}$), had perovskite-type crystal structure, were investigated under reactor irradiation at Japan Materials Testing Reactor (JMTR) in Oarai Research Establishment of Japan Atomic Energy Research Institute (JAERI). Two kinds of the specimens with and without H were prepared. Hydrogen was implanted into the zirconium, deposited on one side of the specimen, by 10 keV H_2^+ ion irradiation at 473 K. When thermal nuclear power was raised up to 50 MW, the temperature of the specimens with and without H increased up to 448-600 and 473-673 K, respectively, by gamma-ray heating of 1.1 and 2.0 kGy/s, respectively. The fast neutrons fluxes for the specimens with and without H were 6.84×10^{16} and 1.55×10^{17} n/m²s, respectively.

The electrical conductivities of the specimens with and without H during irradiation increased with the increase of the thermal nuclear power, and those at 50 MW were higher by about 3-4 orders of magnitude than that without radiation. The RIC for the specimen with H was higher by two orders of magnitude than that for the one without H. The result may show that the RIC takes place due to electronic excitation as well as hydrogen diffusion, enhanced by gamma-ray and neutron irradiations. Moreover, the RIC for the specimen with H at 50 MW decreased with increasing the fast neutron fluence and hereafter became a constant, while that for the one without H hardly changed. The decrement of the RIC is attributed to trapping of activated hydrogen at some defects, produced by neutron cascade collisions.

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08-22

Diffusion of He Interstitials and Small He Clusters at Grain Boundaries in α -Fe

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Grain boundaries generally provide fast diffusion paths for He atoms and small He clusters. Molecular dynamics methods have been employed to study the migration and diffusion mechanisms of He atoms and small He clusters in grain boundaries in α -Fe. Two grain boundaries, the $\Sigma 11\langle 110 \rangle \{323\}$, and the $\Sigma 5\langle 110 \rangle \{111\}$, were used for the current investigations. The low-temperature (about 0 K) equilibrium structures of these grain boundaries were determined using standard molecular dynamics relaxation techniques, with a flexible border condition. The lowest energy configurations of single He atom and small He clusters at the grain boundaries were determined by raising the lattice temperature to 1000 K and then slowly cooling down to 0 K. The migration of He atoms and small He clusters were followed for 10 – 30 ns, at temperatures between 600 and 1200 K. The diffusivity and self-diffusion coefficient of He atoms and small He clusters were obtained by the Guinan method and using the mean square displacements of He atoms. The effective migration energies were determined and compared with the energy barriers calculated using the nudged-elastic band method. Also, the lowest energy paths of He atoms and He clusters were traced out by the Dimer method. We found that He atoms and small He clusters diffuse very quickly in the $\Sigma 11$ GB, where the binding energy of a He atom is high, with one-dimensional behavior along specific directions, but a few directional changes were observed at higher temperatures. He atoms and small He clusters migrate one-dimensionally at low temperature, two-dimensionally at an intermediate temperature and three-dimensionally at higher temperature in the $\Sigma 3$ GB, where the binding energy of a He atom is low. These results suggest that the varying atomic structures of the grain boundaries are important for the diffusivity of He.

08-23

Helium desorption behavior in tungsten irradiated with low energy helium ions at high temperature

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Tungsten divertor plates in fusion reactors will be subjected to helium bombardment and large amount of helium will be implanted into the material. The trapped helium will be re-emitted into plasma by thermal desorption. In order to understand the behavior of helium in fusion reactor condition, thermal desorption from the wall materials irradiated at high temperature must be understood. In the present work, therefore, thermal desorption experiments for tungsten irradiated at high temperature were carried out.

The material used in the present work was high purity (99.95%) powder metallurgy tungsten. The specimens were annealed in a high vacuum and then electro-polished. Helium ions at 8 keV were irradiated at 1073 K. The ion fluence was in the range of 2×10^{21} - 1×10^{22} He⁺/m². Helium desorption behavior was studied by Thermal Desorption Spectroscopy (TDS). The ramp rate of the TDS was constant at 1 K s⁻¹ up to 2500 K.

In case of 2×10^{21} He⁺/m² irradiation, single wide desorption peak appeared between 1300 K and 1700 K, and the total helium desorption was about 1.8×10^{21} He⁺/m². While for the fluence of 4×10^{21} He⁺/m², the peak shifted to lower temperature side about 500 K (between 800 K and 1400 K), and the total helium desorption decreased to 7.8×10^{20} He⁺/m². When the fluence reached 1×10^{22} He⁺/m², helium desorption started at about 500 K (below irradiated temperature) and several fine peaks appeared up to 1700 K. The total helium desorption decreased further (4.5×10^{20} He⁺/m²). It is considered that surface modification such as blistering and exfoliation may change the retention and desorption of helium mentioned above.

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08-24

The Effects of Helium on Irradiation Damage in Single Crystal Iron

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Fusion systems are recognized to have a major issue with the relatively large amounts of helium (He) which are generated during the irradiation damage process. The effect of He on the accumulation of defects and defect cluster and the influence of the resulting microstructure on physical and mechanical properties has been the focus of a large number of experimental and modeling studies over the past twenty years. The present work is aimed at quantifying these effects in ways that were not possible in earlier studies. This is accomplished through systematic and coordinated computational modeling and experiments. The modeling approach employs both molecular dynamics (MD) and kinetic Monte Carlo (kMC) simulations to study the dynamic evolution of He and defect clusters in bcc iron over relevant time scales. The effects of incident ion energies, helium concentrations, and temperature on the evolution of defects including He, vacancies and clusters are simulated with MD. The MD simulations employ the advanced modified embedded atom method (MEAM) to describe

interatomic interaction in the iron-helium system. The defect configuration information from MD is then used in the kinetic Monte Carlo (KMC) simulations to study point defect diffusion and clustering. The kMC model follows the transport or evolution of the major defect entities in the material including, interstitial and substitutional helium, iron SIAs, vacancies, vacancy-clusters, and sinks for the trapping of point defects (dislocations and grain boundaries) and tracks the time evolution of defect clustering and bubble formation as a function of irradiation conditions, times and temperatures. It is found that almost all the transmuted helium is trapped in sink configurations (clusters, dislocations or grain boundaries) within a fraction of a microsecond. The experimental studies use ion implantation to simulate the radiation damage process over a range of He/dpa values and dose levels. The resulting microstructures are characterized using positron annihilation spectroscopy and TEM. These results are compared to the computational findings.

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08-25

Microstructure change and helium release due to tensile stress on austenitic stainless steel implanted low energy helium ion

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The plasma facing materials are subjected to low energy hydrogen isotopes and helium ash from the burning D-T plasma in the fusion device. Particularly, helium strongly affects on damage accumulation and mechanical properties due to the strong interactions with crystal defects and helium. In the present work, microstructure and helium release during tensile stress on 304 stainless steel irradiated with low energy helium have been investigated to examine behavior of helium embrittlement.

Specimen size is 2.8×5.4×0.1mm in gauge and 12.4mm in total length. Heat treatment for the specimens is conducted at 1323K for 30min. The specimens are irradiated with 8-keV helium ions at R.T., 573K and 873K up to the fluence of 3×10^{21} to 3×10^{22} He/m². Tensile tests are performed on the un-irradiated and the irradiated specimens at a strain rate of 3.33×10^{-3} s⁻¹. One specimen is tested to failure, one specimen loaded before the upper yield point, and remaining three specimens strained plastically at 10%, 20% 40% strain. After the tensile test, surface modification and microstructure change are observed with a scanning

electron microscope(SEM) and a transmission electron microscope(TEM), respectively. Nano-indentation hardness test is also carried out. In addition, thermal desorption spectrometry(TDS) is performed on the specimens before and after the tensile tests.

It is shown that surface modification on the irradiated specimens after the tensile test is drastically different from that of the un-irradiated specimens. For example, blisters with a diameter of about 500nm and exfoliation of them are formed on the specimen surface irradiated with 3×10^{21} He/m² at R.T. after the tensile test is stopped at 10% strain. When the irradiation temperature increases, a large number of cracks with a size of about several μ m are formed on the surface irradiated at 573K and 873K after the tensile tests. The yield strength on the specimens irradiated at R.T. and 573K also increases. The nano-indentation tests indicates that the hardness change near surface increases depending on the irradiation temperatures. It seems reasonable to suppose that helium irradiation results in embrittlement of the surface layer of 304 stainless steel. Present results indicate the importance of synergistic effect of helium irradiation and tensile stress on surface modification and erosion under the fusion environment.

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08-26

Evaluation of helium effects on ODS ferritic steels under ion irradiation

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The ferritic steels have been developed as the blanket structural material for fusion reactors. In the recent research of neutron irradiation at the HFIR, 9Cr and 12Cr ODS ferritic steels showed the irradiation hardening but no reduction of elongation after the irradiation at 300 °C up to 2.8 dpa, suggesting that the microstructural behavior and the hardening mechanism of ODS steels were different from those of the conventional ferritic steels.

In the present study, the microstructural evolution and irradiation hardening of 9 to 19Cr-2W ODS steels were investigated using a dual-ion irradiation method. The dual ion-irradiation experiments were performed at the DuET facility, Kyoto University using 6.4 MeV Fe self-ion and energy-degraded 1 MeV He ion. The irradiation temperature was from 300 to 700 °C. For the evaluation of helium effect, both dual-ion and single-ion irradiations were performed up to 20 dpa. The irradiated materials were characterized by transmission electron microscopy (TEM) and the nano-indentation method.

The nano-indentation investigation showed the irradiation hardening occurred at 300 °C, but slight hardening was observed at 500 °C. The TEM investigation for furnace-cooled 9Cr-ODS steel after dual-ion irradiation at 500 °C indicated that a number of cavities were observed in the helium-implanted region. In contrast, there was no observable cavity at the higher damage region over the range of helium implantation. The irradiation effects on high chromium ODS steels are also discussed focusing on the synergistic effects of helium and defect accumulation.

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08-27

Cavity formation in Iron and Eurofer-97 due to He Implantation and Neutron Irradiation

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The present contribution will give an overview of our experimental work on studies of the microstructure of iron and Eurofer-97 after He implantation and neutron irradiation.

In order to investigate the role of He in cavity nucleation in neutron irradiated iron and steel, pure iron and Eurofer-97 steel have been He implanted and neutron irradiated in a systematic way at different temperatures, to different He and neutron doses and with different He implantation rates. The resulting microstructures have been investigated primarily using Positron Annihilation Spectroscopy (PAS) as the main characterisation technique, taking advantage of its sensitivity to cavities in the size range from single vacancies to several nanometres. The temperatures used for the homogeneous He implantations and the neutron irradiations were 50°C and 350°C. Helium doses were 1, 10 and 100 appm with implantation rates covering the range of 0.0006 - 0.02 appm/s. Neutron irradiation doses were in the range of $10^{-3} - 10^{-1}$ dpa (50°C) and 0.1 - 0.3 dpa (350°C).

Like in the case of neutron irradiation, He implantation leads to the formation of nano-cavities both in pure Fe and in Eurofer-97 at 50°C as well as at 350°C. In favourable cases semi-quantitative estimates can be made of the cavity sizes and densities. For both materials at 50°C, He does not seem to enhance the cavity nucleation significantly compared to neutron irradiation to the same displacement damage level, while a clear, but modest effect is observed at 350°C. In the case of Eurofer-97, the cavity nucleation at both temperatures appears to be less efficient than that in pure iron, in particular for implantations at 50°C.

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08-28

Synergistic Effect of PKA and Helium in Fe-0.1 % He Matrix

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Helium effect in low activation ferritic/martensitic steels under 14 MeV intense neutron irradiation is an important and still unsolved issue for fusion reactor materials. The synergistic effect of primary knock-on atom (PKA) events and He on the irradiation induced damage in Fe containing 0.1 % interstitial He is investigated by molecular dynamics (MD) simulations with 10 cases statistics, for energies between 3 and 10 keV and temperatures between 10 and 523 K. The results show that (1) the vacancies produced by cascade are all in the PKA's range region to induce depleted zone, the helium does not influence the Frenkel pair production, (2) during the cascade evolution all recoil iron atoms and helium combine with each other to form Fe-He or Fe-Fe dumbbell interstitials as well as interstitial clusters; (3) the number and size of interstitial clusters increase with the energy of PKA, (4) the number and size of interstitial clusters increase with temperature, (5) a few large clusters consisting of a large number of iron interstitials and only a few helium atoms are produced, the rest being Fe-He clusters with small and medium sizes, (6) the dumbbell interstitials for Fe-Fe and Fe-He are in the <110> and <100> series direction respectively. These new results are compared with the damage produced in the same conditions in Fe, Fe containing of 2×10^{-4} % vacancies, Fe with 0.1 % substitution helium and annealing of Fe consisting 0.1 % He system. They confirm the new synergy existing between the PKA events and the He on the damage produced in Fe - 0.1 % He system.

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08-29

Behavior of Helium in Steel Cr12W2VTaB under Various Implantation Temperatures

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Transmission electron microscopy and helium thermodesorption spectrometry (HTDS) have been used to investigate the helium behavior and gaseous porosity formation peculiarities in low-activating ferritic-martensitic steel Cr12W2VTaB irradiated by 40-keV He⁺-ions up to a fluence of $5 \cdot 10^{20}$ m⁻² at temperatures of 270, 570, 690, 770 and 900 K in two initial states: normalizing annealing + tempering (HT-1); rolling + annealing at 1070–1080 K (HT-2).

It is found that after HT-1 the first bubbles with a maximal diameter d_{\max} of the order of 1 nm are formed at 770 K and large bubbles with $d_{\max} \approx 20$ nm and a mean diameter $\bar{d} \approx 8$ nm are formed at 900 K. On the contrary, the high density of the smallest bubbles are formed already at the temperature of 570 K after HT-2. At 900 K the bubbles have an extremely nonuniform distribution both in sizes and in the sample volume. Together with large faceted bubbles ($d_{\max} \approx 16$ nm and $\bar{d} \approx 7$ nm), local zones of small spherical bubbles ($d_{\max} \approx 4$ nm and $\bar{d} \approx 2$ nm) are observed.

In contrast to austenitic steels having only one main HTDS peak, thermodesorption spectra for steel Cr12W2VTaB have two main peaks owing to polymorphic transformation during heating. For temperatures up to 770 K, the helium desorption activation energy is equal to 2.0 ± 0.4 eV for the second peak. The HTDS peaks widen and gas release begins at lower temperatures with increasing the irradiation temperature.

The obtained data are discussed in terms of the initial structural-phase influence on helium behavior in ferritic-martensitic steel under various ion-implantation temperatures.

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08-30

Dynamic Monte--Carlo modeling of hydrogen isotope reactive-diffusive transport in porous graphite

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Deuterium and tritium will be the fuel used in a fusion reactor in equal mixtures. It is important to study the recycling and mixing of these isotopes of hydrogen in graphite from several points of view:

- (i) impact on the ratio of deuterium to tritium in a reactor,
- (ii) continued use of graphite as a first wall and divertor material,
- (iii) reaction with carbon atoms and the transport of hydrocarbons will provide insight into chemical erosion.

The graphite used in fusion devices as a first wall material is porous and consists of granules and voids. These granules are typically 1-10 micrometer separated by voids which are typically a fraction of a micrometer. The granules consist of graphitic micro-crystallites of size 10-100 nm separated by micro-voids which are typically one nm.

These sub-structures, voids and micro-voids provide a large internal surface area inside graphite where the interstitial atoms can diffuse and react with each other which will affect the hydrogen isotope inventory and recycling behaviour and also chemical erosion.

We use dynamic Monte-Carlo techniques to study the reactive-diffusive transport of hydrogen isotopes and interstitial carbon atoms in a 3-D porous graphite structure irradiated with hydrogen and deuterium and compare with published experimental results for isotope exchange and chemical erosion.

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Computer Simulation of Defect Accumulation Processes in Tungsten under Low Energy Helium Ion Irradiation

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In fusion reactors, plasma-facing materials suffer strong bombardment of plasma particles such as hydrogen (H) isotopes and helium (He) in addition to D-T neutrons. It has been demonstrated that irradiation effects of He is much significant than that of H, but detail mechanism of defect accumulation under He ion irradiation has not been clarified.

For transmission electron microscopy (TEM) observation in W at 293K under irradiation with 8 keV He ions, interstitial loops appear and increase with increasing dose. The density saturate at dose level of around 2.6×10^{19} ions/m². After the dose, the loops pile up as tangled dislocations.

In this work, accumulation of lattice defects is calculated by using rate theory to explain the evolution of interstitial loop under He ion irradiation.

In a calculation for evolution of interstitial loops under high-energy electron beam or H irradiation, it is considered that a nucleus formation, di-interstitial atoms (I₂), of interstitial loop is formed by interstitials with the thermal activation.

However, calculation results only considering that nucleus formation mechanism cannot quantitatively and qualitatively explain the evolution of interstitial loop under He ion irradiation.

It has been shown experimentally that vacancies containing about 7 He atoms are unstable and mutate to di-vacancies as following reaction;



where He is a interstitial helium, I is a self-interstitial and He_{n+1}V₂ is a complex of nHe atoms and di-vacancies. Assuming that the mutation produced interstitial (MPI) remains bound to the mutated complex with some binding energy E_b, it is expected that MPI captures another interstitial atom and forms a nucleus of interstitial loop.

Interstitial loop density obtained from the calculation depends on E_b strongly. The case in which a value of E_b is comparatively low, the density is much lower than that of the experimental value by TEM observation. The case in which a value of E_b is comparatively high, The density is much higher than that of the experimental value. In the case of E_b=0.65eV, the experimentally observed time evolution of interstitial loop can be explained well quantitatively and qualitatively.

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08-32

Mechanisms of Retention and Blistering in Near-Surface Region of Tungsten Exposed to High Flux Deuterium Plasmas of Tens of eV

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Tungsten plates of 99.99% purity annealed at 1200°C were exposed to deuterium plasmas with incident energies ranging from 7 to 98 eV and a fixed flux of 10^{22} D/m²/s, resulting in retention of a large amount of deuterium and blistering of tungsten in the near-surface region. Why could the amount of deuterium retained in the surface region reach many tens of orders greater than both deuterium solubility and equilibrium vacancy concentration? And why could blistering occur in annealed tungsten exposed to deuterium plasmas with energies much smaller than that required to generate displacements?

To reveal the mechanisms of retention and blistering, the behavior of deuterium trapping and the formation of hydrogen isotopes-induced vacancies in the near-surface region of tungsten were studied with a variety of techniques, such as X-ray diffraction (XRD), transmission and scanning electron microscopy (TEM and SEM) and thermal desorption spectroscopy (TDS). The results of small angle XRD at a fixed incident angle of 1.5° indicated that within the

experimental error there was a zero change in the lattice parameter after the plasma exposure. This implies that deuterium does not exist in the lattice interstitial sites, but instead forms a deuterium-vacancy complex and then clusters and further bubbles (deuterium molecules in vacancy clusters and voids) in near-surface region. Cross-sectional TEM observations showed that small blisters with a diameter of around 30 nm and nano-cracks formed in the near-surface region before the formation of larger blisters with diameters of up to a few microns (comparable to grain size). The TDS results strongly indicated that deuterium existed in the molecular form in tungsten after the plasma exposure. The retention of deuterium in the near-surface region is being investigated with nuclear reaction analysis (NRA) and elastic recoil detection analysis (ERDA), and the formation of hydrogen isotopes-induced vacancies in the surface region of tungsten will be further confirmed directly by positron lifetime and Doppler broadening measurements with slow positron beams. In conclusion, crystal defects like vacancies should be generated due to lowering of the formation energy of vacancies by the intrusion of a great number of hydrogen isotope atoms into the near-surface region of tungsten.

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08-33

Atomistic Modeling of the Interaction of He With Interfaces in Fe

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Structural materials of a fusion power system will be exposed to high concentrations of He produced from nuclear transmutation reactions. Helium is essentially insoluble in metals so there is a strong tendency for it to form bubbles that can significantly degrade mechanical properties. A strategy to effectively manage He is to provide a high-density of internal interfaces to serve as He bubble nucleation sites and vacancy-interstitial recombination centers. Nanostructured ferritic alloys are being developed to provide improved creep strength and He management capability compared to conventional steels. A key characteristic of these materials is the high-density ($\sim 10^{24}$ m⁻³) of nanometer-scale (~ 3 nm diameter) Ti-Y-O clusters.

We describe molecular dynamics simulations using Finnis-Sinclair interatomic potentials to assess the interaction of He atoms, vacancies and He-vacancy complexes with grain boundaries and nanoclusters in Fe. The potentials were adjusted to explore the effect of differences in lattice parameter and elastic properties of the nanoclusters relative to the Fe matrix on He trapping efficiency. The relative effectiveness of coherent compared to semicoherent interfaces is examined. Substitutional He atoms are moderately bound a representative set of grain boundaries and to positive misfit coherent nanoclusters that are elastically stiffer than Fe. Interstitial He was more strongly bound to these interfaces. The binding energy roughly correlates with excess volume.

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08-34

08-34 Application of the Master Curve to Inhomogeneous Ferritic/Martensitic Steel

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The ferritic-martensitic steel F82H is a primary candidate low-activation material for fusion applications, and it is being investigated in the joint U.S. Department of Energy-Japan Atomic Energy Research Institute collaboration program. As a part of this program, the master curve methodology was applied to obtain ductile brittle transition temperature (DBTT) of the IAE heat of ferritic/martensitic F82H steel. Three point bend (3PB) and compact tension (CT) specimens were studied in a variety of sizes. The smallest specimen was a 1.6x3.3 mm cross-section 3PB specimen, while the biggest specimen was a 1T CT. It was shown that scatter of fracture toughness data in the transition range was somewhat larger than proposed by tolerance bounds from conventional master curve analysis but similar to what other researchers reported for this steel. It suggests that this material may exhibit inhomogeneity of transition fracture toughness properties compared to conventional homogeneous ferritic steels for which the current master curve methodology was developed. The evolution of conventional master curve analysis to treat randomly inhomogeneous data set is discussed using these data. Statistical analysis of these data confirms inhomogeneity of transition region fracture toughness of F82H steel. It is shown that the tolerance bounds developed using random inhomogeneity provide better representation of the actual scatter of data than that of conventional analysis, while T_0 values derived by both methods are different by only several degrees C.

This inhomogeneity was confirmed by the microstructural analysis on F82H-IEA. It revealed the inhomogeneous distribution of complex inclusion (Al_2O_3 -Ta(V,Ti)O) which tends to be the fracture initiation point of specimens failed with low toughness values.

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08-35

In-reactor Creep Rupture Properties of Ferritic-martensitic Stainless Steel

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The ferritic-martensitic stainless steel developed by JNC (PNC-FMS) is the candidate core materials for the long-life fast reactor. PNC-FMS is mainly applied to the wrapper tube and cladding materials of the advanced fuel assembly. The main chemical composition is 11Cr-0.12C-2W-0.2V-Mo, Nb, which is close to the reduced activation ferritic-martensitic steels in the blanket system of the DEMO fusion reactor. The final heat-treatment for normalizing and tempering is different in wrapper tube and cladding to satisfy the required properties: 1323 K x 10 min and 973 K x 1 h for wrapper and 1373 K x 10 min and 1053 K x 1 h for cladding. The normalizing and tempering temperatures are higher for the cladding tube to improve the high temperature creep rupture strength. The out-of-reactor and in-reactor data including swelling, irradiation creep, tensile and Charpy impact properties have been acquired up to maximum neutron dose of 150 dpa, in order to make material design standard. These data were reported elsewhere.

Our primary concern is in-reactor creep rupture properties of the ferritic-martensitic stainless steel from the design viewpoint. In the case of austenitic stainless steel (PNC316), it was found that in-reactor creep rupture life is significantly degraded, compared with out-of-reactor tests, due to accelerated recovery of dislocation during irradiation. Therefore, the helium-pressurized specimens of PNC-FMS were manufactured, and the in-reactor creep rupture test was conducted using Material Open Test Assembly (MOTA) in FFTF. The test temperatures are at 823 K, 878 K, 943K and 1023 K. The neutron dose and exposure duration are up to 40 dpa and 7,000 h, respectively.

As the result of tests, in-reactor creep rupture life of PNC-FMS was revealed to be completely same as out-of-reactor life. It was demonstrated that there is no degradation in the creep rupture life during irradiation. It is supposed that dislocation structure and precipitates are significantly stable in PNC-FMS even under irradiation.

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08-36

Mechanical Properties and Microstructure of China Low Activation Martensitic Steel Compared with JLF-1 JOYO-II HEAT

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Researches on RAFMs (Reduced Activation Ferritic / Martensitic steels) are carried out worldwide in recent twenty years and have made some inspiring progress. In China, the China Low Activation Martensitic steel (CLAM) has been developed, which is based on the nominal compositions of 9Cr1.5WVTa. Some studies on CLAM steel with small ingots of a few kilograms have been done in China. Recently, several ingots of 20 kg were produced by vacuum induction furnace. Then the ingots were forged and rolled into bars and plates. The heat treatments was normalizing at 980°C for 30 minutes, quenching and then tempering at 760°C for 90 minutes. The mechanical properties of CLAM steel (FDS-HEAT 0408B) were compared with those of JLF-1 (JOYO-II HEAT) by tensile and impact tests. Because of the limitation of irradiation volume, the small specimen techniques were required and employed in this paper. The tensile results of CLAM showed the ultimate strength and yield strength were 670MPa and 512MPa at room temperature, and 373MPa and 327MPa at 600°C, respectively, which were higher than those of JLF-1. The Ductile-Brittle Transition Temperature (DBTT) of CLAM was -115°C by tests with 1/3 size Charpy V-notch specimens, and was lower than those of some other RAFMs. The microstructural analysis by SEM and TEM indicated that the prior austenite grain size and lath width for CLAM were smaller than that for JLF-1. The finer grain and lath structure is considered as one of reasons for the better strength and DBTT for CLAM.

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08-37

Creep Behavior of Reduced Activation Ferritic/Martensitic steels Irradiated at 573 and 773 K up to 5 dpa

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Reduced Activation Ferritic/martensitic steel (RAFs) is the most promising candidate for blanket structural material in fusion reactor. It was anticipated that irradiation creep of RAFs at lower temperature could be significant as it was in austenitic steels, and this would have large impact on the life limit of water cooled blanket system. But irradiation creep behavior of RAFs at lower temperature below 673 K had not yet been reported.

In this work, irradiation creep behavior of RAFs irradiated up to 5 dpa at 573 and 773 K at various hoop stress was reported. The materials used are F82H IEA heat (8Cr-2WVTa), JLF-1 (9Cr-2WVTa), JLF-1 Mn doped alloys and JLF-1 B doped alloy. Pressurized tubes were fabricated with hoop stress conditions ranging from 0 to 400 MPa at irradiation temperatures. Irradiation was performed in the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor (HFIR) up to 5 dpa in the removable beryllium (RB) position. Nominal irradiation temperatures were 573 and 773 K. Creep strain was calculated from tube diameter measured before and after irradiation. The diameter measurements were performed using a laser profilometry system.

At 573 K, irradiation creep was observed in all tubes depend on its hoop stress level. Creep strain for all steels shows linear dependence on the applied hoop stress up to ~250 MPa, but shows larger creep strain than this tendency at the highest stress level (400MPa). The detailed discussion will be presented along with the results of irradiation creep at 773 K and thermal creep at irradiation temperatures with creep tubes.

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08-38

Numerical investigation by finite element simulation of the ball punch test: application to tempered martensitic steels

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Over the years, the small ball punch test technique has been used to evaluate conventional tensile properties of a variety of materials. The development and use of this type of small specimen techniques is indispensable for an efficient use of the limited irradiation volume of the future fusion material intense neutron source. Up to now, empirical correlations between features of the load-displacement curves of the ball punch test and the mechanical properties, such as the yield stress or the ultimate tensile stress, are established on materials in the unirradiated condition. These correlations are believed to be applicable to irradiated materials and they have been very often used to estimate the irradiation hardening. However, it is well known that the overall constitutive behavior of the materials is generally affected by neutron irradiation. Therefore, there is a need to quantify the effect of the constitutive behavior on the correlations. In this paper, we employ a 3D non-linear finite element model for the ball punch test to address these effects of the irradiation-induced changes on the ball punch test curve. We apply first the model on the tempered martensitic steel EUROFER97 in the unirradiated condition with variations in the post-yield behavior, either in the low strain domain (<10%) or in the high strain domain (>10%). The effects on the ball punch test load deflection curve are outlined. Second, we study the effects of the irradiation hardening on the same constitutive behaviors as those used for the unirradiated condition. We show that that the usual correlations must be considered with great care on irradiated materials since strong variation on the strain-hardening may lead to erroneous estimation of the irradiation-hardening. We also propose a novel approach to calibrate the yield stress to features of the ball punch test curve that decreases the uncertainty related to the post-yield behavior and that, as a consequence, makes the technique more reliable for irradiated materials.

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08-39

Interface reactions and control of diffusion at the interface between SiC fibres and EUROFER 97

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EUROFER 97 is the candidate structural material for the first wall of future fusion reactors. The use of this steel would currently limit the maximum operation temperature to 550°C due to its loss in creep strength at higher temperatures. To overcome this limitation, we propose to reinforce EUROFER 97 with SiC long fibres. The idea is to build a metal matrix composite by hot isostatic pressing of EUROFER 97 with SiC fibre reinforcement. This novel SiC/EUROFER 97 composite should allow an increase of the operation temperature up to at least 750°C.

The aim of the present activity is to develop a thermally stable interface between the SiC fibre and the steel matrix. Diffusion in such metal matrix composites can lead to the formation of undesirable compounds in the interface region between fibre and matrix, therefore an appropriate diffusion barrier between fibre and steel is necessary. In our case the main problem is carbon diffusion from the outer fibre coating. The use of Cr, W and Re as diffusion barrier layers was investigated by measuring the diffusion of C into the EUROFER. Cr and Re exhibited an insufficient barrier effect resulting in a large C contamination in the EUROFER after heat treatment at 750°C. In addition the Cr interlayer completely dissolved and significant amounts of Re diffused into the EUROFER. In contrast the W interlayer and Re/W multilayer barriers proved to be much more efficient diffusion barriers resulting in a negligible C and W contamination in the EUROFER layer even after annealing at 750°C. Nevertheless the formation of Fe₃W₃C and α-W₂C was observed in the layers.

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08-40

Dynamical Interaction of helium bubbles with Phase Boundaries or Grain Boundaries in Fe-Cr Ferritic Alloys

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In plasma faced materials, helium bubbles frequently precipitate at boundary interfaces and their correlated effects with radiation induced precipitation of the alloy elements cause complicate grain boundary embrittlement. Therefore, study of dynamical interaction and development of helium bubbles with grain boundary or phase boundary interface is very important for the development of fusion reactor materials. However, only a limited information about this subject is available at present. In the present work, therefore, in-situ TEM study has been performed to reveal dynamical behavior of helium bubbles and their interactions with grain boundaries and alpha- gamma phase boundaries in Fe Cr ferritic alloys.

Small size (several nm) helium bubbles were introduced in Fe-Cr based alloys by irradiation with 10keV helium ions, using a TEM system which was connected with an ion accelerator. Then, the specimen was warmed up to 1200K and their dynamical behavior was continuously monitored by TEM and recorded on video frames.

Trapping of helium bubbles at grain boundaries and their easy motion along grain boundaries were observed at high temperature. It was revealed that most of small helium bubbles were swept out through alpha-gamma phase boundaries during the propagation of the transformation at around 1130K. Similar weeping out of helium bubbles through moving grain boundaries was also observed during the re-crystallization of the specimen.

To reveal a correlation between the bubble movement and microstructure change of grain boundaries or phase boundaries, thermal desorption of helium from the specimen was measured by quadru-pole mass spectrometer and compared with the results of TEM observation.. The big peaks of the helium desorption were observed and respectively correlated to the micro structure changes of the phase transformation and re-crystallization.

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08-41

Hydrogen Transport and Trapping in EUROFER'97

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The gas-phase permeation technique has been used to investigate the interaction of hydrogen with the reduced activation ferritic-martensitic steel EUROFER'97. The measurements were performed over the temperature range 376 – 724 K with hydrogen driving pressures ranging from $2.5 \cdot 10^4$ to $1.5 \cdot 10^5$ Pa. The high hydrogen pressures used have forced the interstitial atomic diffusion to be the gas controlling transport mechanism over any surface effect. The evolution of the hydrogen flux curves with time has been modeled over the non steady-state and the steady-state permeation regimes. The resultant diffusive transport parameters are a diffusivity of $D \text{ (m}^2\text{s}^{-1}\text{)} = 4.57 \cdot 10^{-7} \exp(-22.3 \text{ (kJ mol}^{-1}\text{)}/RT)$, a permeability of $\Phi \text{ (mol m}^{-1}\text{Pa}^{-1/2}\text{s}^{-1}\text{)} = 1.03 \cdot 10^{-8} \exp(-37.4 \text{ (kJ mol}^{-1}\text{)}/RT)$ and a Sieverts' constant of $K_s \text{ (mol m}^{-3}\text{Pa}^{-1/2}\text{)} = 2.25 \cdot 10^{-2} \exp(-15.1 \text{ (kJ mol}^{-1}\text{)}/RT)$.

The wide range of temperatures has allowed a precise characterization of the hydrogen trapping phenomenon occurring at low temperatures in the micro-structural defects of the material. The decrease in diffusivity and the increase in Sieverts' constant, i.e. solubility, induced by trapping have been accounted and modeled. The resultant trapping parameters have been a trap density of $N_t/N_i = 2.56 \cdot 10^{-5}$ and trapping energy of $E_t = 43.2 \text{ kJ mol}^{-1}$. The possible sources of trapping are analyzed and the most probable current mechanism in EUROFER'97 postulated. All the hydrogen transport parameters obtained for EUROFER'97 are compared to the available data corresponding to several steels of the same kind.

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08-42

A Critical Stress-Critical Area Statistical Model of the $K_{Jc}(T)$ Curve for MA957 in the Cleavage Transition

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We modeled the previously reported temperature (T) dependent fracture toughness $K_{Jc}(T)$ database for MA957 with a statistically modified critical stress-critical stressed area (σ^* - A^*) concept based on finite element (FE) simulations the area (A) encompassed by specified normal stress contours (σ). A statistically mediated range of A^* was recognized in our model, corresponding to the intrinsic distribution of K_{Jc} : thus the point at which the $A(\sigma)$ at various T experience the maximum number of intersections was used to define σ^* . The $K_{Jc}(T)$ of MA957 is strongly dependent on the specimen orientation. Analysis of cleavage initiation in the L-R orientation, with the highest K_{Jc} yielded the highest $\sigma^* \approx 3600$ MPa. In contrast, the σ^* for the C-L orientation, with the lowest K_{Jc} , yielded the lowest $\sigma^* \approx 2850$ MPa, while for the C-R orientation with intermediate K_{Jc} , $\sigma^* \approx 3000$ MPa. In the latter two cases, the ligament planes contain directions parallel to the extrusion direction, which also textured in a way that promotes cleavage. The corresponding A^* ranged from ≈ 30 to $400 \mu\text{m}^2$. The σ^* and A^* are controlled by the distribution of cleavage initiation sites primarily in the form of μm -scale Al_2O_3 particles aligned in the extrusion direction and the orientation dependent toughness of the ferrite matrix. The A^* - σ^* , was used to model median $K_{Jc}(T)$ and the corresponding curves at high and low fracture probabilities were determined from a Weibull analysis. The model is in good agreement with previously measured K_{Jc} data, but requires a K_{min} of $10 \text{MPa}\sqrt{\text{m}}$ in the C-L orientation, which less than the standard Master Curve (MC) value of $30 \text{MPa}\sqrt{\text{m}}$. We conclude that the low toughness direction is due to both intrinsic crystallographic and microstructurally mediated factors. The implications of these results to development of high toughness nanodispersion strengthened ferritic alloys is briefly discussed.

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08-43

Ductile Brittle Transition Behavior of F82H After High Concentration He Implantation at 550C

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The He effects on microstructural development and mechanical properties of reduced activation ferritic/martensitic steels have been studied by neutron irradiation experiments of B or Ni doped specimens, or by He implantation technique with accelerators. These results showed that the ferritic/martensitic steels had enough resistance to microstructural changes by irradiation and He implantation, and DBTT shift caused by irradiation mainly depended on the irradiation hardening. However, the effect of fracture behavior after irradiation at higher temperature region (500-600C) had not been clarified yet, because irradiation hardening was small in the temperature range. In this work, to study the He effects on the mechanical property changes of reduced activation steel after higher temperature irradiation, He-ion implantation at around 550C was performed and the fracture behavior of He implanted specimens was analyzed.

Mini size charpy specimens (1.5CVN) were prepared from a F82H IEA heat. Helium implantation was performed by a cyclotron of Tohoku University with a beam of 50MeV α -particles at temperature around 550C. A tandem-type energy degrader system was used to implant He into the specimen homogeneously from the surface to the implanted range of 50MeV α -particles, that was about $380 \mu\text{m}$. Implanted He concentration was about 1000appm. Charpy impact test was performed using an instrumented impact test apparatus of Oarai branch of IMR, Tohoku University. Analyses of absorbed energy change and fracture surface were carried out. Vickers hardness test was also carried out on the He implanted area of the 1.5CVN specimen to estimate irradiation hardening.

Analysis of absorbed energy showed that DBTT increase by the 1000appm He implantation at around 550C was about 80C. Temperature dependence of absorbed energy curve was rather complicated compared to unirradiated specimen. Grain boundary fracture surface was only observed in the He implanted area of all the ruptured specimens in a brittle manner. Irradiation hardening was also detected in the He implanted area. The increase of Vickers hardness after the He implantation was about 20. Microstructural observation will be presented and Helium effect on the fracture behavior will be discussed based on these experimental results.

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08-44

The Influence of Thermal Parameters on Thermal-Fatigue Resistance of Reduced Activation Martensitic Steels

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A systematic, experimental investigation concerning the thermal-fatigue resistance of candidate alloys for structural application in a MFR machine (Magnetic Fusion Reactor) initiated at the Casaccia Laboratories some ten years ago. Initially, a comparison was performed between six martensitic steels, testing either reduced activation or conventional nuances, and an austenitic steel, AISI 316 L(N). Although just a single temperature envelope was used (from 200 to 600°C), the results of that campaign were quite interesting and somewhat unexpected, showing that the resistance to a thermally induced loading status was not driven by the so-called thermal factor only.

The second step of the investigation was addressed to the study of thermal parameter effects on the lifetime of two candidate reduced activation steels, the modified F82H and the Eurofer 97. Both alloys have been tested in temperature ranges from 150-450°C to 250-650°C and using a heating-cooling rate of 5°C/s. Measured lifetimes for tests carried out without hold times lay between 400 and 4300 cycles.

Data analysis showed that the number of cycles to failure can be estimated using a "constraint factor", a semi-empirical quantity given by the product of the (theoretical) hindered thermal expansion and the difference between the monotonic tensile strength of the material measured at the highest and lowest temperature of the thermal cycle.

A few, preliminary tests were performed on Eurofer 97 introducing hold times (up to 1000 seconds at the highest temperature) or using different heating-cooling rates. The effects of these variables are discussed in the presented paper.

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08-45

The Heat Resistant RAFMS RUSFER-EK-181 for Fusion and Fast Breeder Power Reactors Applications

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The R&D results of the Russian manufacture of the reduced activation ferritic-martensitic steel (RAFMS) RUSFER-EK-181 (Fe-12Cr-2W-V-Ta-B-0.16C type) are presented. The steel is settled by Russian industry as heat resistant (the temperature window of the applications is up to 650-700 °C) structural material for active cores of the fusion (DEMO-RF and beyond) and the fast breeder (BN-600, BN-800) power reactors. The industrial heats of the RUSFER-EK-181 and the goods (sheets, rods, tubes) were manufactured. The regimes of the heat treatments for the optimization of the structure-phase states of steel are defined. The chemical compositions and functional properties of the obtained heats and goods fits with corresponding Russian engineering requirements to structure materials for the nuclear reactor cores applications.

Short-term mechanical properties of steel after the standard heat treatment are provided in temperature range -196 °C ÷ +700 °C. Long-term strength and creep of steel at 650 °C and 700 °C with respect to its dependence on the different heat treatment regimes are studied.

Steel specimens were irradiated in the Russian fast breeder reactor BOR-60 (doses up to 10 dpa, irradiation temperatures 320-330 °C). The propensity of steel for low temperature irradiation-induced embrittlement (DBTT shift) essentially depends on regimes of initial heat treatments.

The functional properties and the technology problems (sheets, rods, tubes, welding) of the RUSFER-EK-181 for the assessment of the feasibility of the steel as structural material for the test module DEMO-RF in ITER, the DEMO-RF reactor and the fast breeder power reactors applications are discussed.

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08-46

Preliminary Experimental Investigation on Hot Isostatic Pressing Diffusion Bonding for CLAM

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China Low Activation Martensitic (CLAM) steel is under development in ASIPP (Institute of Plasma Physics, Chinese Academy of Sciences). The design of FDS series fusion reactors and corresponding Test Blanket Module (TBM) is carrying out in ASIPP. In the design, CLAM is selected as the primary candidate material. Thus, the research of the hot isostatic pressing (HIP) bonding technique on CLAM/CLAM, which is one of the primary candidate bonding techniques for manufacturing of the first wall of a fusion reactor, is greatly needed.

The preliminary HIP diffusion bonding experiment on CLAM has been performed. A few machining approaches such as dry-milling, turnery and grinding etc. were used to prepare the sample surfaces and then they were degreased with a mixture of alcohol, ether and acetone in an ultrasonic bath. The samples were joined by HIP diffusion bonding with the compression pressure of 150MPa and the holding time of 2-3h under different temperatures between 950°C and 1100°C. And appropriate post heat treatment was done.

Tests on mechanical properties of the joints such as tensile strength and impact toughness have been performed. Microstructure of the joints was studied by optical microscope, SEM and TEM. Through analysis of the results, optimized parameters for HIP are given. The further studies on the HIP techniques for CLAM will be carried out based on these results and the optimized parameters of HIP.

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08-47

Evaluation of Fracture Toughness Master Curve Shift of JMTR Irradiated F82H Using Small Specimens

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So-called deformation and fracture minibeam (DFMBs), with typical dimensions of 1.65 x 1.65 x 9.2 mm³, are the world's smallest pre-cracked specimens ever used to measure 'fracture toughness', occupying only 1/8 the volume of still very small 1/3 sized pre-cracked Charpy (1/3PCC) bend bars with typical dimensions of 3.3 x 3.3 x 18.3 mm³. Both the 1/3PCC and half thickness variants (1/3HTPCC) specimens have been previously used in a number of irradiation experiments, including in the recent US-Japan HFIR program focusing on both vanadium alloys and reduced activation normalized and tempered martensitic steels (TMS). DFMBs, that were also included in these experiments, have previously been characterized in the unirradiated condition, but not following irradiation. Due to massive constraint loss, and their corresponding very steep transition-toughness temperature curves, the DFMBs are primarily intended to measure shifts (ΔT_d) in the cleavage transition temperature under (primarily) dynamic loading conditions, but not fracture toughness or Master Curve (MC) reference temperature shifts (ΔT_o) per se. Thus irradiations in the Japan Materials Testing Reactor (JMTR) of the IEA program heat of F82H to low doses between 0.04 and 0.12 dpa were carried out at 290°C in order to further qualify these small specimens test methods, as well as to add to the TMS irradiated property database. The JMTR irradiations also included SSJ2 type sheet tensile specimens (4 x 16 x 0.5 mm³) at doses of 0.04 and 0.12 dpa. The post irradiation tests were carried out at the IMR-Oarai Center, Tohoku University. The 1/3PCC were tested statically, yielding ΔT_m shifts estimated from the measured K_{Jm} of $\approx 25 \pm 20$ and 45 ± 20 , respectively. Preliminary adjustments were carried out on the K_{Jm} data to small scale yielding conditions at a reference thickness of 25.4 mm, K_{Jr} , accounting for both statistical and constraint loss effects, using procedures described elsewhere. The unirradiated F82H fell about 10 MPa \sqrt{m} below the previously reported median MC for F82H with a $T_o \approx -100$. The corresponding estimated ΔT_o shifts of $\approx 30 \pm 20$ and 46 ± 20 °C were only slightly larger than the ΔT_m . The changes in room temperature σ_y ($\Delta \sigma_y$) were 50 ± 15 and 74 ± 15 MPa. The corresponding $C_o = \Delta T_o / \Delta \sigma_y$ of 0.63 ± 0.01 is in good agreement with other data at higher doses, especially accounting for a smaller loss of strain hardening in the low dose JMTR data. The ΔT_d derived from dynamic DFMB tests was 30 ± 20 °C at 0.1 dpa (nominal), again in good agreement with the estimated ΔT_o shifts. We also show that the ΔT_d and ΔT_o are in excellent agreement with a $\Delta T_o = C_o \Delta \sigma_y = C_o h(dpa, T_i)$ model where the $h(dpa, T_i)$ was found by fitting a large database on tensile properties.

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08-48

A Critical Stress-Critical Area Statistical Model of the $K_{Jc}(T)$ Curve for MA957 in the Cleavage Transition

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We modeled the previously reported temperature (T) dependent fracture toughness $K_{Jc}(T)$ database for MA957 with a statistically modified critical stress-critical stressed area (σ^* - A^*) concept based on finite element (FE) simulations the area (A) encompassed by specified normal stress contours (σ). A statistically mediated range of A^* was recognized in our model, corresponding to the intrinsic distribution of K_{Jc} : thus the point at which the $A(\sigma)$ at various T experience the maximum number of intersections was used to define σ^* . The $K_{Jc}(T)$ of MA957 is strongly dependent on the specimen orientation. Analysis of cleavage initiation in the L-R orientation, with the highest K_{Jc} yielded the highest $\sigma^* \approx 3600$ MPa. In contrast, the σ^* for the C-L orientation, with the lowest K_{Jc} , yielded the lowest $\sigma^* \approx 2850$ MPa, while for the C-R orientation with intermediate K_{Jc} , $\sigma^* \approx 3000$ MPa.

In the latter two cases, the ligament planes contain directions parallel to the extrusion direction, which also textured in a way that promotes cleavage. The corresponding A^* ranged from ≈ 30 to $400 \mu\text{m}^2$. The σ^* and A^* are controlled by the distribution of cleavage initiation sites primarily in the form of μm -scale Al_2O_3 particles aligned in the extrusion direction and the orientation dependent toughness of the ferrite matrix. The A^* - σ^* , was used to model median $K_{Jc}(T)$ and the corresponding curves at high and low fracture probabilities were determined from a Weibull analysis. The model is in good agreement with previously measured K_{Jc} data, but requires a K_{min} of $10 \text{ MPa}\sqrt{\text{m}}$ in the C-L orientation, which less than the standard Master Curve (MC) value of $20 \text{ MPa}\sqrt{\text{m}}$. We conclude that the low toughness direction is due to both intrinsic crystallographic and microstructurally mediated factors. The implications of these results to development of high toughness nanodispersion strengthened ferritic alloys is briefly discussed.

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08-49

Tensile and transient burst properties of advanced ferritic/martensitic steel (PNC-FMS) claddings after neutron irradiation

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The ferritic/martensitic (F/M) steels are expected to be prospective not only for the long life core material of fast reactors but also for the blanket materials of fusion reactor because of their superior swelling resistance. Japan Nuclear Cycle Development Institute (JNC) has developed a 11Cr-0.5Mo-2W-V, Nb F/M steel (PNC-FMS) for core materials of next fast reactor. The focus of this study is to evaluate the effect of neutron irradiation on mechanical properties of PNC-FMS cladding tubes, especially tensile properties, transient burst properties and ductility loss caused by irradiation embrittlement under higher fluence and temperature conditions.

The PNC-FMS cladding tube samples were irradiated at temperatures between 773 and 1013 K to fast neutron fluences ranging from 11 to 102 dpa in the experimental fast reactor JOYO. Post irradiation tensile tests were carried out at a strain rate of 0.5×10^{-4} /s which was changed to 1.3×10^{-3} /s after yielding. Test temperatures were 773-1013 K in accordance with the irradiation temperatures. Temperature-transient-to-burst tests (TTB tests) were also conducted on the irradiated cladding samples. Samples were internally pressurized by high-purity argon gas, and then were heated linearly to rupture in order to study the rupture strength under ramp heating. The heating rate was 5 K/s and hoop stress conditions were 49, 98, 120 and 196 MPa.

The results of tensile and TTB tests showed that the strengths of cladding samples irradiated at temperatures ranging between 773 and 873 K and fast neutron dose up to 89 dpa were almost as same as those of unirradiated. No significant effect of irradiation on tensile properties and rupture strength were observed under these irradiation conditions. On the other hand, at higher temperatures above 923 K and higher doses up to 102 dpa, tensile strength decreased as compared with that of unirradiated. The rupture strengths of samples that were irradiated at more than 923 K decreased considerably in TTB tests. It is suggested that such significant strength degradation of PNC-FMS claddings irradiated at higher temperatures were caused by the drastic microstructural change during irradiation, especially disappearance of lath martensitic structures and precipitation behavior.

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08-50

Deuterium Retention of Boronized Ferritic Steel Wall in JFT-2M Tokamak

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In JFT-2M tokamak, almost entire region of inner wall of vacuum vessel have been covered by reduced activation ferritic steel, F82H (8Cr-2W), in order to investigate the compatibility of the ferritic steel with high performance plasmas. The inner wall was exposed to approximately 4000 main deuterium discharge shots for 2 years. During this campaign, He glow discharge cleaning was conducted, and also boronization using a glow discharge in 1% B(CH₃)₃ (trimethylboron) + 99% He was 10 times conducted in order to reduce the impurity release. The deuterium retention of the wall affects fuel hydrogen recycling and in-vessel tritium inventory in a fusion reactor, so that it is necessary to investigate the deuterium retention of the wall.

After the shutdown of the JFT-2M tokamak, eight small samples with a size of 9 mm ϕ x 4.5 mm were cut from the ferritic steel exposed to the JFT-2M plasmas. The samples were located at shoulder part in outboard region along the toroidal direction in the JFT-2M. The depth profiles of atomic composition of the samples and the amount of deuterium retained in the samples were measured using Auger electron spectroscopy and thermal desorption spectroscopy, respectively. The relation between the deposited film and the deuterium retention properties was examined.

The thickness of deposited film ranged from 80 nm to 200 nm. The atomic ratio of boron to carbon ranged from 0.05 to 0.2, which was smaller than the ratio in trimethylboron (B/C=0.33). This is owing to the deposition of carbon eroded at the graphite limiter. The boron content was observed to be large at the anode and the gas inlet of trimethylboron. The amount of retained deuterium ranged from 1×10^{16} to 7×10^{16} D/cm². The retained amount was proportional to the thickness of the deposited film. These results indicate that most of deuterium was retained in the deposited film, not in the bulk of the ferritic steel.

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08-51

Assessment of Different Welding Techniques for Joining EUROFER Blanket Components

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In contrast to austenitic steels, untreated welded joints of ferritic-martensitic steels like EUROFER suffer from hardening and embrittlement due to uncontrolled martensite formation in the weld and partially from softening in the vicinity of the heat affecting zones. With respect to specific Test Blanket Module design and assembly requirements for DEMO there might be a significant discrepancy between necessary post-welding heat treatments and their applicability.

Therefore, tungsten-inert-gas welds with EUROFER filler wire, electron beam, and laser welding were used to join EUROFER plates. Specimens were fabricated for a comprising qualification of their mechanical properties by tensile, Charpy, and creep tests. The focus was laid on the study of post weld heat treatments at lowest possible temperatures and for maximum recovery of the joints at the same time. All mechanical tests were accompanied by microstructure examinations.

The lowest reasonable post-welding heat treatment for EUROFER was tempering at 700°C. All tensile and creep specimens fractured outside the welding zones and showed slightly increased strength. But significant differences in impact bending properties could be determined. Both, electron beam and laser welded joints showed ductile-to-brittle transition behaviours nearly as good as the base material while tungsten-inert-gas welds remained rather brittle which necessitated two-step heat treatments including annealing at or above the austenitization temperature. This result clearly favours beam welding techniques for components where high post-welding heat treatment temperatures are inapplicable. On the other hand, unavoidable material flow necessitates beam stoppers which, in turn, restrict their appliance.

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08-52

Grain size effects of irradiated nanocrystalline metals using atomistic simulations

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Understanding the formation of damage defect structures produced at the microscopic level is vital in predicting the life assessments of materials components undergoing irradiation. These defect structures and moreover the material structure of irradiated materials have been seen, both experimentally and computationally, to be affected by the presence of GBs. A window of overlap between experiment and computation exists in the 50nm grain size range where the atomistic resolution of simulation can aid in an increased understanding of the experimental results. An overview of results obtained in the past, as well as new results of large scale molecular dynamics computer simulations of nanocrystalline metals under irradiation in a 6-70nm grain size range are presented in order to address the effect of grain size on materials irradiated. The results are discussed with an emphasis on the role of the grain size, the proximity of the cascade in relation to the surrounding grain boundary network, and the grain boundary structure.

08-53

Atomistic Modeling of Self-Interstitial Dislocation Loops in Fe-Cr Alloys

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High Cr ferritic/martensitic steels are being considered as a structural material for first-wall and breeding-blanket components in future fusion reactor systems. A large number of irradiation defects, including self-interstitial dislocation loops, nanometer vacancy clusters, helium bubbles and voids, in addition to large numbers of transmutants including He and H, will be produced in these materials during high energy (14 MeV) neutron irradiation. Self-interstitial dislocation loops with Burgers vector of $a\langle 100 \rangle$ in bcc Fe have attracted great attention with regard to the formation mechanism, since it is well established that self-interstitial clusters of type $a/2\langle 111 \rangle$ rather than either $a\langle 100 \rangle$ or $a\langle 110 \rangle$, are observed to form in molecular dynamics studies of displacement cascades. Recent atomistic studies have indicated a possible formation mechanism through interactions between $a/2\langle 111 \rangle$ -type self-interstitial cluster dislocation loops. However, the probability of such collisions for $a\langle 100 \rangle$ loop nucleation remains a question.

In this study, the effect of Cr and He atoms on the character of self-interstitial dislocation loops in Fe-alloys is investigated using molecular dynamics simulations with many-body Fe-Cr potentials recently developed. Particularly, this study will focus on the interaction radii and formation of $\langle 100 \rangle$ dislocation loops due to coalescence of $\langle 111 \rangle$ loops in the presence of Cr and He.

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08-54

The role of stacking fault energy in the MD simulations of irradiation induced defects in Ni

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MD simulations were performed to understand the formation of defects following high energy displacement cascades in Ni. Four different empirical potentials are used in order to identify key parameters in defect formation, with a particular focus on stacking fault energy (SFE), for a range of values between 60 to 200 mJ m⁻². The same high energy part was imposed on all potentials. Calculations are performed for temperatures between 10 K to 300 K, and for primary knock-atom (PKA) energies from 5 keV to 40 keV. Common neighbor analysis (CNA) method is used to identify the resulting defect configuration. In particular the occurrence of stacking fault tetrahedra, observed experimentally, is investigated. The PKA induced defects are used as input for kinetic Monte Carlo simulations of the microstructure long term evolution. Results are related to experimental TEM observations performed in single crystal Ni irradiated with 590 MeV protons to low dose at room temperature. The mismatch between the simulations and the observed damage will be discussed. The discussion is focused on the role of SFE on the defect configuration, density and size as a function of dose.

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08-55

Image Simulations of Small Dislocation Loops Based on the Howie-Basinski Equations

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Complete characterisation of the nanometer-sized dislocation loops and other small point-defect clusters commonly found in irradiated materials is difficult. Such defects are often investigated by diffraction contrast methods in the TEM. Image simulations are necessary for a full analysis of the images. The weak-beam method is often used to image small clusters because it offers improved contrast and resolution compared with other imaging conditions. We have developed an image simulation program based on the Howie-Basinski equations of dynamical diffraction theory which is suited to the simulation of weak-beam images of small cluster since it does not make use of the column approximation, which may fail for such conditions.

We have carried out systematic studies of the weak-beam contrast of small dislocation loops in copper. Simulations were carried out for faulted Frank and perfect dislocation loops of size 1-10nm with systematic variations in imaging parameters (the loop orientation, the diffraction vector, the deviation parameter, the loop depth and the foil thickness). Comparisons are made with experiments in ion-irradiated copper. We are able to reach conclusions on the likely visibility of very small clusters, and on the relationship between measured image size distributions and real cluster size distributions.

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08-56

Atomic-scale dynamics of dislocation interaction with vacancy agglomerates in neutron irradiated bcc iron

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Generation of defect clusters occurs directly in high-energy displacement cascades in metals. Further evolution of microstructure can result in formation of extended defects such as dislocation loops, vacancy voids and/or stacking fault tetrahedra, which are visible in TEM. Recent positron annihilation studies of neutron-irradiated iron have demonstrated that there is a significant population of small clusters containing up to a few tens of vacancies that are invisible in TEM: this is supported by MD simulation of displacement cascades. In the present work we have studied the hardening due to the vacancy component of radiation damage in bcc iron. Two types of obstacle for dislocation motion have been considered: (a) compact three-dimensional microvoids and (b) loose vacancy clusters with a high local vacancy concentration. Molecular statics and dynamics have been used to estimate the critical stress necessary for an edge dislocation to overcome such obstacles. The stress has been determined as a function of obstacle size (up to 5nm in diameter) and separation, crystal temperature and dislocation velocity. Dislocation overcome large voids (> 2nm in size) with an Orowan-mechanism-like shape, whereas loose vacancy clusters and small voids are weaker obstacles. The mechanisms involved in these two types of interaction have been analysed.

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08-57

The Effects of Irradiation on the Deformation of Cu: A Comparison between Dislocation Dynamics Modeling and Experiments

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The effects of irradiation on the deformation characteristics of pure copper are evaluated through a comparative study between Dislocation Dynamics (DD) computer simulations and experiments. First, the deformation and dislocation microstructure of unirradiated copper is simulated under uniaxial tensile conditions. Dislocation dynamics simulations of representative volumes in the range 5-10 microns are carried out for a few percent strain, and the results of the computer simulations are compared to detailed experimental examinations. The dislocation dynamics simulations are accelerated by a numerical extrapolation procedure that circumvents massive calculations for dislocation ensemble evolution at every time step. Correlations between experimentally-observed dislocation microstructure and plastic deformation characteristics are established by comparing computer simulations to experiments.

The effects of irradiation on plastic deformation characteristics are examined for specimens that are tested post-irradiation, and for those that are tested in-situ. Computer simulations are carried out in order to establish the salient mechanisms for radiation hardening, embrittlement, and plastic instability. The simulations are performed first at the single dislocation level to determine the effective resistance of irradiated materials to dislocation motion. Such effective properties are then introduced in large-scale DD simulations, and the results of simulations are compared to experiments.

Keywords: Cu, plasticity, hardening, plastic instabilities, dislocation dynamics

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08-58

Molecular Dynamics Study of Influence of Helium Bombardment on Carbon Nanocluster Structure Evolution

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Many unusual mechanical and physical properties of carbon nanotubes can make them very promising for fusion reactor materials. In this relation it is very important to study radiation structural stability and behavior of irradiation – induced defects in nanomaterials. Molecular dynamics simulation of flat carbon clusters and nanotubes under conditions of bombardment with low-energy (10-100 eV) helium atoms and carbon knocked atoms was performed with using well known empirical potentials, in particular, the Tersoff-like potential for C-C interaction. The temperature also was one of the parameters using by the simulation. The temporal evolution and dynamics of vacancies, C-interstitials and helium atoms in flat nanocluster and in nanotube were compared. The effects of secondary collisions, of penetration of atomic particles into nanotube's inside and conditions of release from it were investigated.

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08-59

First-principles calculation of vacancy-solute element interaction in bcc iron

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We perform the first-principles calculation of vacancy (V) and vacancy-solute elements complex in body center cubic (bcc) iron. Interstitial solute elements (carbon and nitrogen) form the vacancy-carbon (nitrogen) complexes. In VCn complexes (VCn, n: number of carbon atoms) of bcc iron, VC3 (2 dimensional configuration) is the most stable complex and the binding energy is largest between VC2 (linear configuration) and VC, equal 0.97eV. In VNn complexes (VNn, n: number of nitrogen atoms), VN is the most stable complex and the binding energy equal 0.86eV. These large binding energies originate from the matrix distortion energy of interstitial elements. We also calculated the vacancy-substitutional solute elements (Mo, Mn, Ni, Cu, Cr, Ti, Si) binding energies in bcc iron. The vacancy-substitutional solute element binding energies are typically around 0.2-0.3 eV, which is not large as for interstitial solute elements.

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08-60

On the capture of small glissile dislocation loops by large sessile loops

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The 14MeV neutrons generated by fusion produce high-energy collision cascades. Recent MD simulations show that self-interstitial atoms produced in these cascades directly agglomerate into small dislocation loops, and many of them are glissile in nature. These small loops will subsequently diffuse and interact with other loops or network dislocations. As a result, the formation of larger loops as well as the evolution of the network dislocations depends also on the capture rate of these small loops rather than of individual self-interstitials only. The strain field of these mobile loops gives rise to a strong interaction with other dislocations that will impact the bias for void swelling. In this study, we employ elasticity theory to evaluate the interaction force in FCC metals between the glissile perfect SIA loops and a sessile large loop. The capture volume of the large loop is then determined by the region in space where this interaction is sufficiently attractive along any of the possible glide cylinders for any of the possible Burgers vectors.

There are only three combinations of the Burgers vectors for two perfect loops in FCC metals, with their angles 0°(parallel), 90°(perpendicular), and 60° respectively.

When the Burgers vectors are parallel, there exists a saddle along the glide direction of the smaller one. If one loop is formed near the other and the distance is closer to the saddle point, the small loop will approach to the closest point of the large loop by the glide motion. The smaller loop will be absorbed by the larger loop with its conservative-climb motion. When two loops are perpendicular, the interaction force is always repulsive and they will not come closer. The more complex case can be expected when the angle between the two loops is 60°. Whether the force is attractive or repulsive depends on the closest location of the two loops.

The capture volume of the loop for other loops can be evaluated by integrating the area where the interaction force reveals attractive. However, when we incorporate the loop rotation by the strain field of other dislocation, this capture volume will become larger.

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08-62

Molecular Dynamics Simulations study of hydrogen isotopic effects in amorphous Silica using an empirical potential

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Fused silica is a material of interest due to its increasing number of applications in many different technology fields. In thermonuclear fusion it is a key component in a number of diagnostics of the Safety and Control Systems of the ITER machine as well as in the final focusing optics of lasers for NIF.

Materials properties of interest (optical absorption, radioluminescence, mechanical properties, ...) are closely related to the presence of defects. These defects can be generated directly by irradiation or by the presence of impurities in the material. Hydrogen is an ubiquitous impurity in this material and, moreover it will be generated in fusion reactors by nuclear reactions with the starting material. Hydrogen isotopes will be deposited also on the surface of the fused silica components coming from the reaction chamber. On the other hand, there are some experimental results that show radiation damage can be different as a function of hydrogen content of the material, indicating that a detailed knowledge of the hydrogen role in fused silica should be fully understood.

In this work we present molecular dynamics simulations to study the effects of different hydrogen isotopes in this material and their interaction with the defect concentration. The interatomic potential developed by Feuston and Garofallini will be used in these studies. The diffusion coefficients and mechanisms of H mobility in fused silica will be calculated and compared with those existing in the literature.

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08-63

Modeling of Sub-cascade Formation in Irradiated Materials by Fast Neutrons with Fusion Energy Spectrum

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A new theoretical model is developed and used for the investigation of sub-cascade formation in fusion structural materials under fast neutron energy spectrum corresponding to the fusion reactor-ITER. The developed model is based on the analytical consideration of elastic collisions between displaced moving atoms into atomic cascade produced by a primary knock atom (PKA) with the some kinetic energy getting from fast neutrons. The criterion of sub-cascade formation is based on the comparison of two calculated values: mean distance between two consequent PKA collisions and the size of sub-cascade produced by PKA. The Tomas-Fermy interaction potential is used for the describing of elastic collisions between moving atoms. The suggested model takes into account electronic losses of moving atoms between elastic collisions of them during the scattering process. The analytical relations for the most important characteristics of sub-cascade are determined including the average number of sub-cascades per one PKA in the dependence on PKA energy, the distance between sub-cascades and the average sub-cascade size as a function of PKA energy. The developed model allows determining the size distribution function in the dependence on PKA energy.

Based on the developed model the numerical calculations for main characteristics of sub-cascades in different materials are performed using the neutron flux and PKA energy spectra for fusion reactor-ITER. So the total numbers of sub-cascades, distribution functions of sub-cascades in dependence on their sizes and generation rate of sub-cascades for different fusion structural materials: Fe, V, Cu and C are calculated for these fusion irradiation conditions. The obtained numerical results for main characteristics of sub-cascade formation under fusion irradiation conditions in these materials are compared with the same results obtained in these materials using neutron energy spectrum of High Flux Irradiation Reactor (HFIR).

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08-64

Dislocation Loop Formation in Irradiated Binary Vanadium Alloys

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Vanadium-based alloys are considered as one of candidate structure materials for fusion reactors and so understanding of physical mechanisms of an effect of solute atoms in these alloys on microstructure change is very important for development of fusion material technology. Experimental investigations show that in irradiated binary vanadium alloys V-A (A=Fe, Cr and Si) the number densities of self interstitial (SIA) loops are found to be much higher than in pure vanadium. This indicates that solute atoms trap SIAs and enhance dislocation loop nucleation.

In the present paper, the di-atomic cluster nucleation model is extended to describe the formation of SIA loops in irradiated binary vanadium alloys, including the effect of undersized solute atoms on SIA loop nucleation and growth. In this model undersize solutes are considered to have strong binding with SIAs and can act as the loop nucleation sites. The suggested model takes into account also the effect of solute segregation to loops and dislocation lines. The segregation of undersized solute atoms on dislocation lines and SIA loops modifies the dislocation bias. Such bias modification affects the nucleation and growth SIA loops too. The influence of these two factors on nucleation and growth dislocation loops in binary vanadium alloys are presented here. It is shown that under irradiation the density of dislocation loops is increased with the increasing of concentration of undersized solute atoms and growth kinetics of SIA loops in these alloys is changed too.

The predictions of the model and performed numerical calculations are compared with observed experimental data for dislocation loop formation and growth under electron irradiation. It is found that the model is able to describe the main features of the experimentally observed nucleation and growth of SIA loops in binary vanadium alloys.

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08-65

Effects of Over Sized Element Sn on Diffusion of Interstitial Clusters in Ni Irradiated by Ions and Neutrons

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In the absence of strong fusion neutron sources, irradiation experiments with fission neutrons, ions and electrons are used for the study of the defect structural evolution. Ion irradiation is a very useful technique, since the damage production rate is high and the setting of irradiation condition is easy. However, there are differences in defect structure evolution between ion and neutron irradiations if we compare them at the same irradiation temperature and the same irradiation dose estimated by dpa. In recent years, it has been shown by experiments and computer simulations that the one-dimensional (1-D) motion of interstitial clusters plays a key role in void swelling in metals under cascade irradiation. In previous papers we showed that the 1-D motion of interstitial clusters was possible to be detected from the defect structures near grain boundaries and the 1-D motion was prevented by over sized elements. In this study, the damage structures induced by ion and neutron irradiations near grain boundaries were compared to clarify the difference of ion and neutron irradiations.

Pure Ni (99.99%) and Ni-2at.%Sn were used in this study. The volume size factor of Sn is 74.08%. The neutron irradiations were carried out at 573 K to doses up to 0.46 dpa using the Japan Materials Testing Reactor and the Kyoto University Reactor. Ion irradiations were carried out from 573 K to 873 K to doses up to 1 dpa using 2.4 MeV Ni ions. After the ion irradiations, the microstructures at the damage peak area were observed by electron microscopy.

From the defect structure of no interstitial clusters near grain boundaries, it was concluded that the 1-D motion of interstitial clusters existed in Ni irradiated with neutrons and ions at low irradiation dose (0.1dpa). In ion irradiated Ni, the 1-D motion was suppressed by high dose irradiation (1dpa). Over sized element Sn prevented the 1-D motion of interstitial clusters. The temperature to develop dislocation structures which indicated no 1-D motion by low damage rate irradiation with neutrons moved upward by 200 K in Ni and by 100 K in Ni-Sn by high damage rate irradiation with ions. In addition, the effects of PKA energy on formation of defect clusters would be also discussed.

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08-66

Small Angle Neutron Scattering Study of Radiation Damage in a Proton-Irradiated Tempered Martensitic Steel

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Series of specimens of the reduced activation tempered martensitic steel EUROFER 97 have been irradiated with 590 MeV protons in the Proton Irradiation Experiment (PIREX) facility, at the Paul Scherrer Institute, at various temperatures ranging between 50 and 350°C and to various doses ranging between 0.3 and 2 dpa. Small angle neutron scattering (SANS) measurements were performed on unirradiated and irradiated specimens of the EUROFER 97, at room temperature and with a saturating magnetic field applied to the specimens. It was found that the scattering associated with the irradiation-induced defects is consistent with a distribution of nano-spheres whose size peaks at about 0.6 nm. The number density of these features increases with the irradiation dose and decreases with increasing irradiation temperature. The size of the defects does not increase with dose in the investigated dose range. The ratio between the nuclear and magnetic scattering intensities is about 2.5, which is indicative of a distribution of nanoscale defects in the form of Cr and Fe carbides, mixed interstitial clusters, and voids or helium bubbles. It is then important to analyse SANS results on the basis of Molecular Dynamics simulations, for instance, of various distributions of different types of irradiation-induced defects. At present, simulation of the nuclear SANS signal was performed. Simulation was performed using the Electron Microscopy Software (EMS) code, which was originally designed to simulate transmission electron microscopy images and diffraction patterns using the multislice technique and which was modified to simulate the SANS signal.

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08-67

Application of Positron Beam Doppler Broadening Technique to Ion Beam Irradiation in Iron and Nickel

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Positron annihilation technique has been widely used for investigations of vacancy-type defect formation because of its high sensitivity to small vacancy-type defects like nanovoids and monovacancies. In the previous conference we demonstrated a unique method using combination of ion beam irradiation and positron beam Doppler broadening measurement to show clear effect of incident ion species (or ion mass) on volume of each nanovoid in iron at room temperature [1]. However, no clear difference was observed in in-situ Doppler broadening measurement under beam-on and beam-off conditions. It is presumably attributed to thermal stability of nanovoids in iron.

In this study, accumulation of vacancy-type defect in iron and nickel by various ion irradiations was investigated with positron beam Doppler broadening technique. For iron, we focused on thermal stability of nanovoids by isochronal annealing of irradiated specimens and irradiation at elevated temperatures. S parameter was increased during irradiation at room temperature to indicate accumulation of vacancy-type defects, and the increment was recovered through isochronal annealing from 373 K to 523 K. The effects of incident ion species on recovery stages will be discussed.

For nickel, in-situ measurement during irradiation was carried out for various specimens (annealed plate, cold-rolled thin foil, annealed thin foil). We discovered the temporary S parameter increase only during irradiation in some conditions, which might be due to unstable vacancy-type defects. Details will be discussed.

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08-68

Microstructural Investigation, Using Small Angle Neutron Scattering (SANS), of OPTIFER Steel Under Low Dose Neutron Irradiation and Subsequent High Temperature Tempering

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This contribution will present the results of a study carried out using small-angle neutron scattering (SANS) to investigate the microstructural effect of low dose neutron irradiation and subsequent high temperature tempering in the reduced activation ferritic/martensitic steel Optifer (9.3 Cr, 0.1 C, 0.50 Mn, 0.26 V, 0.96 W, 0.66 Ta Fe bal wt%). The investigated Optifer samples had been neutron irradiated, at 300°C, at the Petten reactor up to dose levels of 0.8 dpa and 2.8 dpa. Some of them underwent 2 h tempering at 700°C after the irradiation. The SANS measurements were carried out at the D22 instrument of the High Flux Reactor at the ILL-Grenoble. For each irradiated sample a reference sample, submitted solely to a corresponding thermal treatment, was investigated. The SANS results, analyzed considering the differences in nuclear and magnetic scattering, suggest that the post-irradiation tempering promotes the growth of non-magnetic defects, such as He bubbles or microvoids

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08-69

The Evolution of Stacking Fault Tetrahedra and Damage Accumulation in FCC Metals under Cascade Damage Conditions

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By now it is well established that clusters of both vacancies and self-interstitial atoms (SIAs) are produced directly in the displacement cascades in FCC and BCC metals. Furthermore, some of these SIA clusters diffuse one-dimensionally. Over the years, these features of primary damage production and the ensuing consequences have been incorporated in the production bias model (PBM) and various aspects of defect accumulation under cascade damage conditions have been successfully treated both analytically and numerically within the framework of this model. However the evolution of vacancy clusters in the form of SFT produced by cascades in FCC metals and their impact on the overall damage accumulation in the temperature range of practical interests have so far not been considered seriously and systematically.

In an effort to fill in this gap, we have considered the evolution of SFTs in irradiated pure Cu at temperatures below the recovery stage V. Reasonable agreement between the calculated results and experimental observations may be obtained assuming that (a) a direct impingement of cascades on existing SFTs reduces both the rate of SFT accumulation and the defect production efficiency beyond a certain dose level and (b) SFT evolution obeys a simple growth and shrinkage mechanism by absorption of mobile defects. This work has been extended to include the case of irradiation at higher temperatures when SFTs and voids evolve concurrently. The role of the interaction of cascade induced SIAs and their clusters with SFTs on SFT evolution at temperatures above the recovery stage V, particularly the possible transformation of SFTs into Frank loops will be considered. The behaviour of SFTs during post irradiation annealing will be also briefly discussed.

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The influence of stepwise change of irradiation temperature on microstructural evolution of precipitate strengthened copper alloys

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Copper alloys are expected as diverter heat sink materials of ITER component. And it is well known that they are received irradiation damage of 14MeV neutron with varying temperature by pulsed running of the reactor. Recently, a few irradiation experiments have reported that evolutions of damage structure under varying temperature are not same as that of constant temperature. Therefore, comparison of microstructural evolution after constant and stepwise changes of irradiation temperature is very useful. In this present work, to investigate the effect of stepwise change of temperature on microstructure, heavy copper irradiation on copper and its alloys under steady and pulsed varying temperature was performed.

The GridCop CuAl15 (0.15wt%Al content in the form of Al₂O₃ particles) and CuCrZr (Cu-0.8wt%Cr-0.2wt%Zr) were used in this study. Annealed high purity copper (supplied by Johnson-Matthey Chemical Ltd) was also used as a reference material. TEM discs were irradiated up to 30dpa with 2.4MeV Cu²⁺ at

constant temperature of 473, 673 K and periodic temperature variation of 473 / 673 K using a tandem type accelerator at Kyushu University. After the irradiation, microstructures of irradiated samples were observed by Transmission Electron Microscope (TEM).

TEM observation of GridCop CuAl15 before irradiation revealed that alumina particles were dispersed homogeneously. The number density and mean size was $5.3 \times 10^{22} / \text{m}^3$ and 2.1 nm, respectively. The mean grain size of CuAl15 was about 800 nm. On the other hand, the unirradiated microstructure of CuCrZr consisted of small spherical precipitates with the number density of $6.8 \times 10^{22} / \text{m}^3$ and an average size of 4.5 nm. After 30dpa at 673 K constant temperature, voids structure with an average size of 68 nm were observed in CuAl15. However, the strongly dependence of grain size on voids formation was observed. In the case of CuCrZr, voids were not observed. On the other hand, on periodic temperature irradiation, the number density of voids in CuAl15 was decreased as compared with 673K constant temperature irradiation. This result of CuAl15 is good agreement with our previous results of pure copper irradiated under same irradiation conditions.

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08-72

One dimensional Motion of Interstitial Clusters in Ni-Au Alloy

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One dimensional motion of interstitial clusters is important for the microstructural evolution in metals. Alloying elements are expected to affect the motion of interstitial clusters. For example, in neutron irradiated pure Ni, well developed dislocation networks and voids were observed at 573K at a dose of 0.026 dpa by transmission electron microscopy (TEM). Existence of microvoids was detected by positron lifetime measurement even at a low dose level of 0.001 dpa. After the addition of 2at.%Si (-5.81%: volume size factor to Ni) and Sn (74.08%), no voids were detected by TEM observation and positron lifetime measurement. In this paper, the effect of alloying elements on the motion of interstitial clusters was investigated by computer simulation.

Computer simulation was performed in pure Ni and Ni-Au (63.60%) alloy employing the effective medium theory (EMT) potential. The surfaces of model lattice were parallel to (-110), (-1-12) and (111). The lattice with defect clusters was fully relaxed by the static method under fixed boundary conditions. The method did not take into account temperature effects. Small interstitial clusters were constructed by making bundles of crowdions on low index atomic planes, <110> crowdions on {110} atomic planes (loop planes). Alloying element Au was placed to prevent the motion of interstitial clusters. The activation energy required for the motion of interstitial clusters was calculated by integrating the force-distance curve. The number of interstitials in clusters was 1, 7 and 19.

The activation energy for the motion of a crowdion (I_1), a bundle of 7 crowdions (I_7) and 19 crowdions (I_{19}) was 0.036eV, 0.052eV and 0.18eV in pure Ni, respectively. The activation energy of interstitial clusters became high due to existence of the alloying elements. When an Au atom was placed at the center of an interstitial cluster, that of I_1 , I_7 and I_{19} was 2.75eV, 2.65eV and 0.98eV, respectively. I_1 and I_7 cannot thermally migrate if there exists the Au atom at lower temperatures. The activation energy of I_{19} was lower than that of I_1 and I_7 . The effect of position of alloying elements in the large interstitial clusters will also be discussed.

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08-73

MEAM Potentials of Fe, Fe-Cu, Fe-Cr and Fe-C Systems for Cascade Simulations

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A comparative study on the applicability of Modified Embedded Atom Method (MEAM) potentials to the prediction of irradiation defect formation has been performed for pure Fe, Fe-Cu, Fe-Cr and Fe-C alloy systems. The defect properties (formation energies of point defects and interactions between point defects) as well as the fundamental physical properties of relevant material systems are calculated using the MEAM potentials and are compared to corresponding experimental data, high level calculations and those calculated using different empirical interatomic potentials when available. Number of residual primary defects and their size distribution after cascade simulations are also provided for individual systems in comparison with available results from different empirical interatomic potentials. The results of the cascade simulation on the Fe-C interstitial alloy system are those reported for the first time.

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New Mechanism of the Formation of $\langle 100 \rangle$ Dislocation Loops in Iron

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It is known that two types of interstitial-type dislocation loops are formed in ferritic alloys upon irradiation with energetic particles—those with the Burgers vector of $1/2\langle 111 \rangle$ and those with the Burgers vector of $\langle 100 \rangle$ (see Ref. [1]). The mobility of the one-dimensional prismatic glide of $1/2\langle 111 \rangle$ loops is extremely higher than that of $\langle 100 \rangle$ loops [2]. Therefore, such a difference between the values of the Burgers vector will be significant to the microstructural evolution upon irradiation. A few mechanisms have been proposed until date in explanation of the formation of $\langle 100 \rangle$ loops, which are energetically unfavorable to $1/2\langle 111 \rangle$ loops [3]. The latest mechanism proposed by Marian *et al.* [3] attributed the formation of a $[010]$ loop to a conventional dislocation reaction during the coalescence between a mobile $1/2[\bar{1}11]$ loop and a $1/2[11\bar{1}]$ one. However, experimental evidence for these mechanisms has not yet been obtained. In this paper, we investigate the dynamic behavior of loops in pure Fe under thermal annealing by *in situ* TEM, and we propose a new reliable mechanism of the formation of $\langle 100 \rangle$ loops.

The spontaneous change in the Burgers vector of a small $1/2\langle 111 \rangle$ loop with diameters less than approximately 20 to 30 nm into another $1/2\langle 111 \rangle$ or $\langle 100 \rangle$ occurred [4]. In contrast, the Burgers vector of small mobile $\langle 100 \rangle$ loops and larger $1/2\langle 111 \rangle$ loops rarely changed spontaneously. The origin of these phenomena can be understood by the idea that small loops comprise bundles of crowdions (see Ref. [5]). When one loop collided with another, the two loops coalesced. We examined the process of this coalescence and found that it can be attributed to the absorption of one loop by the other, such as that of a $1/2[111]$ loop by another $1/2[111]$ loop, that of a $1/2[\bar{1}11]$ loop by a $1/2[11\bar{1}]$ loop, a $1/2[111]$ loop by a $[100]$ loop, a $[100]$ loop by another $[100]$ loop, and so on. The second process appears to contradict the formation mechanism proposed by Marian *et al.* [3]. In contrast, we propose the following mechanism—small $\langle 100 \rangle$ loops is formed by the spontaneous transformation from small $1/2\langle 111 \rangle$ loops, and the formed $\langle 100 \rangle$ loops in turn will not make a transformation and grow by the absorption of not only SIAs but also $1/2\langle 111 \rangle$ and $\langle 100 \rangle$ loops.

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08-75

A Comparison of Radiation Damage in Ion Irradiated Monolithic CVD- and NITE-SiC

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SiC/SiC composites are promising structural materials for fusion power reactors, due to the superior high-temperature properties, thermo-chemical stability, irradiation tolerance, inherent low-activation and low after heat properties. Recently, the excellent material properties of newly developed NITE-SiC/SiC composites have been demonstrated without irradiation, however, the knowledge about neutron-irradiation effects on these properties is extremely limited.

In this paper the dimensional stability and microstructural change in monolithic CVD- and NITE-SiC after Si²⁺-ion irradiation with and without He⁺-ion injection at high temperature and annealing behaviors were studied. One of the significant differences between the two cubic-monolithic-SiC is the grain size. The size of grains in CVD-SiC is mostly 1-50 microns, and NITE-SiC has very fine grains. Swelling in SiC irradiated up to 3 dpa above 1073 K was measured by precision-surface profilometry. The recovery of swelling started around irradiation temperature by post annealing experiments, where the swelling decreased with annealing temperature in single-ion irradiated specimens. Above annealing temperature of 1273 K, swelling of dual-ion irradiated specimens was increased due to cavity formation at grain boundaries. These results and the comparison of swelling behaviors between CVD- and NITE-SiC are discussed in highlight of the TEM observations.

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08-76

Effect of Solute Elements of Ni Alloys on Swelling and Blistering under He⁺ and D⁺ Ion Irradiation

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Effects of solute atoms on microstructural evolutions and blister formation have been investigated using Ni alloys under 25 keV He⁺ and 20 keV D⁺ irradiation at temperatures from room temperature to 500°C. The specimens used were pure Ni, Ni-Si, Ni-Co, Ni-Cu, Ni-Mn and Ni-Pd alloys with different volume size factors, and the volume size factors of solute elements for the Ni alloys are from -5.8% to 63.6%. Helium atoms were implanted up to doses from 1.0x10¹⁹ to 4x10²¹ ions/m², and deuterons were also implanted up to 4x10²¹ ions/m². After irradiation, the microstructures and surface features were observed by a transmission electron microscope and a scanning electron microscope, respectively.

In previous study, we reported the development of dislocation loops and cavities formed in these specimens. The high number densities of dislocation loops about 1.5x10²² m⁻³ were formed at 500°C in these specimens irradiated to 1x10¹⁹ He/m², and they were approximately equivalent, except for Ni-Si. The mean size of loops tended to increase with volume size factor of solute atoms. In a dose of 4x10²⁰ He/m², the swelling at 500°C was changed from 0.2 to 4.5 %, depending on volume size factors. The number densities of bubbles tended to increase with the absolute values of volume size factor, and the swelling increased with volume size factors. These results were suggested that mobility of helium and vacancy atoms might be influenced by the interaction of solute atoms with them.

In present study, the formation blisters on the irradiated surfaces of these specimens were observed at a dose of about 4x10²¹ He/m². The areal number densities of blisters in the specimens increased with volume size difference of solute atoms. The size of the blisters inversely decreased with it. The results indicate that the formation of blisters is intimately related with the bubble growth. On the other hand, no formations of blisters were observed in the specimens irradiated by deuterons.

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08-77

Modeling Cascade Aging and Dose Rate Effects in Ferritic Alloys

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Fundamental understanding of defect production in displacement cascades is required to model and predict long-term neutron irradiation induced microstructural evolutions. Defect production is generally treated in terms of primary events, occurring in cascades over time scales of less than 100 ps. We describe the development of advanced kinetic lattice Monte Carlo (KMC) methods to simulate the long-term rearrangement (aging) of displacement cascades as well as cascade aging effects on overall damage accumulation in neutron irradiated Fe, Fe-Cu and Fe-Cr-He alloys. Special algorithms have been developed to model self-interstitial atom-vacancy recombination in cascades and long-range point defect and solute diffusion. The simulations reveal the formation of a continuous distribution of three dimensional cascade vacancy-solute cluster complexes and demonstrate the critical importance of spatial, as well as short and long-time, correlated processes, that mediate the effect of dose rate on microstructural evolution under conditions relevant to fusion reactor materials.

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08-78

Using High Entropy Alloys as the First Wall Structural Materials for Fusion Reactors

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A brand new type of alloys are developed recently in materials science field, known as 'high-entropy alloys', which contains more than five major alloying elements up to 12 elements. This type of alloys showed very high mechanical strength and high erosion resistance at elevated temperatures (current data is up to 1000°C) and much better than super alloys. The corrosion resistance in various acidic environments of the alloys is superior to that of stainless steels. And most importantly, the microstructures of the high entropy alloys are very simple instead of very complex as we might imagine which benefit to start further investigations on the alloys. The reason that this type of alloys performed so well is attributed to the very high entropy comes from the mixing of many major alloying elements simultaneously.

As we all aware that proper structural materials are the key factors for fusion reactor to be feasible. In the past, we are always looking for the proper candidate materials from the existing alloys or composites. Nevertheless, high entropy alloys should be one type of materials worth our attention to study for. We have started a project to study the ion-irradiation effects to the microstructural evolution of the high entropy alloys in order to understand the behaviors of these materials under heavy irradiation doses. Due to the nano-crystalline structures and high entropy of the unirradiated materials, we expect that this type of materials will have very stable behavior in microstructural evolution under irradiation. Further studies using dual-beam and triple-beam ion irradiation are also under planning. The first candidate alloy for this study is Cu-Ti-V-Fe-Ni-Zr with equal moles for each of the major elements and mixing together.

08-79

Overview of the Recent Russian Materials and Technologies R&D Activities Related to ITER and DEMO Constructions

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An overview is given of the wide range of activities and major achievements contained within recent R&D being performed within Russia on materials and technologies for ITER and DEMO.

In the Russia, the basic manufacturing materials and technologies have been selected for the ITER, for the two reference DEMO breeding blanket concepts: the self cooling (the structural materials - vanadium alloy) and the ceramics (the structural material – the low activation ferritic-martensitic 12% chromium steel) and for the related long term R&D.

The main activities in fusion materials developments in Russia have been observed and the corresponding long term Programs have been outlined. The review on the recent results of investigations on low activated materials (V-Ti-Cr alloys, RAFM 12% Cr steel), beryllium and superconducting materials has been presented. Among the low activated materials the V-4Ti-4Cr alloy and the RAFM heat resistance steel RUSFER-EK-181 (Fe-12Cr-2W-V-Ta) were studied intensively.

The fabrication of tubes, sheets and other forms from low activation materials have been successfully established and the process of welding has been designed and investigated for them. The mechanical properties and microstructure have been investigated in the wide temperature range up to 800°C.

The activity in beryllium materials combines the domestic studies and international cooperation in the framework of EC-Japan-RF "HIDOBÉ" Project and is aimed on the experimental DEMO blanket modules development in ITER Project.

The progress in enhancement of the properties of superconducting materials, which are the key element of the ITER magnet system, has been presented. The prospects of further developments in superconducting materials for DEMO magnet system has been analyzed. It has been shown that Nb₃Sn strands still remain the most probable candidate for the future fusion reactors.

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08-80

ITER Vacuum Vessel In-Wall Shielding Block Material Requirements

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The material requirements based on the detail design and the development of the fabrication method have been studied for ITER in-wall shielding of the vacuum vessel. The space between the inner and outer shells is filled with pre-assembled or modular shielding blocks. The in-wall shielding material is boron doped stainless steel or ferromagnetic steel. 40 mm thick flat plates will be used. These flat plates will fill 55% (inboard) ~ 60 % (outboard) of the volume between the vessel shells to provide an effective neutron shielding capability. Because the vacuum vessel has a 3-dimensional shape and overhangs when the in-wall shielding is installed into the VV inter-space, the assembly scheme should be considered to fill the space between the shells. The works on the detail design and the development of the fabrication method have been done by Korea. The design works included structural, thermo-hydraulic analyses and development of installation schemes. And, in the mean time the in-wall shielding material investigation has been followed. In this paper, results of the in-wall shielding material investigation were considered for boron doped stainless steel and ferromagnetic steel in light of the recent detail design requirements. Austenite stainless steel contained with 1~2% boron was fabricated and analyzed with ICP-OES (Inductively Coupled Plasma–Optical Emission Spectrometer), as well as the microstructure (precipitation: Cr₂B, (Cr, Fe)₂B) and the mechanical properties.

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08-81

Verification of Design Rules for EUROFER under TBM Operating Conditions

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Existing structural design criteria (SDC) are based on the material data, which do not consider a possible change of material properties such as the tensile strength and the yield stress after a cyclic loading. The aim of this work is to find out whether the available SDC may also be applied to the EUROFER subjected to operating conditions of the ITER test blanket module (TBM) i.e. thermal and mechanical cyclic loading.

Recent experiments performed at FZK have shown that EUROFER exhibits softening during the cyclic loading like other ferritic martensitic steels, see fig. 1, contrary to austenitic steels, which usually harden cycle by cycle. Such a softening results in a remarkable decrease of the lowest stress intensity at a given temperature among the time-independent strength quantities, which is required for some important design rules. Thus, the allowable load is in fact smaller than the limit predicted by design rules nowadays. Evidently, it can lead to a fatal design error.

Within the frame of the present work, a range of the working temperatures and mechanical loads has been specified for EUROFER under consideration of advanced material properties such as the softening cycle by cycle mentioned above and the creep-fatigue damage with EUROFER-specific parameters using the FE code ABAQUS. Thereby, a user-specified material routine (UMAT) developed and implemented by J. Aktaa has been used to describe the creep-fatigue. The FE model considers the current TBM design, usual and accident ITER operating conditions as well as typical problems connected with the TBM development. A comparison of the lifetime assessment results with conclusions obtained by an application of the SDC to results of a linear-elastic simulation allowed a verification of the SDC.

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08-82

The effect of low dose neutron irradiation on mechanical properties, electrical resistivity and fracture character of NiAl bronze for ITER

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Nickel-aluminum bronze (CuAl10Ni5Fe4, UNS No. C63200) is considered as structural material for several applications in the ITER in-vessel components such as divertor and blanket attachments, and remote handling equipment. The main advantages of this material are anti-seizing properties even in vacuum, no generation of sparks when subjected to impact against steel, good mechanical properties and low cost.

However, data on the effect of neutron irradiation on the properties of this alloy are lacking. The paper presents the first results of experimental investigation of the effect of neutron irradiation on hardening, embrittlement, fracture character and electrical resistivity of NiAl bronze at low dose irradiation. Specimens of NiAl bronze were irradiated at a temperature of 150°C to doses of 10^{-3} , 10^{-2} , 10^{-1} dpa in the RBT-6 reactor ($\Phi_{\text{therm}}/\Phi_{\text{fast}} \sim 1$) at Dimitrovgrad. The samples were tensile tested, and the change in the electric conductivity of irradiated materials was measured.

It is shown that neutron irradiation leads to radiation strengthening of NiAl bronze by 35 MPa, the uniform elongation was reduced from 18% to 13%. The change in the electrical resistivity of NiAl bronze is low, at maximum dose this change is about 3%.

The SEM and optical microscopy investigations of irradiated NiAl bronze show that the fracture character of this material is ductile and that the fracture is transgranular. The microstructure of the materials was characterized as having a very inhomogeneous structure consisting of several dendrite type phases.

Finally, the reasons for the high radiation resistance of NiAl bronze in comparison with pure copper and CuCrZr alloy are discussed.

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08-83

Narrow-gap, Thick section, Hybrid Laser Conduction Welding Developments for ITER Vacuum Vessel

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The work described herein is a welding development for ITER, supported in part by the European Fusion Development Agreement, and realised by a co-operation between CEA, Arceuil, France and BAE Systems, Bristol, UK. Each of the nine vacuum vessel (VV) sectors, constructed from two walls, each 60-75 mm thick, of ITER-grade 316L stainless steel, must be joined on-site with four welds using an intermediate splice plate, a total of 1.8 kilometres of weld length for the complete vessel.

Since the ITER reference process of the industrially proven (but slow) multi-pass narrow gap TIG method (NGTIG) requires the simultaneous use of four welding sets, we propose a new process, able to weld in a narrow gap with the same dimension and with the same joint metallurgical quality as that of the NGTIG, but with productivity improved by several times, using a faster welding speed and increased wire deposit rate. Although earlier work has generally utilised a focussed laser beam, in Hybrid Laser Conduction Welding (HLCW), a defocused YAG laser is combined with a TIG torch to achieve the root pass. For the filling passes, while the TIG + YAG process is demonstrated with good quality and repeatability, the investigated MIG/MAG + YAG process promises to increase productivity further.

We explain in this paper how HLCW is a suitable method to produce high-quality, low distortion, high-thickness 316L stainless steel welds in the all-position welding configurations for the on-site fabrication of the ITER VV.

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08-84

Irradiation stress relaxation of ITER candidate bolting materials

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In a European design concept, the ITER first wall panels are attached to the blanket modules by bolts. Two candidate materials for this application are 38% cold worked Alloy 625+ and PH13-8Mo. To deal with the operational loads and to allow for some stress relaxation, these bolts have to have very good tensile properties because they need to be heavily pre-stressed during placement of the panels. During ITER operation, the bolts will lose part of the preload due to stress relaxation as a consequence of irradiation creep. It is therefore important that the stress relaxation with dose level be quantified.

Pre-stressed bolts were assembled and irradiated to several dose levels (0.28 - 2.66 dpa), at temperatures in the range of 271°C to 322°C, and under pre-stress loads typical for the ITER conditions. The stress relaxation bolts were tensile tested after irradiation. To verify the results obtained with the pre-stressed bolts, some constant-curvature bent strips were irradiated as well.

The stress relaxation of Alloy 625+ under irradiation is very large. After an irradiation dose of 2.7 dpa only 20% of the original applied stress is retained with a lower bound going down to a mere 11%. Combined with the observation of irradiation softening this material seems unfit for the design purpose. It is assumed that particularly the cold work is responsible for the excessive relaxation.

The alternative candidate material PH13-8Mo shows much better irradiation behaviour by retaining 60% of the applied stress at the maximum dose level, with a lower bound at 52%. The tensile tests showed a modest irradiation hardening, insuring that the pre-irradiation applied stress will not cause plastic yield in service.

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08-85

Thermal Analysis of the US Solid Breeder TBM (Quarter-port Submodule)

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ITER will provide three equatorial ports where ITER parties can insert their test blanket modules (TBMs) to test their blanket concepts. The ITER TBM programs can be seen as extensive R&D on blanket concepts and the first wall in an integrated fusion environment. Two types of solid breeder TBMs have been proposed by the US TBM program: a quarter-port submodule and unit cell. The quarter-port submodule was selected to be under investigation in this work. The left half of the submodule features layer configuration, where the breeder pebble beds are parallel to the first wall. Edge-on configuration is used in the right half of the submodule, where the breeder pebble beds are placed perpendicular to the first wall.

Extensive thermal analysis of the quarter-port submodule, using a FEA software package, is presented in this paper. The temperature profile of each part of the submodule was studied in both steady and transient states. The results showed that the temperature values of each material and/or part of the submodule are within the design limits of the thermo-mechanics TBM. Also, the results showed that the values of nuclear heating and heat transfer coefficient have significant impact on the temperature profile of the submodule. In addition, a new configuration of the solid breeder and beryllium pebble beds inside the submodule was presented. The temperature profiles of the new submodule were compared with those of the original submodule.

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08-86

Carbon transport, deposition and fuel accumulation in castellated structures exposed in TEXTOR

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Castellated first wall and divertor are proposed as a solution for ITER to maintain its thermo-mechanical durability [1]. However, a concern with such a castellation is the possible accumulation of fuel in the gaps. Dedicated experiments are needed. In TEXTOR molybdenum limiters with ITER-like castellation were exposed in the SOL plasma. In a first experiment, the limiter was exposed under deposition dominated conditions. Carbon deposits were found both on the top plasma facing surfaces and in the gaps. The fuel accumulation in the gaps was estimated to be at least 30% of the overall fuel retention on this limiter [2].

Another castellated limiter was exposed recently to the SOL of TEXTOR but this time under erosion-dominated conditions. The average plasma fluence to the limiter was about 2.0×10^{20} D/cm². After exposure no deposits were detected neither on the plasma facing top surfaces nor on the areas of the gaps with direct view to plasma. However, deposited layers with maximal thickness up to 500 nm were found on the plasma shadowed areas of gaps. These deposits consist of carbon enriched with hydrogen, deuterium, boron and oxygen. Also a significant amount of molybdenum originating from the limiter was found intermixed in the deposited layers. The ERO code was used to model carbon transport into the gaps. Experimental results and comparison with modeling will be presented in this contribution.

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08-87

On the effects of the supporting frame on the radiation-induced damage of HCLL-TBM structural material

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Within the European Fusion Technology Programme, the Helium-Cooled Lithium Lead (HCLL) breeding blanket concept is one of the two EU lines to be developed for a Long Term fusion reactor, in particular with the aim of manufacturing a Test Blanket Module (TBM) to be implemented in ITER. The HCLL-TBM is foreseen to be located in an ITER equatorial port, being housed inside a steel supporting frame, actively cooled by pressurized water. This supporting frame has been designed to house at most two different TBMs, providing two cavities separated by a dividing plate 20 cm thick.

Within the framework of the research and development activities focused on that TBM, at the Department of Nuclear Engineering of the University of Palermo a research campaign has been launched with the specific aim of investigating its nuclear response when operating inside ITER. In particular, the present paper deals with the study of the radiation damage of the HCLL-TBM structural material, taking into account the potential influence of the supporting frame configuration. Therefore, the distributions of Displacement Per Atom and helium and hydrogen production rates have been investigated within the HCLL-TBM structural material, accordingly to various dividing plate thicknesses and supporting frame compositions.

To that purpose a 3D heterogeneous model of HCLL-TBM in a toroidal lay-out has been set-up, taking into account 9% Cr martensitic steel (Z 10 CDV Nb 9-1) as the structural material. The model has been inserted into a 3D semi-heterogeneous one of ITER-FEAT, simulating realistically the reactor lay-out up to the cryostat and providing for a proper D-T neutron source. The supporting frame has been assumed to have a radially layered structure, each layer being composed of a proper homogeneous mixture of water and AISI 316 stainless steel.

The analyses have been performed by means of the MCNP-4C code, running a large number of histories (20.000.000) so that the results obtained are affected by statistical uncertainties lower than 1%.

The results obtained are reported and critically discussed.

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08-88

Experimental Research of Pb(83)Li(17) for Blanket Cooling

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Inherent safety of lead-lithium eutectic induces to carry out the eutectic for application as coolant of blanket of fusion reactor. As against lithium lead-lithium eutectic is explosion-proof and flame-proof. Lead-lithium using allows decreasing MHD-resistance to its flow because of formation oxide insulating coatings on inner surface of ducts.

Research of lead-lithium eutectic as coolant of blanket of fusion reactor is carried out in Nizhny Novgorod State Technical University for a long time. Research consists of two main types of experiments: in situ measurement of insulating coating characteristic $\rho_i \delta_i$ (ρ_i – specific resistance of coating, δ_i – thickness) and measurement of MHD-resistance in transverse magnetic field.

Insulating coating characteristic was measured in range of temperature 300-500 C. Value $\rho_i \delta_i$ was measured during 50 hrs and comes to $(1,1 - 1,25) \cdot 10^{-4}$ ohm·m² MHD-resistance was measured in special test bench with lead-lithium coolant. Induction of transverse magnetic field of electromagnet was up to 1 tesla. Length of test section was 0,5 m, inner diameter of tube was 6 mm.

The analysis of samples from austenitic steel 08X18H10T has shown the following: the weights which have stuck to a surface of samples during extraction from Pb(83)Li(17) with slags was on its surface, have non-uniform thickness and represent eutectic Pb-Li (phases Pb and LiPb) and Li₂CO₃ (friable weights of orange color). The dark film on a surface of samples consists of not identified phase X and oxide Me₂O₃ (Me-Gr, etc.). Thickness of a film on radiographic data makes 1 – 3 microns. Phase X makes a basis of a film and does not concern to known intermediate phases: Fe - Gr - O; Li - Fe - O; Li - Gr - O; Fe - Pb - O; Gr - Pb - O; Ti - Li - O.

Experiments have shown that MHD-resistance of lead-lithium coolant is like one for lead and lead-bismuth coolants. MHD-resistance of lead-lithium coolant essentially differs from MHD-resistance of lithium coolant because last one could not form oxide insulating coatings on inner surface of ducts.

08-89

Numerical Characterization of Thermo-mechanical Performance of Breeder Pebble Beds

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Experimental and numerical approaches have been applied to quantify thermo-mechanical characteristics of ceramic breeder pebble bed assemblies. Due to the lack of a detailed description of heterogeneous material properties, and the significant number of parameters involved, a predictive capability based only on experimental approaches is not enough to characterize the thermo-mechanical behaviors of the pebble bed. The macroscale thermo-mechanical behaviors of a pebble bed material system are highly complex and are related to material properties, microstructure and size of particles, boundary conditions and loading histories, etc.

In this paper, a numerical approach using discrete element method (DEM) has been applied. Our goal is to derive the effective constitutive relations, which are similar to mechanical properties of solid materials, to express the dependence of pebble bed deformation on stress. Numerical results show that, to a greater or lesser extent, the thermo-mechanical behaviors of pebble bed system are inelastic, and the values of effective modulus increase as bed deformation increases. These constitutive relations are dependent on pebble bed temperature and its boundary conditions and also are related to microscale behaviors at the pebble contacts. Numerical results also demonstrate that thermal creep becomes an important deformation mechanism at elevated temperatures. As a result of thermal creep, the contact area between pebbles will grow and stress magnitude can be reduced. Analysis has shown that the stress relaxation period of particulate material assemblies can be much shorter than that of solid materials and the stress exponent factor of the creep mechanism falls between those of the diffusion law and the power law.

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08-90

High Energy Heavy Ion Induced Structural Disorder in Li₂TiO₃

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The structural disorder in Li₂TiO₃ irradiated with high energy xenon (Xe) ions of 18-160 MeV are examined by Raman spectroscopy and X-ray diffraction. Raman peaks of the samples irradiated by 18MeV ions with the electronic stopping power (S_e) of 6.0 keV/nm at surface are slightly reduced as the fluence increase from 2.4×10^{16} to 4.9×10^{17} ions/m². The heights of the 663 cm⁻¹ peak of Ti-O bond vibration and the 422 cm⁻¹ peak of Li-O bond vibration relative to the 352 cm⁻¹ peak of Li-O bond vibration, which are 3.4 and 2.8 before irradiation, are reduced to 2.2 and 1.8 with the increase in fluence, respectively. The height of the 663 cm⁻¹ peak relative to the 352 cm⁻¹ peak is further reduced to 1.7 with the increased S_e value of 9.5 keV/nm. Raman peaks are not observed for the samples irradiated with larger S_e value such as 13.0 and 16.7 keV/nm. Such a reduction and disappearance of Raman peaks indicate that the short-range disordering in Li₂TiO₃ due to the irradiation is significantly connected with the electronic stopping power.

On the other hand, the observed reduction of the (002) supercell X-ray reflections shows that there is no long-range order in Li₂TiO₃ irradiated by the 18MeV Xe ions with the above fluence. These irradiation conditions do not influence the short-range order because of slight reduction in Raman peaks. Thus, these results suggest that the short-range disorder is caused by the larger electronic stopping power as compared with the long-range disorder.

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08-91

Oxidation and stability studies of Beryllium Titanate

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Beryllides are potential candidates to replace beryllium in future fusion power plants due to their improved properties. Among the beryllides, one of the most promising is Ti beryllide due to its lower chemical reactivity. Although, while fabrication routes and properties of Beryllium are well established a lack of knowledge still exists for beryllides.

In this work we present a detailed study of structural stability of titanium beryllides and its oxidation behaviour under air annealing. Both high resolution x-ray diffraction and microbeam techniques were used to follow the evolution of the composition and phases. The microstructure was studied with scanning electron microscopy. Beryllium-titanium intermetallic compounds were produced using two alloys with a nominal composition of Be-5at%Ti and Be-7at%Ti. The as cast samples show the formation of Be₁₀Ti for the Be-7at%Ti alloy while the Be₁₂Ti phase was mostly found in the Be-5at%Ti compound. The Be-5at%Ti alloy reveals intra-grain regions with high concentration of impurities (O, Fe and Ni) and Ti depletion. During thermal treatments up to 800 °C for 1 hour in air, the oxidation occurs preferentially at the beryllide grain boundaries. The stability of the beryllide was followed during the annealing up to 750 °C in vacuum, by in situ x-ray analyses. The results reveal the growth of the Be₁₇Ti₂ phase in the Be-5at%Ti compound. No evidence was found for the presence of BeO phase during the annealing in vacuum.

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08-92

Compatibility between Be-Ti alloys and F82H

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Beryllium (Be) metal is a reference material for a neutron multiplier in the fusion blanket design. However, Be metal may have problems such as high reactivity and large swelling at high temperature (~900°C) and high neutron dose (~20,000appmHe, ~50dpa) in the DEMO fusion blanket. Therefore, it is expected to develop an advanced material that has high-temperature resistance and high-radiation resistance. Beryllium intermetallic compounds such as Be₁₂Ti and Be₁₂V are promising candidates for advanced neutron multipliers from viewpoints of high melting point, high beryllium content, low radio-activation, good chemical stability (low reactivity with hot water and so on). In this study, the compatibility between Be-Ti alloys and structural material F82H was investigated.

Two kinds of Be-Ti specimens, i.e. Be-5at%Ti and Be-7at%Ti that include α Be phase, which have better ductility than stoichiometric Be₁₂Ti, were fabricated as testing materials from beryllium and titanium powder by an arc melting method. Two kinds of diffusion couples, i.e. type 1 (couple of Be-5at%Ti and F82H) and type 2 (couple of Be-7at%Ti and F82H) were prepared to investigate the compatibility. The compatibility tests were carried out by annealing the capsules sealed with high-purity helium (99.9999%) at 600, 700 and 800°C for 100, 300 and 1,000 h. After annealing, the interaction between each coupled specimen was evaluated by X-ray diffraction (XRD) analysis and scanning electron microscopy with electron-probe microanalysis (SEM/EPMA).

Each contact surface of the Be-Ti and F82H was characterized by XRD analysis and SEM observation before annealing. Phases of Be₁₂Ti and α Be were identified in the Be-5at%Ti and Be-7at%Ti by XRD. A needle Be₁₂Ti phase existed in each Be-Ti alloy, and areas of Be₁₂Ti in Be-5at%Ti and Be-7at%Ti were about 65% and 85%, respectively. After annealing, the reaction products such as Be₂Fe were observed on the surface of F82H. The reaction layer was observed by SEM, and the thickness of reaction layer was evaluated. The thickness for the Be-5at%Ti and Be-7at%Ti samples was almost the same. The thickness for types 1 and 2 at 800°C for 1,000 h was approximately 100 μ m, whereas that of Be was 250 μ m. At 600°C for 1,000h, each thickness for Be-Ti and Be was less than 2 μ m and about 10 μ m, respectively.

These results showed that the Be-Ti alloys that include α Be and Be₁₂Ti had the advantage in the compatibility at high temperature.

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08-94

Post-irradiation examination of the Ceramic Breeder materials from the Pebble Bed Assemblies Irradiation for the HCPB Blanket concept

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In the framework of developing the European Helium Cooled Pebble-Bed (HCPB) blanket an irradiation test of pebble-bed assemblies is performed in the HFR Petten. The experiment is focused on the thermo-mechanical behavior of the HCPB type breeder pebble-bed at DEMO representative levels of temperature and defined thermal-mechanical loads. To achieve representative conditions a section of the HCPB is simulated by EUROFER-97 cylinders with a horizontal bed of ceramic breeder pebbles sandwiched between two beryllium beds. Floating Eurofer-97 steel plates separate the pebble-beds. The structural integrity of the ceramic breeder materials is an issue for the design of the Helium Cooled Pebble Bed concept. Therefore the objective of the post irradiation examination is to study deformation of pebbles and the pebble beds and to investigate the microstructure of the ceramic pebbles from the Pebble Bed Assemblies.

This paper concentrates on the Post Irradiation Examination (PIE) of the four ceramic pebble beds that have been irradiated in the Pebble Bed Assembly experiment for the HCPB blanket concept. Two assemblies with Li_4SiO_4 pebble-beds are operated at different maximum temperatures of approximately 600°C and 800°C. Post irradiation computational analysis has shown that both have different creep deformation. Two other assemblies have been loaded with a ceramic breeder bed of two types of Li_2TiO_3 beds having different sintering temperatures and consequently different creep behavior. The irradiation maximum temperature of the Li_2TiO_3 was 800°C. To support the first PIE result, the post irradiation thermal analysis will be discussed because thermal gradients have influence on the pebble-bed thermo-mechanical behavior and as a result it may have impact on the structural integrity of the ceramic breeder materials.

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08-95

Validation of Potential Models for Li_2O in Classical Molecular Dynamics Simulation

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The influence of radiation defects is one of the main factors to determine tritium release behavior from a blanket breeding material in fusion reactors. The classical molecular dynamics simulation (MD) is an attractive technique to address the radiation damage processes, because it can provide atomic-scale information. The reliability of MD results strongly depends on a potential model. Hence, in the present study, we assessed the reported potential models and proposed a new model for Li_2O , a candidate of solid breeding materials, prior to a simulation on the radiation damage processes.

The Buckingham-type pairwise potential was used for the short-range interaction because of its simplicity and reliability for ionic crystals. The coulombic term was treated by the Ewald summation. First, the reported potential models, which were derived by fitting to the result of quantum chemical calculation, were assessed using the GULP code. These models showed good agreement with the experimental results on the lattice constant, the elastic constants, etc. However, the melting points were overestimated by approximately 30 %. In order to reduce this inconsistency, a new model was proposed by the empirical fitting technique in conjunction with the genetic algorithm. The proposed model held a comparable melting point with the experiment, within 10%. This small discrepancy could be ascribed to the exclusion of the surface and the lattice defects in the MD simulation. The formation energies of Li/O Frenkel defect and the migration energies of Li/O vacancy/interstitial were also evaluated. Acceptable agreements with the experimental value were confirmed.

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08-96

Saturation in Degradation of Thermal Diffusivity of Neutron-Irradiated Ceramics at 3×10^{26} n/m²

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Thermal diffusivity is one of the most important factors for fusion reactor materials. In ceramic materials, heat was mainly carried by phonon unlike metals, and neutron irradiated ceramics showed serious degradation in thermal diffusivity. In this work, typical structural ceramics, were neutron-irradiated in the experimental fast reactor JOYO, to fluences of $0.4\text{--}8.0 \times 10^{26}$ n/m² at 646-1039K, and thermal diffusivity was measured by laser-flush method with $t_{1/2}$ analysis at room temperature.

In our previous works (ICFRM-9 and 10), the specimens they were irradiated to neutron fluence of $0.4\text{--}1.4 \times 10^{26}$ n/m² showed very low thermal diffusivity. In this work, specimens they were neutron irradiated to 2.8×10^{26} n/m² showed further degradation of it. However, between a neutron-dose range of $2.8\text{--}8.0 \times 10^{26}$ n/m², it showed very small change and was saturated around 3×10^{26} n/m². All materials showed this inclination, but the saturated value was different with each material, $\beta\text{-Si}_3\text{N}_4$: 3.2×10^{-6} m²/s, $\beta\text{-SiC}$: 2.8×10^{-6} m²/s, $\alpha\text{-Al}_2\text{O}_3$: 1.6×10^{-6} m²/s, AlN: 1.3×10^{-6} m²/s.

A thermal diffusivity is proportional to a mean free path of phonon, and that after an irradiation (l_i) was mentioned as $1/l_i = 1/l_0 + 1/l_d$ where l_0 is that of non-irradiated material and l_d is the mean free path related to neutron induced defect. From the saturated value of thermal diffusivity, l_d was obtained as $\beta\text{-Si}_3\text{N}_4$: 1.2×10^{-9} m, $\beta\text{-SiC}$: 0.8×10^{-9} m, $\alpha\text{-Al}_2\text{O}_3$: 0.6×10^{-9} m, AlN: 0.4×10^{-9} m, and they were roughly corresponding to lattice constant.

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08-97

Some Features of Lithium Penetration into Beryllium Under corrosion Tests in Be-liquid Li-V4 Ti 4 Cr alloy system

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Self-cooled lithium-vanadium blanket is one of the promising concepts of breeding blanket for future fusion reactor. Beryllium proposed to be used in this design of blanket for providing the required tritium breeding efficiency. Corrosion behavior of beryllium in Be - liquid Li - V-4Ti -4Cr alloy system is important and at the same time not clearly understood aspect of beryllium application in fusion.

Recent experimental results on the beryllium corrosion in liquid lithium at high temperature are presented. Experiments have been performed in Be - liquid Li - V-4Ti-4Cr static system during testing for 200 - 500 hours at temperatures from 600 to 800°C. The influence of test conditions (temperature, duration, lithium purity) and beryllium characteristics (microstructure, grain size and chemical composition) on penetration of lithium into beryllium are discussed. The data obtained suggest that lithium concentration in beryllium is rather small even after annealing at 800°C. A distribution of lithium in beryllium specimens is characterized by a high inhomogeneity. The ratio of lithium concentration on the surface and in center of the specimens approaches ~ 102 – 103.

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Electrical Insulating Property of Ceramic Coating Materials in Radiation and High-Temperature Environment

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In the development of ceramic coating materials for the Li/V blanket system, irradiation effect is one of the important factors affecting the electrical insulating performance. The results of neutron and gamma ray irradiations previously performed at low temperature (< 50 °C) indicated that the degradation of the insulating property due to the radiation induced conductivity (RIC) will not prevent the candidate materials from keeping required performance (<10⁻² S/m) at high dose rate (several kGy/s) in the blanket. In the present study, relation between the RIC and temperature was examined for the prediction of the performance in a high temperature environment of the blanket.

Irradiation of gamma rays was performed at ⁶⁰Co irradiation facility of ISIR (Institute of Scientific and Industrial Research) of Osaka University. The specimens irradiated were discs (10mm dia. x 1mm) of Er₂O₃ and Y₂O₃ made

with the sintering method. Silver electrodes of 5 mm in diameter were made by vapor deposition on the surfaces for voltage supply and induced current measurement. The initial electrical conductivities were order of 10⁻¹⁴ S/m. The radiation induced conductivity (RIC) was evaluated from change in the induced current under irradiation of ~2 Gy/s. The temperature dependence of the RIC was examined up to ~550 °C.

The evaluated RIC were 5.1 x 10⁻¹¹ S/m (Er₂O₃, 2.1 Gy/s) and 3.0 x 10⁻¹⁰ S/m (Y₂O₃, 2.4 Gy/s) for the bias voltage of +250 V at room temperature. Although the conductivities without irradiations, which were order of 10⁻¹⁴ S/m at room temperature, increased by heating in low temperature region, significant change in those under irradiation (1-2 x 10⁻¹⁰ S/m) was not observed up to ~400 °C for Er₂O₃ and ~300 °C for Y₂O₃, respectively. At higher temperature, the conductivities without and under irradiation were almost same magnitude and increased with temperature. The conductivities at ~550°C were ~9 x 10⁻⁸ S/m (Er₂O₃) and ~2 x 10⁻⁶ S/m (Y₂O₃). The present results indicates that the degradation of insulating performance due to the RIC weakly depends on temperature and will not prevent the achievement of the required performance in a radiation and high temperature environment of the blanket.

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08-99

Measuring Creep Strain of Individual Li_2TiO_3 Spheroids

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Predictive capabilities for the thermo-mechanical behavior of ceramic breeder pebble beds are based on different approaches, one of which is the Discrete Element Method. This method requires characterization of the individual interparticle contact behavior, such as displacement as a function of contact force, temperature and time. As local contact stresses are higher than the ultimate compressive stress of the material, stress-dependent effects like creep and sintering between the particles cannot be predicted by available data on bulk properties. These effects are important in proposed solid breeder pebble beds for fusion reactor blankets in the projected operational temperature range between 500°C and 900°C. Although basic mechanical properties of bulk Li_2TiO_3 material are known [1], experimental data on creep behavior have never been reported in the literature, to the best of our knowledge. Extensive experimental work has been carried on to characterize the mechanical behavior of Li_2TiO_3 pebble bed assemblies as a whole [2], but the results can not be used to derive individual pebbles' contact properties.

Research is ongoing to provide experimental data on creep strain of pairs of contacting Li_2TiO_3 pebbles of spheroidal shape, roughly 2mm in diameter. The goal of the experiment is to measure creep deformation under variable

compressive forces between 3 and 15 N at temperatures up to 800°C. The equivalent compressive load on the pebbles cross sectional area is between 1 and 5 MPa. The deformation is measured with linear velocity-displacement transducers with 1 mm range and sub-micron precision. Preliminary measurements were made at room temperature before the installation of the heating equipment to determine the force-displacement characteristics of the pebbles. This behavior can be characterized as following a power-law dependence $d = AF^n$. The preliminary results showed a linear behavior, with a scattering in the exponent between 0.7 and 1.2 for different pebbles, attributed to their irregular shape. This effect is negligible at high temperatures, where the creep deformations are expected to be one order of magnitude higher. Creep deformation data for a larger sample of the same material are being collected by measuring the displacement as a function of temperature, compressive force and time. Results obtained will be compared to creep behavior of bulk materials by means of Finite Element Analysis, with material properties based on data for other Li breeder ceramics [3].

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- 2: L. Bühler, J. Riemann, Journal of Nuclear Materials 307-311 (2002) 807-810
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**Plenary Session 09 – Materials for Present and Future Nuclear Power Systems –
Lessons Learned for Fusion**

09.1

Critical Questions in Materials Science and Engineering for Successful Development of Fusion Power*

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It is the general conclusion of all national programs that the development of high-performance reduced-activation structural materials is essential for the successful development of fusion power. After more than two decades of research only three candidate materials systems appear to have the potential to meet the low activation goals: ferritic steels, V-Cr-Ti alloys, and SiC/SiC composites. At this conference, as well as past ICFRMs, the status of our knowledge and the development of these materials has been reviewed and new information has been presented. In this paper we will draw upon experience gleaned from previous programs to develop materials for high temperature structural applications to identify and discuss some of the most critical issues that must be addressed in the development of these candidate materials for fusion structural applications. We will then examine ongoing programs for development of fusion materials to identify areas in which additional research effort is presently required. Critical issues to be discussed include radiation-induced solute segregation and implications on phase stability in the development of high performance alloys/ceramics; the effects of very large amounts of helium on mechanical properties and the implications for alloy design/development; development of high temperature design methodology and incorporation of radiation effects into this methodology; the effects of radiation damage on flow localization, and the implications and approach to control the phenomena; and considerations of mass transfer and corrosion in complex fusion systems.

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09.2

Materials Degradation in Commercial Nuclear Power Reactors: Lessons Learned and Implications for Future Fusion Power Systems

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The management of materials in power reactor systems has become a critically important activity in assuring the safe, reliable and economical operation of these facilities. Over the years, the commercial nuclear power reactor industry has faced numerous “surprises” and unexpected occurrences in material behavior in the nation’s operating commercial power reactors. Mitigation strategies have sometimes solved one problem at the expense of creating another. Other problems have been successfully solved and have motivated the development of techniques to foresee problems before they occur. Other anticipated problems have never materialized. This paper will review the various successes and failures of materials in reactor systems and the technical approaches to managing materials degradation issues in the current Light Water Reactor (LWR) fleet that have occurred over the last 30 years, and explore whether there are “lessons learned” that might be applied to proactively managing the considerable materials degradation challenges in future fusion systems. The recent activity in identifying materials systems for the more demanding conditions of GEN IV reactor designs and accelerator driven systems (AFCI) will also be included. Examples of both successes and failures of materials due to the demanding operating environment will be provided with a focus on structural materials with some attention given to fuels, and with an eye toward extending lessons learned from these experiences to fusion reactor systems.

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Parallel Oral Session 10A – Materials Issues for Current and Future Fission Energy Systems

10A.1

Generation IV Reactor Integrated Materials Program

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An integrated R&D program is being conducted to study, quantify, and in some cases, develop materials with required properties for the reactor systems being developed as part DOE's Gen IV Program. The goal of the National Materials Technology Program is to ensure that the materials research and development needed to support Generation IV applications will comprise a comprehensive and integrated effort to identify and provide the materials data and its interpretation needed for the design and construction of the selected advanced reactor concepts.

- For the range of service conditions expected in Generation IV systems, including possible accident scenarios, sufficient data must be developed to demonstrate that the candidate materials meet the following design objectives:
- acceptable dimensional stability including void swelling, thermal creep, irradiation creep, stress relaxation, and growth;
- acceptable strength, ductility, and toughness;
- acceptable resistance to creep rupture, fatigue cracking, creep-fatigue interactions, and helium embrittlement; and
- acceptable chemical compatibility and corrosion resistance (including stress corrosion cracking and irradiation-assisted stress corrosion cracking) in the presence of coolants and process fluids.

Additionally, it will be necessary to develop validated models of microstructure-property relationships to enable predictions of long-term materials behavior to be made with confidence and to develop the high-temperature materials design methodology needed for materials use, codification, and regulatory acceptance.

The major materials issues for the four primary systems being considered within the U.S. Gen IV Reactor Program—the Very High Temperature Reactor, the Gas-Cooled Fast Reactor, and the Lead-Cooled Fast Reactor—will be described along with the R&D currently planned to address them.

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10A.2

Overview of PERFECT Project for Prediction of Irradiation Damage in Fission Reactor Components

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PERFECT is an Integrated Project of the 6th Framework Program of the European Union, which started on January 2004. It is devoted to the multi-scale modeling of the radiation induced modifications of the in-service properties of the low alloy steels of the Pressure Vessels, and, of the austenitic steels of the Internals, in Light Water nuclear plants. The project encompasses modeling development and experimental validation at the relevant scale.

Atom-scale modeling of the microstructure is essential, since only at this scale the physics of radiation damage is reliably described. For the low alloy steels, the main results concern the energetic of point defect clusters and interaction with the main alloying elements. Based on these data, new improved inter-atomic potentials, using different methodologies are being developed and consequences on primary damage studied. First ab initio based multi-scale modeling of radiation damage will be presented. Quantifying the point defects obstacle forces via Molecular dynamics and developing the Dynamics of Discrete Dislocations including dislocation forest and solute hardening are progressing.

Prediction of the RPV fracture toughness requires mastering the intermediate scale of the Representative Elementary Volume (REV) of the bainite structure of the steel. Progresses in defining the REV and the first modeling based on crystalline plasticity behavior will be described.

Progresses in modeling the parameters supposed to control the so-called Irradiation Assisted Corrosion Cracking of austenitic steels: (i) grain-boundary segregation, (ii) plastic flow channeling and (iii) electro-chemistry boundary conditions will finally be given.

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10A.3

Cross-Cutting Materials Issues for the Fusion and Spallation Neutron Environments

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Irradiation conditions experienced by materials for use in spallation neutron sources are extreme and may lead to severe materials degradation. The spallation neutron source is generally a high density material or fluid which is bombarded with a high energy proton flux to produce a spallation neutron flux. In most applications, the energy of the protons incident on the target ranges from 500 to more than 1000 MeV. Because of the high energy (>10 MeV) the target and structural materials can accumulate hydrogen and helium from spallation at rates up to more than 500 appm H/dpa and 150 appm He/ dpa. The time averaged displacement damage rates are similar to those in high flux reactor cores and fusion reactor first walls. Typical solid target and structural materials include tungsten, tantalum, ferritic/martensitic steels, nickel superalloys, aluminum alloys and austenitic stainless steels and liquid target materials include lead-bismuth, lead and mercury. For the solid target materials, corrosion by the water coolant must be considered. The liquid target materials add the issue of liquid metal corrosion to the displacement damage and spallation gas effects. To assess the effects of the spallation environment on the properties of prototypic spallation target and structural materials, irradiations have been performed and are presently in progress using the high energy proton beam lines at the Los Alamos Neutron Science Center (LANSCE) and the SINQ proton beamline at the Paul Scherrer Institut. The materials issues in the spallation environment strongly overlap with those expected in a fusion environment as accumulation of helium in a fusion environment can be as high as 10 appm He/dpa. Thus, recent data on the effects of high energy proton and spallation neutron irradiation on the mechanical properties will be presented and the application to irradiation effects in a fusion spectrum will be explained.

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10A.4

Materials Design of NITE-SiC/SiC for VHTR/GFR and Fusion

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SiC/SiC composites have been considered as one of potential candidates for generation IV gas reactors, such as VHTR and GFR, and fusion reactors. However, the total performance of SiC/SiC ever reported was insufficient to start large scale R & D. The recent results from NITE-SiC/SiC were excellent including results from neutron and ion irradiation experiments.

The materials requirements are quite different depending on application and design. This requires material design to meet reactor design and application target.

The effort to make large scale production with different material geometry and specification is on-going utilizing the production line of Ube Industries, Ube.

Start with the brief review of the material requirements for VHTR, GFR and fusion reactors, recent results will be presented.

The results includes, ceramics and refractory metal coating and joining of NITE-SiC/SiC and NITE-SiC.

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Parallel Oral Session 10B – Functional Materials

10B.1

Effects of Neutron Irradiation on the Properties of Functional Materials for Fusion Applications

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Functional materials should play important roles in heavy irradiation environments in fusion systems including the ITER. Irradiation effects modify properties of functional materials dynamically as well as irreversibly. Among irradiation effects, the electronic excitation effect will result in formation of electronic defects and bulk thermal heating in general. However, it sometimes results in structural changes through a radiolysis, especially when its dose rate is high. Also, the electronic excitation effect will sometime modify stability of defects complexes and increase mobility of point defects and ions. In the meantime, the atomic displacement effect will modify structures of functional materials, resulting in changes of mechanical properties and dimensions. Some properties of functional materials depend on their microstructures such as p-n junctions. In that case, the atomic displacement effect will change properties seriously with only a small dose. Also, the atomic displacement effect will enhance transport phenomena of atoms and ions by increasing concentration of thermally unstable point defects.

In fusion systems, irradiation effects of neutron will dominantly bring in property changes of functional materials. Neutron irradiation will give the electronic

excitation effect and the atomic displacement effect concurrently. Thus, to understand property changes of candidate functional materials in fusion systems, in-situ type irradiation tests is indispensable under neutron associated irradiation. Also, evaluation of the dynamic change of mobility of defects and ions will be essential. In general, the electrical conductivity evoked by the phenomenon of radiation induced electrical conductivity (RIC) is dominant under the irradiation, where the electronic excitation effect plays a major role. However, sometimes, the ionic conduction and the protonic conduction can play an important role there. Enhancement of the ionic conduction will lead to phase separations and resultantly result in the electrical breakdown. Protonic conductivity will also influence the electrical conductivity and the so-called radiation induced electromotive force (RIEMF).

The present paper will describe behaviors of electrical conductivity of candidate ceramics under the JMTR fission reactor irradiation and under the FNS 14MeV neutron irradiation. Some oxide ceramics, which have possibility of the protonic conduction, do not reveal distinct RIC under the FNS irradiation where the electronic excitation effect is marginal. Some ceramics reveals large RIEMF, which can be explained by effects of the protonic conduction. These results indicate importance of hydrogen in radiation effects in ceramics. Some ceramics revealed quasi-breakdown in the course the JMTR irradiation, which will imply enhancement of the ionic conduction.

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10B.2

Radiation Effects on the Deuterium Diffusion in Oxides

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Different oxides, mainly fused silica and aluminium oxide, are candidate materials to be used as optical or radiofrequency vacuum windows in different diagnostics critical for the safety and control systems as well as for the heating and current drive systems of ITER. These windows must also act as confinement barriers for tritium and other radioactive products. During operation they will be exposed to a low pressure mixture of D, T, He and other ions, an intense neutron and gamma radiation field, and bombardment by charged and neutral particles.

Several results in the literature indicate that the diffusion mechanism in such oxides may be modified under radiation giving rise to a large increase in the effective diffusion coefficients. To help clarify this phenomenon, radiation effects on the behaviour of the implantation profile of deuterium have been measured in a qualitative way for fused silica and aluminium oxide.

Deuterium has been introduced into the samples by low energy ion implantation (30 keV and 50 keV). Some of these samples have been then irradiated with different radiation sources at different temperatures to induce diffusion. Possible modification of the implantation profile has been determined by Elastic Recoil Detection Analysis (ERDA) using 30 MeV Si ions, by measuring the deuterium concentration and profile before and after the irradiation. It is observed that irradiation with both ionising and displacement radiation induce changes in the D profile, being the effect of displacement radiation much higher than the effect of ionising radiation.

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10B.3

H and OH Effects on Ion Beam Induced Luminescence in SiO₂

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Understanding irradiation effects on SiO₂ based materials is important from the standpoint of developing optical functional materials such as optical fibers and windows, which can be used for diagnostics in fusion devices. It is well known that doping with impurity elements can improve radiation resistance for optical absorption and can change optical properties including light emission. Luminescence measurements during ion bombardment are especially useful because they allow us to monitor the dynamic processes involved in damage creation, excitation and relaxation of the glass network. In the present work, ion beam induced luminescence was studied for SiO₂ glasses in connection with effects of H and OH.

The luminescence of the visible wavelength was measured for specimens with different H and OH contents during the proton and helium bombardment with an energy range between 2 keV ~ 2 MeV, at a temperature range between 295 and 600 K. Concentration depth profiles of hydrogen were determined by Elastic Recoil Detection Analysis, and state of hydrogen was examined by Infrared (FTIR) absorption measurements.

For MeV proton, the higher intensity of the luminescence around 450 nm were obtained for specimens with lower concentration of OH. Results for energy and dose dependence of the luminescence intensity suggested that B2alpha oxygen deficient centers were produced or activated by electronic excitation, especially for specimens with lower OH content. The intensity of the luminescence did not depend on the incident flux in a range between 10¹² and 10¹⁴ ions/m²s for MeV proton. On the other hand, the light emission efficiency slowly decreased with an increase of incident flux of keV H and He ions, indicating effects by collisional cascades. A flat distribution of hydrogen was observed in the depth far beyond the projected ranges of incident keV H ions, with a concentration of about 1 at.% at a fluence of 1 x 10²²H/m². No difference was found for thermal release behavior between originally contained H and implanted H in the interior. Effects of the implanted H on the luminescence and on the network structure will be discussed to account for damaging process during the ion bombardment.

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10B.4

Surface Electrical Degradation of Helium Implanted SiO₂

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Oxides will be used in ITER in heating and current drive and also diagnostic systems where they will play important roles as electrical insulators and optical components. These materials will be subjected to neutron and gamma irradiation, and additionally to bombardment by low energy ions and neutral particles. These low energy particles will deposit most of their energy at or very near the surface and hence the local damage and/or degradation of the physical properties at the vacuum surface could be very high. In particular degradation of electrical resistivity or optical transmission are important issues for both diagnostics and heating and current drive systems.

KS-4V (SiO₂) samples, 10x10x0.9 mm³ in size, were implanted with 54 keV helium at temperatures between 50 and 450 C up to a dose of 10¹⁷ ions/cm². The samples were mounted in a system which permitted one to implant and measure the surface electrical conductivity in high vacuum as a function of dose. After implantation, both optical absorption measurements and SEM X-ray analysis were performed.

The electrical conductivity over the SiO₂ implanted zone increases by more than seven orders of magnitude. Such severe surface electrical degradation is due to the loss of oxygen from the implanted surface. The loss of oxygen also reduces the material band gap at the surface and as a consequence the optical transmission is severely reduced. Implantation temperature plays an important role, where one observes that electrical degradation is higher and optical degradation is lower for the higher temperature.

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10B.5

Crystallization and Microstructure of Lithium Orthosilicate Pebbles

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For the European Helium Cooled Pebble Bed (HCPB) blanket slightly overstoichiometric lithium orthosilicate pebbles (Li₄SiO₄ + 2.5 wt% SiO₂) have been chosen as one promising breeder material. The lithium orthosilicate (OSi) pebbles are fabricated from lithium hydroxide (LiOH) and silica (SiO₂) by a melt-spraying process in a semi-industrial scale facility at Schott AG, Mainz.

Due to the rapid quenching of the melt, depending on the pebble size, different microstructures can be observed in the pebbles as received from the producer.

While a certain amount of the very small pebbles solidify amorphous, in the majority amount of the pebbles two phases crystallize by heterogeneous nucleation, lithium orthosilicate and the high temperature phase Li₆Si₂O₇. Microscopic investigations on cross sections reveal some pores and cracks in the crystallized pebbles in contrast to the amorphous ones.

As pores and cracks will affect the mechanical properties of the pebbles, the crystallization process of amorphous pebbles was studied as a possibility to decrease the amount of cracks and pores in the final product. Therefore amorphous pebbles were selected, and the development of phases and microstructure during crystallization was studied by thermal analysis, X-ray diffraction, and electron microscopy. Different heat treatments of the pebbles were carried out to minimize the evolution of cracks and pores.

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Parallel Oral Session 11A – H & He Effects in Irradiated Materials

11A.1

The Transport and Fate of Helium in Nanostructured Ferritic Alloys at Fusion Relevant He/DPA Ratios and DPA Rates

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Understanding, modeling and managing the effects of high levels of He and dpa on microstructural evolution and property changes is a primary objective of fusion materials research. We recently implemented an approach to producing a wide range of controlled He/dpa ratios at fusion relevant dpa rates under mixed spectrum neutron irradiation using α -implantation from Ni bearing layers from $^{58}\text{Ni}(n_{\text{th}},\gamma) \rightarrow ^{59}\text{Ni}(n_{\text{th}},\alpha)$ reactions. Thin 1 to 4 μm layers of NiAl deposited on TEM discs produce a uniform He deposition zone of 6 to 8 μm in adjacent steel specimens (note the approach can be used to implant any material). The NiAl layers were extensively characterized by precision weighing, SEM/EDS, XRD, FIB/SEM and AFM. Various model alloys and martensitic steels, as well as nanostructured ferritic alloys, were injected in the JP26 experiment in the peripheral target position in the HFIR to 5, 10 and 20 appm He/dpa ratios at a dose of 4 dpa at $T_i = 300$ and 400, and to 10, 20 and 40 appm He/dpa at 10 dpa and 500°C. The objective is to characterize the transport, fate and consequences of He and the effects He/dpa variations in alloys with a wide range of starting microstructure (e.g., grain size, cold work and second-phase particle distributions). The focus of this paper is on the fate and consequences of a He/dpa = 40 at 10 dpa and 500°C in the NFA MA957. MA957 contains a high concentration of 1-3 nm scale Y-Ti-O solute clusters as well as slightly larger (> 3 nm) $\text{Y}_2\text{Ti}_2\text{O}_7$ oxides. In particular, we explore trapping He at dislocations and nano-scale solute clusters and oxides particles. Sections were fibbed from the uniformly implanted region of the discs and examined by variety TEM techniques. The results are analyzed with a multiscale model described in a companion paper. Another companion paper describes the effects of similar He implantation in Eurofer97.

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11A.2

Retention and Desorption of Deuterium in Model Alloys of Ferritic Steel

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Retention and desorption of injected deuterium into model alloys of ferritic steel were investigated by using TDS, XPS, and TEM complementary. Three model alloys (Fe-5Cr, Fe-9Cr, Fe-15Cr) and Pure-Fe were prepared as test materials. After rolled to 0.2 mm thickness, some of them were annealed at 1023 K for 2 hours to reduce dislocations. As-rolled and annealed specimens were irradiated at room temperature with 8keV-deuterium ions up to the fluence of $3 \times 10^{21} \text{ D}_2^+/\text{m}^2$, and successively thermal desorption of D_2 and HD under the constant heating rate (1K/s) was measured using quadruple mass spectroscopy.

In general, retention of deuterium in the rolled Fe-Cr alloys was quite low and most of them desorbed up to 700 K. Remarkable desorption was observed even at room temperature, which indicates the existence of rather weak traps. The retention decreased in the annealed specimens. This fact indicates that the dense dislocations provide good trapping sites for deuterium diffusing deeply into the specimen. Detrapping from the dislocations occurs around 550-650 K. A special feature of the Fe-Cr alloys was the remarkable decrease of the retention above the fluence of $3 \times 10^{21} \text{ D}_2^+/\text{m}^2$. The composing elements and their chemical states at the surface were examined by XPS. It was found that the surface of the alloys was covered with oxide layer of about 7 nm thickness, which is just the thickness of sputtering erosion by the irradiation of 8 keV- D_2^+ for $3 \times 10^{21} \text{ D}_2^+/\text{m}^2$. Taking into account that 8 keV- D_2^+ is injected in the much deeper region (projected range is about 30 nm), it is likely that the surface oxide layer acts as desorption barrier and once the layer is removed by the sputtering desorption during and after irradiation occurs actively. As a result, retention of deuterium decreases above $3 \times 10^{21} \text{ D}_2^+/\text{m}^2$ as observed. Interstitial type dislocation loops are formed in the narrow subsurface region. But their density and size are too low to explain the retention of the deuterium, namely, these defects don't work as main deuterium trapping site.

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11A.3

Effect of Implanted Helium on Flow and Fracture Properties of 9Cr Martensitic Steels*J. Henry¹, L. Vincent¹, X. Averty¹, B. Marini¹, P. Jung²*¹CEA, F-91191 Gif-sur-Yvette, France; ²FzJ, D-52425 Jülich, Germany

In addition to radiation induced embrittlement of 9Cr normalised and tempered martensitic steels, the potential role of high helium contents accumulated in first wall and blanket structures of fusion reactors is a major concern. The detrimental role of helium at high temperature on the mechanical properties of austenitic and martensitic steels is well documented. However, helium effect at low temperature, typically below $0.4 T_m$, (where T_m is the melting temperature) is still a matter of controversy.

This paper presents an experimental characterization of helium effects on the mechanical properties of 9Cr martensitic steels using the implantation technique. Tensile tests performed on 100 μm thick specimens homogeneously implanted with 600 appm up to 5000 appm He at temperatures below 550°C, had shown that helium can induce a drastic ductility loss together with an intergranular fracture mode depending on the helium content and implantation temperature. Based on microstructural characterization by Transmission Electron Microscopy and Small Angle Neutron Scattering of the implantation-induced microstructure, it had been proposed that the brittle intergranular fracture mode results from the combined effects of pronounced intergranular hardening and weakening of prior austenite grain boundaries due to helium. In order to quantitatively characterize helium effects on the fracture properties of 9Cr martensitic steels, a set of fracture experiments was performed: subsized Charpy specimens were implanted in the notch with 34 MeV ³He particles then tested at room temperature in bending under quasi-static loading followed by fractographic examinations by Scanning Electron Microscopy. Finite elements simulations of the tests were carried out using as input data the experimental tensile behaviours of the steel in both the pristine and implanted conditions. These computations have allowed to determine the fracture stress values for the onset of crack propagation as a function of helium content and implantation temperature.

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11A.4

The Transport and Fate of Helium in Martensitic Steels at Fusion Relevant He/DPA Ratios and DPA Rates*R.J. Kurtz^a, G.R. Odette^b, T. Yamamoto^b, D.S. Gelles^a, P. Miao^b, B.M. Oliver^a*^aPacific Northwest National Laboratory, Richland, WA 99354 USA; ^bUniversity of California, Santa Barbara, CA 93106-5070 USA

Understanding, modeling and managing the effects of high levels of He and dpa on microstructural evolution and properties changes is a primary objective of fusion materials research. We recently implemented an approach to producing a wide range of controlled He/dpa ratios at fusion relevant dpa rates under mixed spectrum neutron irradiation using in-situ α -implantation from Ni bearing layers from ⁵⁸Ni(n_{th},g) \rightarrow ⁵⁹Ni(n_{th},a) reactions. Thin 1 to 4 μm layers of NiAl were deposited on TEM discs producing a uniform He deposition zone of 6 to 8 μm in adjacent steel specimens (note the approach can be used to implant any material). The NiAl layers were extensively characterized by precision weighing, SEM/EDS, XRD, FIB/SEM and AFM. Various model alloys and martensitic steels, as well as nanostructured ferritic alloys were injected to <1, 5, 10 and 20 appm He/dpa ratios at dose of 4 dpa at $T_i = 300$ and 400, and <1, 10, 20 and 40 appm He/dpa to 10 dpa at 500°C as part of the JP26 experiment in the peripheral target position in the HFIR. The irradiation matrix is broadly aimed at characterizing the transport, fate and consequences of He and the effects He/dpa variations for alloys with a wide range of starting microstructure (e.g., grain size, cold work and second-phase particle distributions). In this study we specifically explore the effect of the highest He/dpa (≈ 20 to 40 appm He/dpa) and T_i on the microstructure of Eurofer97. Cross section specimens were prepared by attaching 3 mm diameter Cu sections to both sides of the disk followed by sectioning and thinning by Ar ion milling to produce electron transparent regions in both implanted and unimplanted parts of the specimen. The thinned specimens were examined in a JOEL 2010FE microscope under a variety of imaging conditions. Bubbles were found in the implanted region at all three T_i , with estimated maximum diameters of ≈ 10 , 6.5 and 2.5 nm at 500°C (≈ 10 dpa and 380 appm He), 400°C (≈ 4.3 dpa and 90 appm He) and 300°C (≈ 4.3 dpa and 90 appm He), respectively. At the 500°C 10nm faceted cavities were observed, that may actually be voids. The corresponding minimum bubble size ranged from ≈ 1.5 to 2 nm, near the resolution limit. The other irradiated microstructures were also characterized, with special emphasis on the association of bubbles with other features. Low-load microhardness measurements were used to assess the effects of He/dpa and T_i on irradiation induced strength elevations.

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The Behavior of Martensitic Steels at High Irradiation Dose and Helium Concentration – an Overview of STIP Results

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Martensitic steels are candidate materials for the first-wall blankets of fusion reactors and structural materials in high-power spallation targets as well. In both applications, the helium production in the steels is significant and may introduce serious degradation of mechanical properties, particularly in the case of spallation targets. To understand helium effects on these steels is of essential importance for both of fusion and spallation materials communities. SINQ target irradiation program (STIP) has provided a good opportunity for such studies, in which martensitic steels such as T91(9Cr-1MoVNb), HT-9, MANET-II, F82H, Optifer, Eurofer-97 have been irradiated up to 20 dpa and about 1700 appm He in a temperature range of 100 to 450°C. The post-irradiation examinations have been conducted and some of T91, HT-9, F82H and Optifer samples have been investigated. Tensile tests demonstrate a significant hardening and reduction of

ductility for all samples irradiated at $\leq 300^\circ\text{C}$. Some samples of about 19 dpa and 1600 appm He irradiated at about 350°C show also brittle behavior in testing at 400°C . On the hand, Charpy impact and small punch tests illustrate enormous DBTT-shift at high doses and He concentrations. The DBTT-shift does not saturate in dose range of 1-5 dpa as normally observed by neutron irradiated samples, while increases with dose and helium concentration. The maximum DBTT-shift detected is over 400 K. To understand the mechanical testing results, microstructural analyses have been carried out using different techniques such as transmission electron microscopy (TEM), small angle neutron scattering (SANS) and anomalous small angle X-ray scattering (ASAXS). Both TEM and SANS investigations demonstrate helium bubbles formed in the samples. At high doses and high helium concentrations, bimodal helium bubble formation was observed. ASAXS analysis reveals that Cr-rich α' phase also formed in high dose samples. In addition, helium and hydrogen release measurements are been performed using thermal desorption spectrometry (TDS) technique, which will provide the information about helium and hydrogen trapping in the irradiated steels.

In the presentation, discussions will be focused on radiation and helium effects. The differences and similarities of the conventional steels (T91 and HT-9) and reduced activation steels (F82H and Optifer) will be also discussed.

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Parallel Oral Session 11B – SiC Composites

11B.1

Current Status and Critical Issues for Development of SiC Composites for Fusion Applications

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Silicon carbide (SiC)-based ceramic composites have been studied for fusion applications for more than a decade. The potential for these materials have been widely discussed and are now understood to be: 1) the ability to operate at temperature regimes much higher than metallic alloys, 2) an inherent low level of long-lived radioisotopes reducing the radiological burden of the structure, and 3) perceived tolerance against neutron irradiation up to very high temperatures.

The primary focus of attention in the fusion materials community towards the development of SiC composites has been to address the fusion-specific key

feasibility issues. Most importantly, fundamental performance of SiC composites under irradiation has been demonstrated through various neutron irradiation campaigns. Fundamental effects of irradiation, including the effect of transmuted / implanted helium, on mechanical and thermo-physical properties and microstructural development in composites and the constituents have been studied extensively. At the same time, development of novel materials and optimization of conventional materials for nuclear applications were pursued. Additionally, design issues such as the need to develop leak-tight components, the need for joining the materials with low-activation additives, and issues regarding coolant / breeder compatibility have been addressed.

The purpose of this paper is not only to review the recent progress in the area of SiC composite development for fusion, but to make the case that SiC composites are progressing from a stage of proof-of-principal to one that has been defined sufficiently and is ready for system demonstration. Also addressed in this paper is the overall status of SiC composites outside of fusion, such as the Generation IV nuclear power plant, where SiC composites are being tested and validated for control rod structural application. Finally, the application of these materials in the Test Blanket Module for ITER will be presented.

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11B.2

Mechanical Characterisation of Commercial Grade Tyranno SA/CVI SiC Composites*B. Riccardi¹, M. Labanti², E. Trentini², S. Roccella¹, E. Visca¹*¹ENEA C.R. Frascati, POB 65, I-00044 Frascati (RM) –Italy; ²ENEA C.R. Faenza, I-48018 Faenza (RA) –Italy

SiCf/SiC ceramic matrix composites are candidate materials for fusion reactors because of their good mechanical properties at high temperature, chemical stability and radiation resistance. Objective of the present work, in the framework of the European fusion material programme, was the mechanical characterization of 2D composites manufactured by using Tyranno SA fiber performs infiltrated by CVI and finally SiC coated by CVD.

Low cycle flexural fatigue behaviour has been investigated at RT and 1000°C by means 4-point bending tests performed at a typical frequency of 1 Hz in argon atmosphere. The applied stress value was above and below the levels necessary to cause matrix cracking. The specimens have been cut from large SiC/SiC plates and their size (80 x 10 x thickness) allows for a good accuracy of results.

The creep behaviour was investigated by means of constant load stress rupture test still by using a 4-points bending tests but reduced specimens size. The tests have been carried out in controlled atmosphere at 600 and 1000°C up to 1000 h. The possibility of using a protective coating to avoid the detrimental effect of oxidation was investigated.

The results have shown that the stress level above or below the composite non-linear stress-strain behaviour begins is an important factor for the control of the composite overall performances and the properties at high temperature are significantly reduced from those at room temperature. Microstructural examination was performed on the fractured specimens and it revealed the composite failure mechanisms that appeared under cyclic and static loading.

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11B.3

Elevated Temperature Swelling of Neutron Irradiated SiC*L.L. Snead¹, Y. Katoh, and S. D. Connery*

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Under neutron irradiation silicon carbide (SiC) undergoes significant, temperature dependent swelling. This swelling can be broken into three separate regimes. For temperatures less than ~ 180°C interstitial mobility is limited and the crystal reaches a critical dose threshold for amorphization resulting in a volume increase of 10.8 %. Other physical properties such as strength, fracture toughness and thermal conductivity also undergo substantial changes. Above this temperature, perhaps due to both silicon and carbon interstitials becoming mobile, swelling is dominated by simple point defects and small defect clusters resulting in swelling dropping from ~ 3% to near zero for irradiation temperature of ~1000°C. Moreover, swelling in this regime results in little mechanical property change with thermal properties showing only moderate degradation at the higher temperature. Above around 1000°C swelling of SiC is not well defined, though appears to enter into a new regime where interstitial loops likely dominate swelling. It is expected that in this regime swelling will not saturate and will go through a temperature of maximum swelling.

The definition of the upper swelling regime for SiC is particularly important in that it currently defines the assumed upper operating temperature for using SiC/SiC in fusion power devices, nominally chosen as 1000°C. This paper will present results from a neutron irradiation experiment ranging in temperature from the middle of the point-defect swelling regime (~ 600°C) well into the upper swelling regime (~1500°C). Results will be presented on swelling, microstructure, and thermal conductivity for chemically vapor deposited, single crystal SiC, and SiC/SiC composites.

11B.4

Irradiation Effect on NITE-SiC/SiC Composites

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Silicon carbide and SiC composites are significantly attractive materials for nuclear application in particular due to exceptional low radioactivity, excellent high temperature mechanical properties and chemical stability. Despite of the excellent potential of SiC/SiC composites, the prospect of industrialization has not been clear mainly due to the low productivity and the high material cost. Chemical vapor infiltration (CVI) method can produce the excellent SiC/SiC composites with highly crystalline and excellent mechanical properties. It has been reported that the high purity SiC/SiC composites reinforced with highly crystalline fibers and fabricated by CVI method is very stable to neutron irradiation. But the production cost of the CVI method is high. The novel processing called Nano-powder Infiltration and Transient Eutectic Phase (NITE) Processing has been developed based on the liquid phase sintering (LPS) process modification. The NITE processing can achieve both the excellent material quality and the low processing cost. The productivity of the processing is also excellent, and various kinds of shape and size of SiC/SiC composites can be produced by the NITE processing. The objective of this work is to understand irradiation effect of the NITE-SiC/SiC composites.

The SiC/SiC composites used were reinforced with high purity SiC fibers, Tyranno™ SA and fabricated by the NITE method. The NITE-SiC/SiC composites were irradiated at 4.2 dpa and 1000 °C. Mechanical properties of non-irradiated and irradiated NITE-SiC/SiC composites were evaluated by tensile test. The fracture surface was examined by SEM. Although both the non-irradiated and the irradiated NITE-SiC/SiC composites showed brittle fracture behavior like monolithic ceramic due to the lack of fiber/matrix interphase, the tensile strength of the NITE-SiC/SiC composites increased following the neutron irradiation. The mechanism of increasing of the strength will be discussed with microstructural characterization.

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11B.5

Strength of Neutron Irradiated SiC/SiC Composite with Multilayer SiC/PyC Interface

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Silicon carbide (SiC) matrix composites are being developed for structural applications in fusion systems for their promise of high-temperature performance and low-induced activation. In recent years the stability of highly crystalline fibers has led to composites with very good irradiation stability. However, the effect of high-dose irradiation on the fiber/matrix interface, typically thick (200 nm) carbon, remains as a potential critical issue. In non-nuclear applications, a multilayer interphase composed of sequences of the very thin (<50 nm) pyrolytic carbon (PyC) and SiC results in good composite strength and oxidation resistance. The fundamental question is whether the anisotropic irradiation-induced swelling of these thin graphite layers will impact composite performance. The primary objective of this study is to determine the benefit of moving to the multilayer SiC interphase and the potential for very high-dose irradiation performance of this system.

Neutron irradiation was carried out on multilayer interphase composites fabricated from the irradiation-stable Hi-Nicalon™ Type-S fiber composite. The matrix was chemically vapor infiltrated SiC. The dose and temperature range was 0.1~7.7 dpa and 873~1573 K, respectively. The effect of irradiation on the tensile and interfacial shear properties was evaluated by cyclic loading tensile and push-in/-out methods.

Tensile result of the multilayer interface SiC/SiC composites indicated no significant degradation of tensile modulus and proportional limit failure strength after neutron irradiation up to 1 dpa at 1073~1273 K. However, a slight increase of ultimate tensile strength (UTS) was obtained. The increase of UTS is attributed to a small degradation of the multilayer interface. This observation is supported by noting a change in the fracture pattern from brittle to quasi-ductile failure after neutron irradiation, resulting in the better performance of the intact fibers. This study will provide quantitative data of interfacial parameters after neutron irradiation and detailed failure mechanism of the multilayer interface SiC/SiC composites.

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Poster Session 12

Investigations of Helium Effects on Cavity Evolution in Metals under Cascade Damage Conditions

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The concurrent generation of helium and cascades in metals and alloys under fusion irradiation conditions gives rise to a serious concern regarding the performance and lifetime of materials used in the structural components of a commercial fusion reactor. This concern stems from the considerations that at elevated temperatures the continuous production of helium may enhance swelling in the grain interior and may cause accumulation of helium at grain boundaries which may lead to grain boundary embrittlement. In recent years, significant progress has been made in understanding swelling behaviour in metals under cascade damage conditions within the framework of the Production Bias Model (PBM) which takes into account the production and one-dimensional diffusion of clusters of self-interstitial atoms. However the problem of helium assisted cavity evolution has not yet been treated systematically and quantitatively within the framework of the PBM. The present work addresses this problem.

In order to establish a proper understanding of the effect of helium on the evolution of cavity microstructure under cascade damage conditions, detailed numerical calculations based on a two-dimensional kinetic equation for the size distribution function of helium-vacancy clusters have been carried out for pure iron irradiated with neutrons, high energy helium ions and for the case when neutron irradiation has been occurred following after helium pre-implantation at the same temperature. A newly developed grouping method has been used to integrate the kinetic equation. Calculations have been carried out within the framework of the PBM taking into account the main mechanisms for diffusion of atomic He under irradiation, such as the interstitial mechanism, SIA-He replacement mechanism and He-divacancy mechanism. Furthermore, the calculations also evaluate the impact of Brownian-like motion of small He-vacancy clusters on the evolution of cavity nuclei. The calculated results are compared with the results of simple analytical calculations. For the case of low temperature irradiation (around the recovery stage III) the results are found to be in good accord with experimental results. Some preliminary results calculated for the same irradiation conditions and temperatures above the stage V are also discussed.

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12-3

Thermal Helium Desorption of Helium-Implanted Iron

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Ferritic martensitic steels will experience severe irradiation induced degradation of many important performance sustaining mechanical properties in fusion environments, driven by simultaneous production of displacement defects and high concentrations of helium. A key issue is coupled transport and fate of all defect, gas and solute species. In this work, we focus on determining the mechanisms of helium interaction, trapping and migration mechanisms in iron and ferritic alloys. Thermal helium desorption spectroscopy (THDS) measurements have been performed on nominally pure iron specimens, implanted with helium under different conditions. Helium-implantations were performed at energies from 20 to 500 keV and at doses from 10^{11} cm⁻² to 10^{14} cm⁻² on iron specimens as a function of grain size and dislocation density. The experimental results yield the desorption temperature, the activation enthalpy for desorption, the attempt frequency for desorption, and an indication of the types of defects from which helium is desorbing. The experimental results are compared with recent molecular dynamics and kinetic Monte Carlo simulations on the energetics and migration mechanisms of helium, and its interactions with point defect clusters and extended defects in iron.

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12-4

Characteristics of Traps for Hydrogen in Helium-irradiated Copper

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Radiation damages in metals produce deep potential sites, which would act as traps for hydrogen isotopes. For evaluation of the tritium inventory, characteristics of the traps in helium-irradiated copper were experimentally studied. One side of a pure copper membrane was continuously exposed to deuterium plasma in the whole experiment. The plasma-facing side was irradiated by 0.8-MeV ³He⁺ ions with a dose of 1.5×10^{21} m⁻². Before and after the irradiation, deuterium depth profiles were observed by nuclear reaction analysis with the reaction of D(³He,p)⁴He.

The result showed that a large amount of the traps were produced by the irradiation and the deuterium concentration increased by 10,000 times at 398 K. Evolution of the shape of the deuterium depth profile with the membrane temperature indicated that there existed two types of the traps, called trap1 and trap2, here.

Assuming quasi-equilibrium for deuterium atoms between the solution site and the trapping site, the enthalpy difference in the two sites was estimated to be about 0.7 eV for trap 1 and 0.6 eV for trap 2 from the deuterium concentration and the deuterium permeation flux through the membrane. The averaged production rate of the trap to the number of displaced host atoms was estimated to be 1×10^{-3} for trap 1 and 3×10^{-4} for trap 2. The traps were not annihilated at 575 K. From these results, it was concluded that the tritium inventory in pure copper would not be so significant at elevated temperatures.

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12-5

Kinetic Monte Carlo Simulations of Substitutional Helium Diffusion

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Highly mobile vacancy-helium cluster complexes are likely to play a key role in intergranular and intragranular helium bubble nucleation which impacts void swelling and high-temperature intergranular embrittlement. We present a kinetic Monte Carlo (KMC) model to simulate the long-term aging evolution of insoluble helium produced at high rates in nuclear (n, α) reactions in Fe. Our KMC simulations investigate the migration and clustering of He and the mechanisms of vacancy-helium cluster formation and mobility to predict helium bubble nucleation through cluster coalescence events.

The dimer method was used to obtain the activation energies of the saddle points for all of the vacancy – atom exchanges in the vicinity of small helium-vacancy clusters, using semi-empirical Fe-He potentials. The activation energies are found to vary from 0.02 eV for substitutional He exchange with a vacancy to about 1.1 eV for vacancy – iron exchanges that move the vacancy from a first to third nearest neighbor position from one of the helium atoms. The results for small vacancy – helium cluster migration are compared to available analytic theories of cluster diffusion and experimental data obtained from thermal helium desorption spectroscopy.

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12-6

Effect of Helium on Mechanical Properties in HIP-Bonded Reduced-Activation Ferritic/Martensitic Steel

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Reduced-activation ferritic/martensitic steel (RAF/Ms) is the first candidate structural material for a blanket which will be installed in a fusion reactor like DEMO reactor. F82H steel is one of the RAF/Ms, and the blanket of DEMO reactor will be fabricated by using RAF/Ms. The blanket has complicated structure and it is very difficult to fabricate by welding joints only. The structural part is therefore fabricated by diffusion bonding method like hot isostatic pressing (HIP) bonding method. It needs to examine the changes of mechanical properties of the HIP-bonded specimens due to irradiation and helium produced by nuclear transmutation reactions. The tensile properties of HIP-bonded F82H steel irradiated by neutrons were reported. In this study, effect of helium on mechanical properties of F82H steel for the HIP-bonded region is discussed.

Disk-type specimens (3 mm in the diameter, 0.28-mm-thick) were sampled from HIP-bonded region in a test blanket partial mock-up made from F82H steel. He⁺-ions with 50 MeV by a cyclotron accelerator were uniformly implanted in the specimens, up to about 85 appm (and 0.03 dpa), using by an energy degrader and scanners. After the helium implantation, the SP (small punch) tests were performed at temperatures from about -100 K to 973 K, and the fracture regions of the specimens were observed by a SEM.

In the non-helium implanted specimens, the maximum load decreased gradually at the temperature range of a room temperature (RT) to about 750 K, and it decreased largely at higher temperature range (750 K ~ 973 K). On the other hand, the large decrease was not seen at 750 K ~ 973 K in the helium-implanted specimens. The maximum load of helium-implanted specimen was about 2.5 times higher than that of the non-helium implanted one, at 973 K. Although it seemed that the maximum load of the helium-implanted specimen is recovered due to elevated temperature, especially at 973 K, it was notably higher than that in the non-helium implanted one. Fracture energy of helium-implanted specimen was comparable to that in the non-helium implanted one, at RT ~ 973 K.

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12-7

Effect of Irradiation Damage up to 50 dpa on Swelling Behavior and Microstructural Development in SiC/SiC Composites

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Continuous silicon carbide (SiC) fiber reinforced SiC matrix (SiC/SiC) composites are known to be attractive candidate materials for first wall and blanket components in fusion reactors. In the fusion environment, helium and hydrogen are produced and helium bubbles can be formed in the SiC by irradiation of 14-MeV neutrons. Authors reported the synergistic effect of helium and hydrogen as transmutation products on swelling behavior and microstructural development in the temperature range of 800 to 1300 °C up to 10 dpa. However, the SiC/SiC composites for fusion reactors might be irradiated to more than 50 dpa at the end of life. In this study, the effect of irradiation damage up to 50 dpa on swelling behavior and microstructural development in SiC/SiC composites was investigated.

SiC/SiC composites were irradiated by the simultaneous dual/triple ion irradiation (Si²⁺ and He⁺ / Si²⁺, He⁺ and H⁺) at 1000 °C. The displacement damage was induced by 6.0-MeV Si²⁺ ion irradiation to the range of 1 to 50 dpa. The microstructures of irradiated SiC/SiC composites were observed by TEM.

No He bubble and no microstructural change were observed in SiC/SiC composites irradiated to 1 dpa at 1000 °C. On the other hand, He bubbles were observed in every SiC/SiC composite irradiated to more than 5 dpa at 1000 °C. The average size of He bubbles was increased with increasing the irradiation damage. Almost the all He bubbles were formed at the grain boundaries in the matrix irradiated up to 10 dpa. On the other hand, He bubbles were also formed in the grains of the matrix irradiated up to 50 dpa.

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12-8

Atomistic Modeling of Helium Interacting with Dislocations in Alpha-Iron

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The presence of helium in fusion reactor materials is nearly inescapable. An important first step in mitigating its deleterious effects on the mechanical properties of fusion materials is to understand the fate of helium with respect to its interaction with various microstructural features. Molecular statics, molecular dynamics and the dimer method of potential surface mapping are used to study the fate of helium in the vicinity of $a/2\langle 111 \rangle\{110\}$ edge dislocations and $a/2\langle 111 \rangle$ screw dislocations in alpha-iron. Interstitial helium atoms can easily migrate to dislocations, where they are strongly bound. Near the edge dislocation He interstitials assume crowdion positions in the layer of atoms at the slip plane, and they can diffuse along the dislocation with migration energies of 0.4 – 0.5 eV. The binding and migration energies of helium correlate strongly with excess volume in the edge and screw dislocation cores. Helium at jogs, helium clusters and helium-divacancy complexes are also examined.

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Effectiveness of Helium Bubbles as Traps for Hydrogen

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Peculiarities of bubble microstructure evolution and ion-implanted helium and hydrogen behavior were investigated in ferritic-martensitic Cr12MoWSiVNbNB (EP-900) and austenitic Cr18Ni10Ti steels irradiated by 40-keV He⁺-ions up to a fluence of $5 \cdot 10^{20} \text{ m}^{-2}$ in the temperature range of 570–900 K. Unirradiated samples and samples with a previously created helium porosity were implanted by 25-keV H⁺-ions up to a fluence of $5 \cdot 10^{20} \text{ m}^{-2}$ at room temperature. The samples microstructures were studied by transmission electron microscope and the hydrogen content was determined by the method of reducing melting in vacuum.

It is found that overpressured helium bubbles are formed up to a temperature about 820 K and pre-equilibrium bubbles – at higher temperatures in both the steels. The characteristic property of bubbles evolution in ferritic-martensitic steel is their preferred distribution on dislocations. Therefore, the helium bubbles in the BCC lattice are not effective traps for hydrogen because of even absorbed by helium bubbles hydrogen atoms easily leave the samples along the dislocations. Hydrogen is retained significantly greater in austenitic steel with a homogeneous bubble distribution in matrix at that the quantity of the trapped hydrogen substantially increases with decreasing the helium pressure in the bubbles. At the same time it is shown that hydrogen is trapped in the samples without helium by order of magnitude greater than that in samples irradiated by He⁺-ions. In other words the neutral sinks in the steels (structural defects, second phases boundaries et al.) are more effective traps for hydrogen than helium bubbles.

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12-10

Effects of Precipitation Microstructures on Bubble Distribution in Helium-Embrittled Fe-Ni-Cr Alloys

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In fusion reactors, helium embrittlement has been considered to be one of the most critical issues which determine the upper limit temperature of first wall/blanket structures. A lot of researches have been hence conducted on this problem for many years and it has been elucidated that the stated phenomenon is strongly influenced by helium bubble microstructure. In this study, relations between material microstructures and bubble distributions were investigated through detailed and quantitative TEM observation on helium-embrittled materials with a special emphasis on the effect of precipitate structures, in order to obtain information beneficial for future alloy development.

The microstructural data used were sampled from helium-implanted and creep-ruptured 316 type stainless steels and Fe-25%Ni-15%Cr alloys. For the purpose of simulating transmutational generation of helium in the reactor, it was implanted into them at 873 or 923 K by α -particle irradiation at a cyclotron.

Cumulative helium concentrations and implantation rates were varied from 50 to 1200 appm and from 4×10^{-4} to 3×10^{-2} appm/s, respectively. Creep rupture tests were performed in either in-situ or post implantation mode at the same temperature as that of corresponding irradiation under applied stresses between 120 and 470 MPa.

The amount of helium preserved in grain interiors was only weakly dependent on the number density and the total interface area of intragranular precipitates. Excessive enhancement of precipitation in the matrix, therefore, seems to be not so much advantageous. Bimodal size distribution of helium bubbles was often discerned both in the matrix and at grain boundaries. Its origin in the latter location could be attributed to rather sparse dispersion of intergranular precipitates and be a reason of severer embrittlement. The number density of grain boundary bubbles increased when the axis of applied stress approaching the surface normal to a grain boundary. This result agrees well with the fact that cracking of helium-embrittled materials occurs primarily at grain boundaries nearly perpendicular to the applied stress.

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12-11

Irradiation Embrittlement of Martensitic Steels: Hardening and Non-Hardening Mechanisms and the Effect of Helium

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Data on irradiation embrittlement of 7-9Cr tempered martensitic steels has been compiled from the literature, including results from neutron, spallation proton (SP) and He-ion (HI) irradiations. Simple, phenomenological-empirical fitting models were used to assess the dose (dpa), irradiation temperature (T_i) dependence of sub-sized Charpy V-notch impact transition temperature shifts (ΔT_c) and a smaller dataset for master toughness curve reference temperature shifts (ΔT_0). A analysis of paired ΔT_c or ΔT_0 - $\Delta\sigma_y$ datasets show that embrittlement is dominated by a hardening mechanism below about 400°C, at least up to some level of He. However, the Charpy shift coefficient, $C_c = \Delta T_c / \Delta\sigma_y \approx 0.4 \pm 0.2$ is lower than that for fracture toughness $C_k = \Delta T_0 / \Delta\sigma_y \approx 0.6 \pm 0.1$, indicating that sub-sized Charpy tests may provide *non-conservative* estimates of embrittlement. The C_c increases at $T_i \geq 400^\circ\text{C}$, indicative of a possible non-hardening embrittlement (NHE) contribution. Analysis of thermal aging ΔT_c data supports this conclusion, and the NHE regime may be shifted to lower temperatures by radiation-enhanced diffusion. Variations in the hardening-shift coefficient, $C_c = \Delta T_c / \Delta\sigma_y$, are used to evaluate potential non-hardening helium embrittlement (NHHE) contributions to ΔT_c at temperatures from 300 to 400°C. The limited, scattered, and potentially confounded data collectively suggest that there is a minimal NHHE up to a few hundred appm. However, a NHHE contribution appears to emerge at higher helium concentrations, estimated to be more than 400 to 600 appm. The NHHE is accompanied by a transition from transgranular cleavage to intergranular fracture (IGF) at high $\Delta\sigma_y$. Synergistic combinations of high $\Delta\sigma_y$ and severe NHHE weakening of grain boundaries could lead to very large $\Delta T_c \geq 400^\circ\text{C}$ in first wall and blanket structures at fusion spectrum dose levels above 50 to 75 dpa. Limitations of this database and analysis are briefly described and a micromechanical model for NHHE is proposed.

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12-12

The study of interaction between dislocation and helium atom

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The irradiation of first wall materials in a fusion reactor with 14 MeV neutrons will not only create displacement damage but also generate hydrogen and helium at high rates. Especially for helium, it seems to be inevitable that it will form many kinds of helium-defects complexes, since it is insoluble in most metals and alloys. Our previous thermal helium desorption spectra showed that helium atoms were desorbed at around 800 K from cold worked iron samples. The peak height gradually increased with increasing dislocation density, suggesting the existence of helium-dislocation complex. The formation of the helium-dislocation complex could be one of critical issues for microstructural development and mechanical property changes during irradiation, since dislocations are generally known to play an important role, not only on void formation via the so-called dislocation bias, but also on the ability of material's deformation via dislocation movements. In the present study, theoretical and computational work was done to investigate interactions between a dislocation and a helium atom, which will provide a profound insight into the possibility of the formation of helium-dislocation complexes.

The interaction energy of an interstitial helium atom under a dislocation elastic field was calculated by elastic theory. The interaction is attractive at the tensile side of an edge dislocation, while it is repulsive at the compression side. On the other hand, for a screw-dislocation, the elastic field is isotropic around the dislocation line, and the interaction is very small but always attractive. The effective capture radius of an edge-dislocation to an interstitial helium atom is much larger than that of a screw-dislocation. It indicates that helium atoms far from an edge-dislocation may be more preferentially absorbed than that from a screw-dislocation.

A molecular dynamics simulation was performed to investigate the binding state between an edge-dislocation and a helium atom. The calculated binding energy of a helium atom to an edge-dislocation was 2.3 eV. This value is consistent with the experimental annealing temperature 800 K. Accumulation of helium atoms to edge-dislocation will be discussed.

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12-13

Effects of interstitial impurity on behavior of helium-defect complexes in vanadium studied by THDS

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Vanadium alloys are considered to be one of the leading candidates for the fusion reactor blanket material. Although vanadium has many attractive features for the fusion application, its use at high temperature will be limited by the helium embrittlement. Effects of the helium on the macroscopic mechanical properties have been studied rather extensively, while the evolution processes of helium-defect complex, especially during earlier stage, is not clearly understood. This is mainly because of the complexity of helium behavior due to the large amount of interstitial impurities (C, N, O etc.) in vanadium. The objective of present paper is to investigate effects of impurities on helium clustering behavior using THDS (Thermal Helium Desorption Spectrometry) which is one of the most effective techniques to study He-V-X(X=C,N,O etc.) type complexes.

Specimens used were pure vanadium. Oxygen and Nitrogen contents in samples were systematically controlled by regulating the atmosphere during annealing or by using Zr-treatment technique (O: 140-1600, N: 430-2600appm). THDS were performed in the following manner to examine the nature and behavior of helium-defect complexes. First, helium implantation at the accelerating voltage of 1keV(defect induced) were conducted up to 3.2×10^{17} ions/m², then the specimen was heated to 1800K at a constant rate of 40K/s while monitoring the release rate of helium gas with quadropole mass spectrometer.

The observed desorption peaks were assumed to be concerned with vacancy type defects which are one of the effective trap sites of helium in metals. Single helium atom trapped by a mono-vacancy is the simplest helium cluster in metals. In vanadium, however, it has been reported in earlier papers that most of vacancies are decorated by interstitial impurities, mainly oxygen, and implanted helium produces He_nV_nX-type defects (X=C, N, O). The desorption peaks of 550K, 700K and 940K were assigned as He_nOV, He_nOV₂ and He_nOV_m, respectively. Population of these peaks increased with oxygen concentration. On the other hand, some peaks which were independent of oxygen concentration were deduced as oxygen free defect clusters such as He_nV_n.

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12-14

Desorption of Tritium and Helium from High Dose Neutron Irradiated Beryllium

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The effect of high dose neutron irradiation on tritium and helium release behavior in beryllium is described in this paper. Beryllium specimens were irradiated in the SM and BOR-60 reactors with neutron fluence ($E > 0.1$ MeV) $(5-16) \times 10^{22}$ cm⁻² at 70-100°C and 380-420°C. Mass-spectrometry technique was used in out of pile tritium release experiments during stepped annealing within 250 - 1300°C temperature range. The total amount of helium accumulated in irradiated beryllium samples varied from 6000 appm to 7200 appm.

The first signs of tritium release were detected at temperature of 312 - 445°C and helium – at 500 - 740 °C. The temperature of the maximal rate of tritium release agrees with the upper temperature of tritium release (745-775°C). It was shown that the tritium release with a maximal rate is followed by a sharp acceleration of helium release rate that could be considered as an evidence of co-existence of partial amounts of tritium and helium in common bubbles.

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The Influence of Hydrogen on the Fatigue Behaviour of Base and GTA Welded Eurofer

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9CrWTaV reduced activation ferritic/martensitic steels (RAFMS) actually are prime candidate materials for structural applications in advanced fusion energy systems. Eurofer'97 belonging to this current alloy generation is the European reference RAFMS for ITER Test Blanket Module and DEMO structural components. It is the subject of extensive characterization about its performances before and after irradiation for reference data-base constitution before starting system design.

Useful information about fundamental physical-mechanical properties and behaviour under low-dose neutron irradiation of Eurofer has already been produced, while major uncertainty remains concerning potential steel damage by hydrogen from transmutation reactions and other sources during fusion reactor operation.

This paper presents an investigation performed within EFDA Technology Programme about hydrogen effects on the fatigue properties of base Eurofer'97 and its gas-tungsten-arc-welded (GTAW) joint. For this purpose, specimens were subjected to fully-reversed load-control low cycle fatigue (LCF) tests at room temperature and under electrochemical charging.

It was found that increasing hydrogen content in the range 2-7 wppm caused increasing loss of base-steel LCF performances compared to hydrogen-free situation, but enlarged data scatter in terms of lifetimes and cracking modes especially at lower frequency. Specimen fracture followed the striated path typical of true mechanical fatigue in un-charged conditions but varied from intergranular (IG) to transgranular (TG) as internal hydrogen increased. Careful inspection on a very microscopic scale suggested that the damaging mechanisms likely involved hydrogen-induced plastic flow modification and atomic bond decohesion. A number of experimental evidences also suggested the inhomogeneous character of Eurofer microstructure in terms of grain size and boundary precipitation.

LCF failure of un-charged GTAW Eurofer always occurred by true mechanical fatigue across the soft overaged region of the heat affected zone (HAZ). Comparatively, premature fracture from replicated tests on hydrogenated samples underwent either by brittle microcleavage across the large grains of the hard molten pool and after reproducible numbers of cycles, or within the soft overaged HAZ. Hydrogen in this latter case promoted internal embrittlement by localized shear starting from coarsened carbides at grain boundaries, and was associated with significantly scattered lifetime reduction, which supported the inference previously made of original parent metal heterogeneous microstructure.

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12-16

Analytical Estimation of Accessibility to Activated Lithium Loop in IFMIF

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International Fusion Materials Irradiation Facility (IFMIF) is an accelerator-based intense neutron facility for development of fusion materials. The neutrons are generated by deuterium-lithium (D-Li) reactions in a liquid lithium target of IFMIF. Radioactive nuclides are generated through the D-Li reaction and neutron irradiation on the target vessel made of stainless steel. The radioactive nuclides, such as corrosion products, beryllium-7 (^7Be) and so on, circulated in a lithium loop are removed by impurity traps or deposited on inner surface of component in the lithium loop. The lithium loop is planned to be maintained annually after each 11-month operation. Activation of the lithium loop significantly affects accessibility to and maintenance scenario of the loop.

Dose rate around typical sections / component of the lithium loop was calculated employing a code QAD-CGGP2R that can deal with 3-D model of activated objects. Deposition rate of the radioactive nuclides on the component was assumed as parameter survey, since it depends on temperature and flow condition of the components and removal performance of the impurity traps. As result, the most effective nuclide was ^7Be . Its dose rate was several orders higher than $10 \mu\text{Sv/h}$ a limit considering ICRP recommendation. Furthermore its half-life is 53 days, which is longer than planned maintenance duration of 1-month. Therefore, significant reduction of ^7Be deposition by a cold trap and radiation shielding are needed.

In this paper, investigations on concepts of radiation shielding on the loop component and maintenance scenario, remote handling and access control for example, consequently performed after the analysis of dose rate will be also presented.

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12-17

Irradiation facility LiSoR

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LiSoR (Liquid metal – Solid metal Reaction) is a unique irradiation facility in which a specimen is attacked simultaneously by flowing liquid metal and proton beam while stressed in tension. LiSoR loop and the test section was designed and constructed for irradiation with 72 MeV protons generated by PSI Philips cyclotron to investigate mainly the effect of liquid metal corrosion and embrittlement under irradiation onto the ferritic/martensitic steel T91.

When a solid metal is exposed to a liquid metal environment, various physico-chemical processes may take place. Up to now it is not even possible to forecast the stability of different liquid metal – solid metal combinations and if the solid metal is irradiated during exposure to liquid metal it seems to be totally impossible to estimate the behaviour of the solid. Recently it was reported that proton irradiation was associated with an increase in corrosion rate of HT-9 steel (ferritic/martensitic) in lead bismuth eutectic [1]. Therefore complex experiments like LiSoR are strongly required to investigate simultaneously the different impacts on steels.

Up to now 5 LiSoR test sections have been irradiated. After each irradiation cycle the test section plus the specimen was disconnected from the loop and transported into a hot cell for disassembling and examination.

In this paper the experience gained with the LiSoR irradiation facility before, during and after irradiation are discussed and additionally selected results of PIE (post irradiation examination) on irradiated specimens are presented.

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Designing Experiments for the International Fusion Materials Irradiation Facility

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The development of fusion power requires a facility for assessing the behaviour of materials subjected to damage from neutrons with an energy in excess of 14 MeV and fluences of up to 150 atomic displacements per atom (dpa). These are numbers outside of the scope of studies based on fission reactors. On the other hand, the proposed International Fusion Materials Irradiation Facility (IFMIF) will enable experiments to be conducted that better fit the requirements of fusion power plants.

It is appropriate to think about a suitable set of materials experiments to be conducted once the facility becomes available. It is recognised that irradiation experiments will be necessary over the range 250-550°C with fluences in the range 10-150 dpa.

When designing experiments it is natural to suggest a uniform test matrix over the domain of interest. This may not be an ideal approach since the uncertainties in knowledge are unlikely to be uniform, and it may be the case that the effects of individual variables are non-linear.

In the present work we suggest a set of experiments designed using a model created using available data from the published literature, for reduced-activation martensitic steels. It has been possible to identify gaps in knowledge on the basis of model-perceived uncertainties, and to access trends in order to determine appropriate gaps between experiments. The work has been used to suggest an optimised series of experiments.

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Transmutation Analysis of Realistic low-Activation Steels for Magnetic Fusion Reactors and IFMIF

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A comprehensive transmutation study of the low activation steels was performed for irradiation simulations in the high flux test module (HFTM) of the International Fusion Material Irradiation Facility (IFMIF) neutron source and the first wall of ITER and DEMO magnetic fusion reactor.

To carry out this analysis we have considered several austenitic steels (SS304, ITER SS 316 and the Ti-modified 316SS/PCA), some low activation (IEA-modified F82H/F82H-IEA and EUROFER 97) and oxide-dispersion-strengthened (ODS) (LAF-3 -with base composition similar to that of F82H) ferritic steels (FS). All these kind of steels have been considered for one or other reason in the selection of structural materials for magnetic or inertial fusion reactors.

Here, we classified the concentration of intended and impurity elements in these steels into three categories to study element-by-element the generation and depletion of solids and gaseous transmutants: i) typical intended elements in reduced-activation ferritic steels, ii) impurity elements in reduced activation FS and some major and minor intended elements is SS and Cr-Mo steels, and iii) impurity elements in steels.

The majority of steels constituents will not change under irradiation, while some major and minor intended elements will do 5-20%/1fpy. Impurities generated are also studied. The most important transmutations effects are produced in IFMIF, with a harder neutron spectrum.

The transmutation calculations were conducted with the ACAB code to handle the numerous reaction channel for neutron energies over 20 MeV. The activation cross-section libraries processed are IEAF-2001 for IFMIF and EAF2003 for DEMO and ITER. IEAF-2001 contains the neutron activation cross-section files for 679 nuclides, including stable and isomeric states up to 150 MeV. We have used the group wise IEAF-2001 library in GENDF format with 256 neutron energy structure. A set of benchmark calculations has been performed to validate the computational for intermediate energy activation and transmutation calculations.

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12-20

Reduction of radioactive inventories in the IFMIF test cell

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The International Fusion Material Irradiation Facility (IFMIF) is projected for the testing and qualification of fusion reactor materials under the irradiation conditions of a future fusion power reactor. It will use 40 MeV deuteron beams striking a liquid Lithium jet target to generate 10^{17} neutrons/second with energies extending up to 55 MeV.

Considerable effort has been conducted over the past years for the neutronic characterization and design optimization of the IFMIF test cell which includes the Lithium target with back plate and loop components, the test assemblies with high, medium and low flux test modules accommodating the irradiation rigs and material specimens, neutron reflector and shield plugs. Neutronics and activation calculations performed in this frame include Monte Carlo transport calculations with the McDeLicious code for the entire IFMIF test cell and the activation calculations with the ALARA inventory code and IEF-2001 cross-section data. A

detailed 3D geometrical model of the entire IFMIF test cell including all components was applied in these calculations. Nuclear responses such as the nuclear heating, the displacement damage accumulation and the gas production were calculated for the irradiation specimens in the test modules. In addition, the activation of the test cell components and the elemental transmutations of the materials irradiated in the test modules has been assessed. The rather high radiation loads and the resulting high radiation dose levels obtained for the test cell walls indicated the need to integrate a dedicated shield around the test modules.

This work is devoted to the nuclear design optimisation of a shielding block arranged around the test modules with the objective to reduce the radiation load and the activation in the test cell walls. The neutronic calculations are performed with the McDeLicious Monte Carlo code to optimise both the material and the geometry configuration with regard to its shielding efficiency. The nuclear heating and the radioactivity generated in the test cell walls and the lithium loop are used as criteria for the shield optimization. The effect on the irradiation performance of the material specimens in the test modules is assessed for the optimized shield configuration.

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Thermo-hydraulics and Technology of Neutron Lithium Target for IFMIF

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International Fusion Materials Irradiation Facility (IFMIF) is under development now to supply high-energy neutrons for irradiation of structural materials of future fusion power plants.

IPPE and VNIINM take part in the IFMIF Liquid Lithium Target problems via the ISTC Project #2036 named at the headline of the paper. Main tasks of the Project are: investigation of lithium jet hydrodynamics, evaporation of lithium from free surface of jet, lithium flow interaction with structural materials, elaboration of methods and devices for monitoring impurities and for purification of lithium.

Special Lithium Test Facility (LTF-M) was constructed. It allows testing target assembly mock-up at original curvature of back wall, lithium velocity, temperature and vacuum. Width and thickness of lithium jet are smaller by a factor of 2.5-3 in comparison with IFMIF target. The curvature of back wall results in centrifugal force (and pressure) preventing lithium boiling in vacuum conditions. Double reduced nozzle (by Shima model) is used to produce flat velocity profile of the lithium jet with minimum turbulence intensity. Wave amplitude at lithium jet surface should not to be more than ± 1 mm at the lithium velocity within 10 – 20m/s.

Water jet was studied by means of moving electro contact probe, Pitot tube and photo camera as well. Thickness of the jet, velocity profile and amplitude of waves were measured at different points. Some features of the target assembly mock-up and nozzle were clarified.

Target assembly mock-up for lithium tests is manufactured, mounted at the LTF-M and tested. The mock-up is supplied with electro contact probes placed at 12 points under surface of lithium jet. Sapphire windows are used to illuminate and observe lithium jet, to make photos and video clips.

Investigations of lithium evaporation from free surface of the jet are being performed at special test section attached to the target assembly mock-up. Erosion and corrosion impact on structural materials are being studied using "Rotating Disk" test facility supplied with small circulation lithium loop equipped for conditioning lithium.

Some methods and devices for monitoring impurities in lithium are tested. Soluble getters for deep purification of lithium from impurities (≤ 10 ppm) are investigated.

Main results of all the investigations are presented.

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12-22

Neutron Induced Activation of the IFMIF Lithium Loop Corrosion Products

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The assessment of the neutron activation of corrosion products generated in one year of IFMIF lithium loop operation due to the interaction between lithium and Stainless Steel SS-304 has been performed. This paper describes the approach used for and present the results obtained.

The study was accomplished through the following phases:

- 1) neutron spectrum calculation in the lithium target via MCNP-4C2 with McEnea neutron source model;
- 2) inventories calculations and decay gamma sources production via ANITA-IEAF activation code package; the calculations were performed by considering a lithium mix composition containing lithium impurities and corrosion products referred to 200 wppm of Steel SS-304 corresponding to a corrosion rate of 0.2 $\mu\text{m}/\text{y}$ and a SS-304 wetted surface of 572 m^2 ; an irradiation scenario reproducing the integrated (in eleven months of operation) neutron flux responsible for the activation of the circulating corrosion products facing the deuteron beam was considered;
- 3) decay gamma transport analysis for dose rate evaluations via both VITENEA-IEF/SCALENEA-1 and MCNP-4C2 systems for the Longest Pipe of the Lithium loop.

The following conclusions can be drawn by the results analysis:

- dose rates at 50 cm from the Longest Pipe are 198 $\mu\text{Sv}/\text{h}$ and 85 $\mu\text{Sv}/\text{h}$ at 1 day and 1 week from the plant shutdown, respectively
- considering the average 20 mSv/a regulatory limit in Europe for "Radiation Worker" and the four-week period of annual maintenance activities in Li loop, the zone around the piping, exceeding 125 mSv/h, has to be declared "Restricted Access Area"
- the worker radiation protection could be a concern for IFMIF if a good purification system of Li will not continuously run
- specific procedures and worker protection equipments (such as Remote Handling tools) and shielding have to be considered to maintain the Li loop.

Sensitivity analysis have also been performed to evaluate the impact of the choice of different neutron source models, of irradiation scenarios and of corrosion rates/lithium mix composition on the dose rates.

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Neutron and deuteron activation calculations for IFMIF

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The materials for future fusion devices such as DEMO require testing to high fluence so as to accumulate significant amounts of damage. Such testing is planned to be carried out in IFMIF, a facility where a beam of accelerated deuterons impinges on a flowing lithium target producing neutrons. These neutrons will have a maximum energy of about 55 MeV and in order that activation calculations can be carried out, the nuclear data must contain cross sections covering a similar energy range. Conventional activation code systems such as EASY, designed for the activation of fusion devices, contain data up to only 20 MeV. To overcome this limitation EASY has recently been extended so that the current version EASY-2005 can be used with a neutron spectrum extending to 55 MeV.

A description of the EASY-2005 system is given; the data libraries are large, containing 62,637 excitation functions on 775 different targets from H-1 to Fm-257, and with decay data for 2,192 nuclides. A new library in the same format as the neutron library has been added to EASY to cover another significant source of activation from deuteron-induced reactions.

Calculations of the activation of materials in many regions of IFMIF, including the structure, samples and the flowing lithium target have been carried out. Results of these calculations are reported, and the contribution of neutrons above 20 MeV to the activation discussed. Preliminary calculations using the deuteron library have been made and the activation from deuterons and neutrons is compared in the target region.

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12-25

Cavitation detection to avoid material erosion in Liquid Lithium Target Facility for IFMIF

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In the IFMIF (International Fusion Material Irradiation Facility) testing facility, the required high energy neutrons emission will be procured by reaction of two D+ beams with a free surface liquid Lithium jet target flowing along concave back-wall at high speed (> 20 m/s). The Lithium height in the experimental loop and its relevant static pressure, the high flow velocities and the presence of several devices for the flow control and the pressure reduction increase the risk of cavitation onset in the target system. Special attention has to be taken in the primary pump, in the flow straightener, in the nozzle and their interconnections where the local pressure decreases and/or velocity increases or flow separations could promote the emission of cavitation vapour bubbles. The successive bubble re-implosions, in the higher pressure liquid bulk, could activate material erosion and transportation of activated particulates. These bubbles, if emitted close to the free jet flow, could also procure hydraulic instability and disturbance of the neutron field in the D+ beams-Lithium target zone. Therefore, the cavitation risk must be properly foreseen along the whole IFMIF Lithium target circuit and its occurrence at different operating condition should be also monitored by special instrumentation.

ENEA, having a consolidated experience in the cavitation and subcooled boiling detections for nuclear applications since '80, has patented for these purpose a specific accelerometer gauge called CASBA-2000.

ENEA, in close co-operation with JAERI, has performed in 2004 a specific test campaign at Osaka University Lithium facility aiming at demonstrating the capability of the CASBA-2000 to detect the onset of the cavitation noises on parts or components of a IFMIF reference lithium target circuit.

This activity was performed during the hydraulic tests on the flow instabilities at outlet of a straight nozzle simulating part of the IFMIF Lithium target circuit.

Two ENEA CASBA-2000 accelerometers were mounted onto the loop close to the inlet of the Electromagnetic Pump (EMP) and at outlet of the flow straightener upstream the nozzle.

The paper presents the results of the experimental test campaign with the detection of cavitation noises, only at EMP inlet, with different Argon pressurizations > 0.05 MPa absolute and with the lithium velocities at nozzle outlet < 20 m/s.

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Material Irradiation Conditions for IFMIF Medium Flux Test Module

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International Fusion Materials Irradiation Facility (IFMIF) is an accelerator driven neutron source, which is designed to perform material irradiation at conditions very close to that of future fusion reactor up to the anticipated lifetime of structural materials. Besides irradiation of material samples at the high flux test module, in situ creep-fatigue tests for structural materials at creep-fatigue test module (CFTM) and tritium release experiments for breeder blanket materials at tritium release module (TRM) are foreseen at the medium flux test module (MFTM) of IFMIF. As it was shown previously, creep-fatigue tests under irradiation provide less conservative estimate of the structural material fatigue lifetime. Beryllium is one of the candidate breeder blanket materials subjected during operation to the high neutron load will be tested at TRM. The present study is devoted to the evaluation of the creep-fatigue specimen and beryllium irradiation conditions at MFTM of IFMIF.

The neutron transport calculations were performed using McDeLicious code and updated global geometry model of IFMIF. Spatial variations of displacement damage, gas and heat production rates were calculated both for CFTM and TRM. It was shown that maximum displacement damage rate at CFTM is about 13 dpa/fpy. The damage rate variation along the specimen length does not exceed $\pm 10\%$ and is even smaller along the gauge length. The analysis shows that TRM can meet closely fusion typical He and dpa production rates in Be, while the T-production is somewhat above fusion conditions in the present test module design. Removing the CFTM and shift of the whole TRM upstream increases the damage rate to about 60% of that for fusion reactor and provides tritium and helium production rates comparable with fusion. In addition the important He/T ratio nicely fits the fusion relevant value after one year of irradiation at IFMIF TRM.

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Shielding analyses of the IFMIF Test Cell

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The IFMIF D-Li neutron source will produce an intense high energy neutron field to provide the material irradiation conditions of a D-T fusion power reactor. With two deuteron beams of 125 mA each accelerated to 40 MeV deuteron energy, a neutron source intensity of 10^{17} s^{-1} will be achieved during full power operation. This will result in a maximum neutron flux density around $10^{15} \text{ cm}^{-2}\text{s}^{-1}$ in the lithium target with a neutron spectrum extending up to 55 MeV. Accurate shielding calculations are required to assess the shielding performance of the test cell with the target, the test modules, reflectors and shield modules, the surrounding walls and the test cell cover to prove that the safety requirements can be met to allow for personnel access to the maintenance rooms during the operation of IFMIF.

Full three-dimensional shielding calculations of the IFMIF test cell were performed to this end using a recently developed computational scheme for coupled Monte Carlo/deterministic (S_N) transport calculations that enables the use of a detailed geometry model of the test cell in the Monte Carlo calculation. The generation of D-Li source neutrons and their transport through the entire test cell is simulated with the McDeLicious Monte Carlo code, developed previously for IFMIF neutronics calculations. The neutron transport through the thick concrete walls of the test cell is described by means of 3D (deterministic) S_N calculations using the TORT code with a boundary source distribution as provided by the McDeLicious calculation at the inner surface of the test cell wall. In this way flux and dose rate distributions can be assessed with high accuracy in the neighbouring rooms of the test cell. Shielding calculations for the test cell cover, on the other hand, are complicated by a large number of penetrations through which neutrons will stream. Such problems are best handled by Monte Carlo calculations making use of various variance reduction techniques as available with the MCNP code.

In the paper, the computational approaches for the shielding calculations are outlined and results are presented of the IFMIF test cell shielding analysis including flux and dose rate distributions in the maintenance and access rooms.

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12-28

Corrosion Resistance and Thermal Aging Behavior of High-Cr Oxide Dispersion Strengthened Ferritic Steels in Super-Critical Pressurized Water

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Oxide dispersion strengthened (ODS) steels have been considered to be very promising for the application to advanced nuclear power plant as structural materials, because they are highly resistant to thermal recovery of the material structure as well as to neutron irradiation embrittlement. In order to improve the corrosion resistance, increasing chromium concentration has been required. However, it often resulted in the degradation of mechanical properties or thermal aging embrittlement. In this work, the effects of chromium concentration on the corrosion resistance in the SCPW and the thermal aging embrittlement of high-Cr ODS steels have been investigated.

Various ODS steels, which have 14 ~ 22 wt% chromium concentrations, were made by mechanical alloying method. Corrosion tests were performed in a SCPW environment (738 K, 25 MPa). Weight gain was measured after exposure to the SCPW for 2000 hr. For the improvement of corrosion-resistance, the effects of chromium and aluminum on the corrosion behavior were investigated. In order to evaluate the mechanical properties and the effect of thermal aging, miniaturized Charpy V notch tests and tensile tests were performed before and after aging at 773 K up to 4300 hr. The microstructure observations were conducted by TEM.

ODS steels with high concentration of chromium over 14 wt% and addition of 4.5 wt% aluminum showed high resistance to corrosion in the SCPW. An increase in the chromium concentration resulted in the more suppression of corrosion. As for aging effects, the 19Cr-ODS steel showed a large shift in the DBTT after aging at 773K even after 100 hr. In case of 14Cr and 16Cr ODS steels, almost no thermal aging embrittlement was observed even after the aging up to 4300 hr. Therefore, the maximum Cr concentration can be considered to be 16wt% for keeping balance in the corrosion resistance and mechanical properties. The mechanism of thermal embrittlement in high-Cr ODS steels will be discussed based on the TEM microstructure observation.

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12-29

Small Angle Neutron Scattering Study of ODS martensitic/ferritic materials

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Ferritic/martensitic alloys reinforced by nanoscale oxide clusters exhibit high creep strength as well as potential high resistance to radiation damage. However, the tensile or creep behaviour of ODS (Oxide Dispersion Strengthened) steels varies strongly from one alloy to the other. Small Angle Neutron Scattering (SANS) is a very powerful technique to characterize the nanometric Y₂O₃ oxides at the various stages of the fabrication process and we used it to identify the most promising fabrication routes to obtain the final optimized microstructure.

First, we showed that the much thinner distribution of nanoscale clusters in the 12YWT steel (Fe-12Cr-2.5W-0.4Ti-0.25Y₂O₃) compared to the commercial ODS steels (MA957 and PM2000) could explain its higher creep strength. Then, we examined different parameters which could influence the oxide distribution in the final product. We studied the effect of the milling conditions and also we tested different kind of mills. Moreover, we explored diverse ways to introduce yttrium, titanium and oxygen in the material by milling yttrium rich intermetallic compounds and yttrium oxides with a Fe-Cr matrix.

Among the main results, we showed that milling can induce a kind of "solid solution". The (Y-Ti) oxides precipitation occurs during thermal treatment and depending on the annealing conditions, a very fine size distribution can be obtained similar to the one observed in the 12YWT steel. Those results bring new openings to improve the final microstructure of ODS steels and the efficiency of the mechanical alloying process.

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12-30

Fracture toughness of the newly designed EU ODS-EUROFER steel

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Increasing the operating temperature of future fusion power systems is one of the keys to improving their efficiency. The use of reduced activation ferritic/martensitic steels (RAFM) restrict the temperature of operation to around 550°C. For this reason, during the past few years, within the frame of the European Fusion Programme, material studies have begun to be performed related to the RAFM oxide dispersion strengthened (ODS) steels, produced by mechanical alloying techniques, in order to demonstrate the benefits of ODS steels for structural applications in fusion power plants. The use of these materials in a specific blanket design would allow an increase in the operating temperature of the reactor up to 650°C and possibly up to 700°C.

The European activities related to the research and development of ODS materials are now mainly focussed on the metallurgical characterization of a particular ODS steel produced on the basis composition of Eurofer'97 (9Cr1WVTa) plus Y₂O₃ as oxide reinforcement, called EU ODS-EUROFER. The results obtained up to now in the RAFM ODS predecessors of the EU ODS-EUROFER have shown, among others properties, low fracture toughness. Consequently, this mechanical property must be well investigated in this newly designed and developed ODS steel.

Fracture toughness determination of structural materials is of great importance for integrity analysis. In reactor pressure vessel steels, the application of the Master Curve approach allows the determination of the full fracture toughness versus temperature curve testing few small specimens. This approach is based on the statistical analysis of cleavage fracture, and could also be appropriated to evaluate fracture toughness data of RAFM ODS materials in the transition region. The aim of this paper is to present the Master Curve of the EU ODS-EUROFER steel. For this, fracture toughness tests will be performed in plates and bars in order to also evaluate the fracture toughness in different product forms.

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12-31

Potential Microstructural Features Affecting Creep Property of ODS Steel

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Oxide dispersion strengthening (ODS) is the most effective way to improve creep property of Reduced Activation Ferritic/Martensitic steel (RAF). ODS steel has attracted attention for nuclear power plants applications, such as fusion and fast breeder reactors, because of its low swelling, high thermal conductivity, and high energy conversion efficiency.

ODS steel has a high density of small Y-Ti-O complex oxide particles dispersed in the matrix. Thus many researches have studied creep properties by focusing on oxide particles and dislocations in TEM observations, but are still insufficient to clarify creep mechanism. Potential microstructural features affecting creep property should be studied. The material production of ODS steel involves many processes, such as MA (Mechanical Alloying), degassing, canning, hot extrusion, and heat treatments. These procedures cause anisotropic and complicated microstructures. There are many potential microstructural features affecting creep property, for example, pores on PPB (Prior Particle Boundary) aligned parallel with the extrusion direction, intra/intergranular precipitates, oxide inclusions, and two matrix phases (tempered martensite matrix and δ -ferrite matrix), except oxide particles and dislocations.

In this work, potential microstructural features affecting creep property of 9Cr ODS steels were studied to clarify creep mechanism completely. A potential microstructural feature, PPB pores, was observed by FE-SEM. PPB pores were linked each other and developed into large cavity on PPB during creep. The PPB cavity enhanced creep crack propagation and degraded creep property. Reducing PPB cavities was performed by using larger MA powder. From the result, the reduction of PPB is revealed to be one of the effective ways to improve and guarantee creep property.

The role of other potential microstructural features will be discussed in this presentation.

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12-32

Effect of Milling on Morphological and Microstructural Properties of Raw-powder Particles for High-Cr Oxide Dispersion Strengthened Ferritic Steels Production

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Oxide dispersion strengthening (ODS) ferritic steel is a very promising candidate for a high thermal efficient advanced fusion blanket structural material. Material performance of the ODS steels is considered to depend on the dispersion conditions of the oxide particles such as Y_2O_3 and/or TiO_2 . The dispersibility and filling state of raw-powder processed by mechanical alloying (MA) have been also considered to be important factors for producing a good final product with desired properties, i.e. high-temperature strength and corrosion resistance.

In this work, we investigated the effects of the milling on morphological and microstructural properties of raw-powder particles for production of high-chromium ODS steels. Moreover, the chemical composition, distribution and size of the resultant powder were evaluated to understand the dispersibility of the oxide particles.

Raw-powder particles prepared by MA for a high-Cr ODS steel were examined by means of laser diffraction scattering method and X-ray diffraction measurements as a function of milling time. Morphological observation was performed by scanning electron microscope.

It was found that the secondary particles, which were larger particles condensed with fine primary particles, were formed from the raw-material powders at the early stage of milling. On the other hand, with increasing milling time the particle size distributions of the as-prepared powder gradually shifted toward the smaller sizes and the distribution peak widths were also remarkably narrowed. The XRD and SEM results revealed that the powder particles having high ductility such as Fe is difficult to be reduced their sizes, even though a high energy was given by the MA. A statistic analysis indicates that the particle size and its distribution peak widths were reduced by the milling. In the presentation, a procedure for preparation of raw-powder particles to produce high performance ODS steels will be discussed based on the experimental results.

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12-33

Mechanical and Microstructural Behavior of Y_2O_3 ODS EUROFER 97

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Dispersion strengthening appears to be the most promising approach to widen the operating temperature window of the ferritic/martensitic steels. Oxide dispersion strengthened (ODS) steels produced by mechanical milling and hot isostatic pressing (HIP) are considered as potential structural material for future fusion reactors. In Europe, efforts have been concentrated to produce these materials using the reduced activation ferritic/martensitic (RAFMs) steel EUROFER 97. The preliminary results indicate that ODS EUROFER 97 with 0.3 wt % Y_2O_3 exhibits high creep resistance and tensile strength at temperatures up to around 650 °C but a reduced fracture toughness and a high ductile-brittle transition temperature (DBTT) compared to conventional steels. To assess the capabilities of ODS EUROFER is essential to determine whether the worsening of the impact properties is an inherent characteristic or is related to its production process. Charpy impact tests performed on ODS EUROFER with Y_2O_3 particles point out that lack of toughness could be inherent. These tests, as well as others previously reported, were performed on as-HIPed 100 % ferritic ODS EUROFER. Mechanical and microstructural results from ferritic/martensitic ODS EUROFER have not been reported so far.

In an attempt to investigate the mechanical and microstructural characteristics of ferritic/martensitic ODS EUROFER we have produced an ingot $\varnothing 24 \times 35$ mm³ using as starting material EUROFER 97 gas atomized by Studvick containing 0.3 wt. % of Y_2O_3 nanosized particles. The milled powder, canned and outgassed, was consolidated by HIP at 1373 K and 200 MPa for 2 h under Ar followed by cooling at rate of 30 K/min keeping constant the pressure of 200 MPa during cooling.

The material, which results fully dense, is characterized by X-ray diffraction, differential thermal analysis and electron microscopy. Hardness, tensile and impact Charpy tests are performed on the material tempered at 1023 K for 90 min to determine its mechanical properties and characteristic DBTT.

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12-34

Microstructural characterisation of reduced activation EUROFER ODS alloy after long-term annealing

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In this work we present results on the studies carried out on two reduced activation ferritic-martensitic ODS alloys based on the Eurofer 97 composition but fabricated by two different routes (CEA, Germany and CRPP Swiss). They were studied before and after annealing at 700 °C during 3600 hours by means of optical microscopy, XRD, SEM and TEM. Results show that the yttrium oxide dispersion changes after annealing with a redistribution of the yttria particles towards the grain boundaries and migration of Y to the scale formed at the metal atmosphere interface. The outer Y diffusion is stronger in the CRPP alloy due to the presence of porosity. Nevertheless the oxidation is stronger in CEA ODS alloy due to the beneficial effect that the Y diffusion towards the scale produces on CRPP alloy. XRD results show that the oxides formed in the surface of both alloys are mainly Fe-Cr oxide with some Mn-Cr spinels formed in CRPP ODS. High resolution transmission electron microscopy experiments are running actually and the results will be also presented and discussed at the conference.

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12-35

Improvement of 9%Cr ODS Steel by a Microstructural Modification during the Post Consolidation Process

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9% Cr ODS steel samples were prepared by a mechanical alloying of the elemental powders of Fe, Cr, V, Ti together with Y₂O₃ powder, a HIPping at 1150°C/10.3MPa (1500psi) for 4 hr, and a hot rolling at 1050°C. Mechanical alloying parameters (ball to powder ratio and the milling time) were optimized by analyzing the chemical uniformity, the size distribution of the milled powders. Microstructure of the HIPped and the hot rolled samples were observed by using an optical microscope, a scanning and a transmission electron microscope. Equiaxed grains were observed in the HIPped samples, but the mean grain size of the 0.2% V added sample was two orders of magnitude smaller than that of the other one which did not contain V. On the other hand the hot rolled samples, V containing or not, showed similar grain sizes. M₂₃C₆ carbides were frequently found on the grain boundaries of both samples after any process.

It was attempted to modify the microstructure of the ODS steel to improve the characteristics of the fracture and the fatigue behavior. Fine carbonitride precipitates were introduced to the matrix by a minor chemistry control. They acted as a grain refiner and increased the impact absorption energy and decreased the fatigue crack propagation rate. Also the effects of the partial recrystallization and the cooling rate during the normalizing treatment on the mechanical properties were investigated. The one-half recrystallized sample showed a better mechanical behavior than the fully recrystallized (normalized) one; lesser anisotropic mechanical properties and a higher yield strength at the intermediate temperature range (500 to 700°C).

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12-37

Properties of Friction Welding between 9Cr-ODS martensitic and Ferritic-martensitic steels

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Japan Nuclear Cycle Development Institute has examined the friction welding between the oxide dispersion strengthened (ODS) steel and the ferritic-martensitic 11Cr-0.5Mo-2W,V,Nb steel (PNC-FMS) for the applicability as the dissimilar welding for ODS. This welding method is effective for manufacturing ODS components in some parts of the blanket system of a DEMO fusion reactor as well as long-life cladding tubes in advanced fast reactor fuel elements.

The friction welding was conducted by using the continuous drive friction welding equipment. An end face of a 9Cr-ODS round bar (22mm in diameter) butted on a PNC-FMS square bar (25mm sides) rotating at a rated speed of 2,200rpm to generate friction heat for the welding. The friction pressure and the burn-off length were fixed at 16kgf/mm² and 1.0mm, respectively, which are typical conditions for the welding of carbon steel bars. The upset pressure was applied for 3 sec immediately after stopping the rotation by the braking device. Neither cracks nor peelings were observed in the weld zones in the upset pressures from 20 to 30kgf/mm².

The heat affected zone as welded condition showed significant rise in Vickers hardness. Thus, the heat treatments were conducted to adjust the hardness to the available level for cold working. Approximately the same hardness were obtained for ODS and PNC-FMS by heat-treated at 1323K for 1 hr, followed by slow furnace cooling and then tempering at 1053K for 1 hr (FC-T), though the welded zone showed slightly lower hardness than the base materials. On the other hand, ODS had higher hardness than PNC-FMS by normalizing at 1323K for 1h and tempering at 1053K for 1 hr (NT). The results of tensile tests conducted after NT showed that the welded joint had higher strength than PNC-FMS in temperatures up to 773K.

The cold rolling with 50% reduction was performed by using plate specimens taken from the welded joint after FC-T. No defects were observed in the welded zones. The tensile test at 673K of the plate specimens after NT showed that the cold rolling had no significant effect on the tensile strength of the welded joint.

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12-38

Direct Correlation Between Chromium Phases and Impact Behavior Of ODS Steels

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Recently developed Reduced Activation Ferritic Martensitic (RAFM) ODS steels on the basis of the European RAFM reference steel Eurofer 97 showed good tensile and creep properties, acceptable ductility, but poor impact behavior. Selecting a specific production route for ODS-Eurofer steel of the second generation, which included rolling and appropriate thermal treatments, DBTT could be shifted from values between +60 and +100°C for hiped ODS-Eurofer of the first generation to values between -40 and -80°C.

The microstructure of ODS-steels was investigated after different thermal and thermo-mechanical treatments by means of High Resolution Transmission Electron Microscopy (HRTEM) for structural analyses, and analytical TEM applying techniques like Energy-Filtered TEM (EFTEM) and energy-dispersive X-ray analysis (EDX) to detect the elemental distribution in nanometer scale. In "as-hipped" ODS-Eurofer steel of the first generation a decoration of the grain boundaries with Cr-rich precipitations was detected. Besides this, elemental EDX mapping evidenced the existence of Cr-depleted areas at grain boundaries. The chromium concentration in these areas is remarkably lower compared to the grain bulk. Thermal as well as thermo-mechanical treatments of the ODS material change size and spatial distribution of the Cr-containing precipitations. Depending on the treatments, morphology and density of the Cr-rich phases can be systematically varied. The significantly improved Ductile-to-Brittle-Transition-Temperature (DBTT) in impact tests and increased high temperature tensile ductility can be directly linked to the changes in precipitation morphology and concentration.

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Microstructural Evaluation of Hot Rolled Plates of Oxide Dispersion Strengthened 8Cr-2W and 8Cr-1W Steels

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Oxide dispersion strengthened (ODS) products exhibit mechanical anisotropy based on their manufacturing methods. To trace the origin of the anisotropy of ODS steels, microstructures and creep properties of hot rolled plates with compositions of Y₂O₃-TiO₂ dispersed 8%Cr-2%W (F-82H) and 8%Cr-1%W have been investigated in this study.

Creep tests have been carried out on specimens sampling along both the rolling and the cross direction at 700 and 750 °C up to 2000 h. Microstructural analyses were made on the normalized and tempered condition by using OM, SEM, TEM and XRD. Creep ruptured specimens were also investigated. The ratio of δ -ferrite for 8%Cr-2W ODS steel was about 20% and that for 8%Cr-1%W ODS steel was below 10% in volume fraction and the δ -ferrite was elongated in the rolling direction. Superior creep strength of the ODS steels were caused mainly by not only finely dispersed Y-Ti complex oxide particles but also the highly strained matrix which was concluded from the XRD measurements. It seems that elongated δ -ferrite strongly affects on the anisotropy of creep properties. However, M₂₃C₆ carbide particles are finer than the usual martensitic heat resistant steel and the distribution of M₂₃C₆ is non-uniform. The later factor should also influence the creep anisotropy.

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Structure and stability of nano-size oxide in ODS steels

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Oxide dispersion strengthened (ODS) steel is one of the candidate materials for fast reactors and fusion reactor components, because of the excellent resistance to void swelling and high-temperature creep property. For progressing ODS ferritic steels the key issue is to control the distribution of the nano-particle. Recent results showed that Y-Ti complex oxide particles precipitate from non-equilibrium solid solution during fabrication process of MA and hot extrusion. In this study, we focus on the microstructural details and the stability of the Y-Ti complex oxide under electron and ion irradiation.

The materials used in this study are three types of ODS steels: Fe-16Cr-18Ni-3Mo-Ti, Y, O (Cr-Ni, fcc) and Fe-9Cr-2W-Ti,Y₂O₃ (9Cr, bct) and Fe-13Cr-2.8W-Ti,Y₂O₃ (12Cr, bcc). Nano-structure of the particles was analyzed by a high resolution microscopy (HRTEM) and an atom probe field ion microscopy (AP-FIM). To evaluate the stability of ODS particles, electron irradiation was carried out in HVEM of 1.25 MeV to 30 dpa at RT – 673 K. Ion irradiation was also performed by TIARA of JAERI Takasaki to 50 dpa at 473 to 723 K, where the triple beams consisted of Fe³⁺, He⁺ and H⁺ ions.

(1) The shell-core structure in complex oxide particles of Y, Ti and O was detected by HRTEM and AP-FIM. Ti or Y segregated at the shell with the order of 1 nm. The shell-core structure assumed to be formed during final fabrication process; hot extrusion at 1000K, which has important role for reducing mismatch between oxide particle and matrix. Therefore, additional elements can control the mismatch and homogenize the size of the oxide particles.

(2) The ODS particles were relatively stable during both of irradiation, but a limited shrinkage was observed by precise observation of electron irradiation: ODS particles in 12 Cr steel were shrunk with 8% during electron irradiation to 30 dpa at 723 K. This suggests that the recoil dissolution can be active at a limited condition.

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12-41

Heavy-ion Irradiation Effects on the Morphology of Complex Oxide Particles in Oxide Dispersion Strengthening Ferritic Steels

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The mechanical strength of oxide dispersed strengthening (ODS) ferritic steels depends on the dispersibility and microstructural stabilities of oxide particles. Yttria was selected as a strengthening element for ODS ferritic steels. Other elements such as titanium and aluminum are added to fine the dispersed particles and improve the mechanical properties of ODS steels. Recent development of ODS steels at Kyoto University has revealed that the aluminum addition to 19Cr ODS steels having yttria-titanium complex oxides resulted in the formation of the yttria-alumina complex oxides. In this work, the microstructural stabilities under heavy-ion irradiation was investigated focusing on the characteristics of yttria-titanium and yttria-aluminum complex oxides.

The materials used were the ODS ferritic steels with different Cr concentrations from 14 to 19%. Yttrium, titanium and aluminum powders were added for the mechanical alloying. The milled powders were consolidated by the hot extrusion at 1423K. The as-received ODS materials were annealed at 1323K for 1h. A 1.7 MeV tandem accelerator for heavy-ion irradiation at Institute of Advanced Energy, Kyoto University was used to induce irradiation damages. The irradiated and thermal annealed ODS steels were cut and polished by a focused ion beam processing for TEM investigations. The 19Cr-2W-4Al-0.3Ti-0.35Y₂O₃ ODS ferritic steel had several kinds of precipitates including fine dispersed oxides. The dispersed oxides in this steel were investigated as yttrium-aluminum complex oxides. The yttrium-aluminum complex oxides kept stability after the ion-irradiation at 773K to 20 dpa. Both the thermal and irradiation effects on the dispersed oxides were not detected. The slight growth of oxide was observed after the irradiation at 973K to 20 dpa. The characterization results will be shown after the analysis by Field-Emission TEM, EDX and EELS observations.

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12-42

The Microstructure and of MA957 Nanostructured Ferritic Alloy Joints Produced by Friction Stir and Electrospark Deposition Welding

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Nanostructured ferritic alloys (NFA) promise outstanding high temperature creep strength and the potential for managing radiation damage, including high helium levels in fusion reactor structures. A major challenge to the use of NFAs is joining. The excellent properties of NFAs derive from a high concentration of 1-3 nm scale Y-Ti-O solute clusters, with secondary contributions from a coarser distribution of slightly larger (> 3 nm) Y₂Ti₂O₇ oxides. Conventional fusion welding processes are generally not useful, since melting-solidification destroys the nm-scale features. Joining processes, either involving very rapid melting and re-solidification or avoiding melting entirely, are preferable. We present the results of two such approaches to joining NFA MA957: friction stir welding (FSW) and electrospark deposition (ESD) welding. The FSW and ESD welds were produced by the Edison Welding Institute as described in a companion paper. The main focus of this paper is the effects of the joining processes on the nm-scale and other key features. The microstructures of the base metal and joints were characterized by TEM, and the fine scale features in the FSW and ESD welds were also evaluated by SANS. The FSW processes produced satisfactory high strength, defect free joints. The ESD welds, deposited in air, contained porosity, hence, will require additional development. The finest scale features in the FSW weld were degraded, but the severe deformation produced an attractive fine-scale, equiaxed grain structure along with a significant density of dislocations and oxide particles.

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Ductile-to-Brittle Transition Temperature Shift in Ferritic Steels under High Fluence Neutron Irradiation

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Neutron irradiation in ferritic steels can induce matrix damage, precipitation, grain boundary segregation, etc., in materials, which results in the degradation of mechanical properties, especially ductile-to-brittle transition temperature (DBTT) shift. Irradiation embrittlement includes hardening embrittlement (e.g. copper-related precipitation) and non-hardening embrittlement (e.g. phosphorus intergranular segregation). In this study, we focus on the effect of inter-granular segregation causing by high neutron fluence on DBTT shift. Molecular dynamic (MD) simulation and solute drag models are employed to calculate the radiation-induced inter-granular segregation. The DBTT shift is estimated according to the amount of grain boundary segregation, matrix hardening and neutron fluence. Practical measurements of neutron irradiation-induced DBTT shifts at 270°C and 400°C are made by small punch and mini-charpy test technology. In addition 9% mechanical pre-straining (cold working) is carried out to simulate macroscopic effects of neutron irradiation. Predicted DBTT shifts are compared with experimental data for C-Mn and 2.25Cr1Mo steels.

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Effect of Neutron Dose and Temperature on Tensile and Fracture Toughness Properties of Titanium Alloys

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Two types of high strength titanium alloys i.e. Ti6Al4V and Ti5Al2.5Sn were studied in as received condition. The tensile and the fracture toughness tests were carried out both in the unirradiated and neutron irradiated conditions. Tensile specimens of Ti6Al4V alloy were irradiated with fission neutrons at 50°C to displacement doses of 0.001, 0.01 and 0.1 dpa and at 150°C to 0.3 dpa (NTP). Fracture toughness specimens of Ti6Al4V and Ti5Al2.5Sn alloys were irradiated at 150°C to a dose level of 0.3 dpa (NTP).

Tensile properties of the ($\alpha+\beta$) alloy showed initial softening followed by hardening and lack of work hardening when neutron dose increased from 0.001 to 0.1 dpa at the ambient temperature. At a dose level of 0.3 dpa the ($\alpha+\beta$) alloy showed a clear change in tensile properties when irradiated and tested at up to 150°C compared to those at 350°C. Hardening and plastic instability was observed at temperatures up to 150°C whereas clear work hardening behaviour was observed at 350°C. Fracture toughness of the irradiated (α) and ($\alpha+\beta$) alloys seemed to increase almost linearly with increasing temperature where as that of ($\alpha+\beta$) alloy decreased at 350°C.

At low dose level of 0.001 dpa the ($\alpha+\beta$) alloy showed softening and at higher dose levels increase in hardening and lack of work hardening at the ambient temperature. The ($\alpha+\beta$) alloy seemed to suffer from plastic instability when irradiated to a dose level of 0.3 dpa and tested up to 150°C. At elevated temperatures a substantial amount of hardening in the ($\alpha+\beta$) alloy was observed in irradiated condition. Earlier studies have shown that fracture toughness behaviour of the irradiated (α) and ($\alpha+\beta$) alloys were quite similar at ambient temperatures. At elevated temperatures fracture toughness of the irradiated ($\alpha+\beta$) alloy seemed to reduce more than that of the (α) alloy when compared to unirradiated condition.

The large irradiation hardening and loss of fracture toughness in the ($\alpha+\beta$) alloy appears to be related to radiation dose level, temperature and radiation-induced precipitation in the ($\alpha+\beta$) alloy.

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12-46

Influence of radiation induced defects on fracture behavior in highly pure SiC

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Highly pure and high crystalline SiC have been considered as reinforcement fiber and matrix components in SiC/SiC composites because of its excellent microstructural and mechanical properties under severe neutron and ion irradiations. Majority of studies in connection with SiC-based materials are in progress. Irradiation behaviors such as swelling, amorphization, microstructural evolution, thermal shock, thermal conductivity, mechanical- and physical-property changes are of interest. One of the most important issues of SiC-based materials for fusion structural applications is those fracture behaviors under expected irradiation conditions.

In this present works, silicon self-ion irradiation was carried out at DuET facility; Dual-beam for Energy Technologies, Kyoto University, to simulate fusion conditions. To investigate deformation and fracture behaviors of SiC, microstructural analysis was carried out with observing cross-section underneath an indentation.

Clear increase of indentation fracture toughness was shown in the ion-irradiated SiC compared to that of unirradiated SiC. Many micro-cracks were created around the indentation induced deformation region. Indentation induced slip-band was exhibited on the (111) plane both of the virgin and the ion-irradiated SiC. The slip band in the ion-irradiated SiC was one of the sites for micro-crack creation. Formation of the micro-cracks may dissipate the fracture energy for brittle fracture like unirradiated SiC. Extension of the created micro-cracks was disturbed by a number of strain fields around point defect clusters. Various toughening behavior of SiC by radiation induced defects will be discussed.

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12-47

Modeling of austenitic alloy hardening under irradiation

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Austenitic alloy hardening under irradiation has been under research for more than four decades. Irradiation-induced degradation of mechanical properties is the major problem for structural materials in nuclear reactors.

In this work, we will present a model, which combines theoretical models of microstructural evolution with experimentally obtained dependencies. The density of point defects, loops and cavities will be calculated by rate equations. For simplification, cascade effects are ignored in the model. Some of point defects, or freely migrating defects generated by irradiation are annihilated by mutual recombination. Interstitials, which escape from mutual recombination, are diffusing faster than vacancies and form interstitial clusters, or dislocation loops. When loops grow large enough to become unfaulted, network dislocations are formed. Both dislocation loops and network dislocations absorb interstitials more than vacancies, i.e. the dislocation is a biased sink toward interstitials, resulting in vacancy supersaturation in the matrix, and cavities nucleate and grow.

This microstructural evolution model will be used to predict hardening of austenitic alloys under different irradiation conditions. Obtained defect sizes and densities will be used in dispersed barrier hardening model. A comparison of calculated and experimentally obtained data will be made.

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12-48

Effects of Reductions in Strain Hardening on Irradiation Induced Transition Temperature Shifts

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Irradiation hardening, usually characterized in terms of the yield stress change, $\Delta\sigma_{ys}$, leads to corresponding increases in the temperature (ΔT_o) referencing the fracture Master Curve (MC) $K_{Jr}(T)$. Assuming hardening dominated embrittlement, the relationship is $\Delta T_o = C_k \Delta\sigma_{ys}$, where C_k typically $\approx 0.6 \pm 1$ for irradiations of tempered 8Cr martensitic steels around 300°C. However, irradiation also reduces the strain-hardening $\Delta\sigma_{sh}(\epsilon)$, and can even lead to strain softening in some irradiation and strain regimes; these effects must be accounted for in developing irradiation embrittlement ΔT_o models. We report a systematic study of the combined effects of $\Delta\sigma_{ys}$ and $\Delta\sigma_{sh}(\epsilon)$ on ΔT_o . The assessment is based on a local cleavage fracture criteria used in a model that determines the loading conditions (K_{Jc}) when a critical stress (σ^*) contour and encompasses a critical area (A^*). The finite element code ABAQUS is used to model the blunting crack tip field $A(\sigma_{22})$, where σ_{22} is the normal stress perpendicular to the crack plane. Based on the assumption of an invariant constant master curve $K_{Jc}(T - T_o)$ shape, the critical stress $\sigma^*(T)$ increases slightly with temperature in the athermal regime of the yield stress $\sigma_{ys}(T)$. While T_o is usually in the thermally activated temperature range for unirradiated materials, large $\Delta\sigma_{ys}$ elevate T_o into the athermal regime. However, the increase in T_o in due to $\Delta\sigma_{ys}$ (> 0) is reduced by the corresponding $\Delta\sigma_{sh}(\epsilon)$ (< 0). The effects of parametric variations of $\Delta\sigma_{sh}(\epsilon)$ and $\Delta\sigma_{ys}$ on C_k and $\sigma^*(T)$ are described. The effects of the test temperature on $\Delta\sigma_{ys}$ are also considered. These results are discussed in context of the effects of irradiation and irradiation conditions (dose and temperature) on $\Delta\sigma_{sh}(\epsilon)$ and $\Delta\sigma_{ys}$. It is also shown that changes in microhardness (ΔH) correlate more directly with ΔT than $\Delta\sigma_{ys}$.

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12-49

Brittle-Ductile Transitions in Vanadium and Iron-Chromium

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We report ongoing experimental work on fracture and brittle ductile transitions in vanadium, iron and iron - 9% chromium single crystals and polycrystals. Tests were carried out by four-point bending of pre-cracked specimens in the temperature range 77K - 300K, at a variety of strain rates. In contrast to many previous experiments on metals, we have found "sharp" brittle-ductile transitions in all tests so far, with no rise in fracture toughness with increasing temperature below the transition temperature, and fully ductile behaviour above it. For a given material, variation of the brittle-ductile transition temperature with strain rate allows us to estimate an activation energy for the controlling process; for vanadium this was found to be $\sim 0.5\text{eV}$; and for iron - 9% chromium $\sim 0.3\text{eV}$.

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12-50

Comparison of irradiation creep of γ -TiAl alloy under He- and H- implantation

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In-situ irradiation creep tests were performed for a Ti-47Al-2W-0.5Si alloy. The samples of such TiAl alloy have been homogeneously implanted with helium and hydrogen under uniaxial tensile stresses from 20 MPa to 450 MPa to a maximum dose of about 0.2 dpa (1700 appm-He in case of He-implantation) with displacement damage rates from 4.7×10^{-7} to 1.8×10^{-6} dpa/s at temperatures of 300°C and 500°C. Straining of the miniaturized dog-bone specimen under implantation was monitored by LVDT measurement while resistance was derived by four-pole technique. Subsequently the recovery of resistance was studied by post-implantation annealing experiments. Creep compliance, i.e. creep rate per dose rate and stress, was almost independent of temperature in the investigated region which did not show remarkable difference between He- and H-implantation. In case of He-implantation, the resistance of TiAl samples increased with dose and showed a tendency to saturate above 0.05 dpa, reaching total changes of 50% at 300°C and 30% at 500°C. These results are explained by a decrease of short range ordering in the ordered alloy. During post-implantation annealing from 200°C to 900°C with step of 50°C, resistance recovered clearly with a sharp stage at around 625°C. But surprisingly, no change of resistance under H-implantation was measured.

12-51

Effect of Cold Work on the Radiation-Induced Deformation of Austenitic Stainless Steels

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Austenitic stainless steels will be used in ITER as a major structural material. Although they will be used mainly in an annealed condition, cold-working is possible at various parts during construction or operation. Cold work introduces a martensite in the case of unstable steels like SUS 304 besides network dislocations. In this study, effect of cold work on the irradiation creep was experimentally examined and compared between stable (SUS 316L) and unstable (SUS 304) austenitic stainless steels. Radiation-induced stress relaxation behaviors were also evaluated using the obtained irradiation creep results.

Irradiation experiments were carried out with 17MeV protons (damage rate: 2×10^{-7} dpa/s). The creep apparatus has a loading system utilizing vacuum and a laser strain-measurement system with a resolution of 0.01 μm . Specimens with different levels of cold work (5% and 25%) were examined. The gauge size was $2.0 \times 10 \times 0.15$ mm and the grain sizes after the final annealing were about 13 μm . Specimen temperature was kept at $288 \pm 0.15^\circ\text{C}$ by Joule heating.

Stress dependence of the obtained irradiation creep rates was different between the two steels; linear in SUS 316L and quadratic in SUS 304, regardless of the cold work level. This difference influenced the stress relaxation; faster in SUS 316L. The behaviors of SUS 316L are consistent with the computer simulation for austenitic stainless steels with evenly distributed network dislocations, while those of SUS 304 appear to be originated from the high density of dislocations localized in the vicinity of martensite platelets.

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The Dependence of Obstacle Strength on Copper Precipitate Diameter in Fe-Cu Alloys Studied by in-situ TEM Observation

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It is widely accepted that the radiation-induced obstacles to dislocations are the primary reason for the radiation embrittlement of fusion reactor materials. Such obstacles are precipitates, dislocations, dislocation loops, etc. Extensive experimental studies on the relation between defect structures and mechanical properties were performed to show the validity of Orowan equation, i.e. the hardening, $\Delta\tau$, the defect diameter, d , and the defect density, N , are related using the obstacle strength parameter, α , by the equation $\Delta\tau = \alpha\mu b(Nd)^{1/2}$, where α is a very important factor in combining the radiation-induced obstacles and macroscopic mechanical properties. In recent MD simulation studies, the dependence of obstacle strength on its diameter was investigated. However, since obstacles responsible for hardening are often too small to observe by transmission electron microscopy (TEM), it is difficult to compare MD results with experimental observations.

In this study, we performed in-situ TEM observations of dislocation gliding through copper precipitates in thermally aged or irradiated Fe-Cu alloys under stress in order to investigate the dependence of obstacles strength on copper precipitate diameter.

Pure Fe, irradiated Fe-0.3%Cu and thermally aged Fe-1.0%Cu samples were prepared. The interaction between dislocations and obstacles were observed in-situ in a TEM with a tensile holder. The angle ϕ between the tangents to the two arms of the dislocation at the obstacle, the parameter characterizing the obstacle strength, was determined at the point of dislocation break-away from the obstacle. Only a single type of precipitate or PDC is assumed for the obstacle, hence the angle ϕ depends solely on the size of the obstacle. The density of obstacles was determined from the dislocation segment length l between two adjacent pinning points. The randomness of the pinning point separation has been taken into account by using relationship by Foreman and Makin, and compared with macroscopic mechanical properties.

For the Fe-1.0%Cu sample after 20 minutes aging at 525°C, Cu precipitate diameter was approximately 1 nm and the density of Cu precipitate was on the order of 10^{23} m^{-3} by 3DAP analysis. The average of obstacle strength, $\cos(\phi/2)$, and the segment length given by in-situ TEM observation were estimated to be 0.22 and 111 nm, respectively. The density of Cu precipitate derived from in-situ TEM observation was on the order of 10^{23} m^{-3} , which is in agreement with that estimated from the 3DAP analysis.

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12-53

Fracture toughness characterization of JLF-1 steel after irradiation in HFIR up to 5 dpa

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Fracture toughness specimens of the ferritic-martensitic steel JLF-1 were investigated before and after irradiation in two capsules at two different temperatures in the Oak Ridge National Laboratory High Flux Isotope Reactor (HFIR). The bottom and top parts of these capsules were loaded with disk-shaped compact tension [DC(T)] specimens that were used for fracture toughness characterization. This small (12.5 mm in diameter with thickness of 4.6 mm) DC(T) specimen was developed at ORNL for testing irradiated materials. DC(T) specimens were irradiated in each "low-" and "high-" irradiation temperature capsule up to ~3.8 dpa. Irradiation temperatures were measured by thermocouples. In the low-temperature capsule, DC(T) specimens were

irradiated at an average temperature of ~250°C. In the high-temperature capsule, specimens were irradiated at an average temperature of 377°C. Small, 3.33x3.33x25 mm) precracked Charpy specimens were irradiated in the middle of both capsules at ~300 °C and 500 °C up to 5 dpa. Static three-point bend testing of these specimens was performed to determine fracture toughness. Transition fracture toughness was evaluated in terms of reference temperature T_0 for each irradiation temperature and dose and compared to unirradiated T_0 . Current fracture toughness shifts compared to T_0 shifts of F82H and 9Cr2WVTa steels irradiated at similar conditions. In addition, these results are compared with available impact Charpy data for this material.

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Irradiation Behavior of Ti – 4Al – 2V (PT – 3V) Alloy for ITER Shield Blanket Modules Flexible Attachment

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Titanium alloys have been identified as candidate structural materials for the first wall, the blanket and the magnetic coil system of fusion reactors because of their high specific strength, low elastic modulus, low swelling tendency and their fast induced radioactivity decay.

Owing to the advantageous combination of rigidity, strength, shock resistance and thermal physical properties, titanium alloys are recommended as structural material for flexible attachments of the shield blanket modules in ITER reactor. Envisaged operating conditions (temperature 150 – 260 °C, rather high cyclic loads, damaging dose ~ 0.2 dpa) are such that it would be necessary to take into account changes in strength, plasticity, life time under low-cycle fatigue (LCF), fracture toughness resulting from the in-service effects.

The present paper summarizes the results of the radiation resistance analysis of the forged billet manufactured from Ti – 4Al – 2V (PT-3V) alloy. In-pile tests were performed in IVV-2M reactor at 240 – 260 °C to reach neutron damaging dose 0.35 – 0.42 dpa. After irradiation tensile properties, life time under LCF, fracture toughness characteristics (J_c and $J_{0,2}$) at the temperature 260 °C and 20 °C have been determined.

The irradiation effect has resulted in the appreciable changes in tensile mechanical properties under irradiation temperature. Radiation hardening under irradiation temperature reached 80 – 120 %. Reduction of plasticity characteristics was found as great as no more than ~ 50 %. Alloy remained capable for strain hardening. The values of uniform elongation did not drop below 2.5 %. At 20 °C the changes in tensile mechanical properties due to irradiation were less significant than those at temperature 260 °C.

Irradiation has resulted in a slight decrease of life time under LCF in the range of large strain amplitudes (1 – 2 %) and almost no change in life time in case the strain amplitudes were less than ~ 1 %. Irradiation caused significant reduction in fracture toughness at irradiation temperature the of $J_{0,2}$ were reduced in ~ 4.5 time while the values of J_c were reduced in 2.5 time.

Obtained results of the irradiation effect evaluation for investigated alloy were compared with those for ($\alpha + \beta$)-alloy Ti-6Al-4V.

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12-55

Surface Roughening Mechanisms for Tungsten Exposed to Laser, Ion, and X-ray Pulses

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Tungsten is a candidate material for a variety of applications in Magnetic and Inertial Fusion Energy (IFE) fusion systems. For example, it is proposed as a divertor plate material in a duplex structure attached to a copper heat sink for high heat flux applications in ITER. Also, it is proposed as an armor material for the High Average Power Laser (HAPL) IFE dry wall chamber design because of its high temperature capabilities. However, experimental data show that the surface of tungsten exposed to laser, ion, and X-ray irradiation undergoes substantial roughening with a variety of patterns and features. Control of surface conditions is essential to the design of these systems, since it can lead to crack formation, adverse effects on heat absorption because of emissivity changes, and eventual failure.

We first review recent experimental data on the effects of laser, ion and x-ray energetic pulses on the evolution of surface morphology and roughness of single and polycrystalline tungsten to identify the variety of patterns and length scales and their dependence on the type and magnitude of irradiation pulses. Then we present a model for the evolution of surface roughness as a result of the balance between destabilizing elastic strain energy caused by thermomechanical strains and near surface accumulation of defects on the one hand, and stabilizing surface and near surface atomic diffusion on the other. Results of the model determine the conditions for surface roughness evolution and the effects of radiation fluence and pulse intensity on surface morphology.

Keywords: Laser, ion, x-ray, surface roughening, tungsten

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12-56

Examination of Crack Tip Microstructures in F82H on the Lower Shelf

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Martensitic steels are being considered for fusion first wall applications. The effect of irradiation is found to alter the fracture toughness of these steels. In order to better understand deformation response during crack propagation, a study has been initiated to investigate details concerning the deformation processes occurring at the crack tip and how that behavior is affected by irradiation.

The dislocation microstructures have been examined ahead of the crack tip of a fatigue pre-cracked compact tension specimen of F82H loaded at -196°C to 25.6 MPa m^{1/2}. It is found that dislocation densities within subgrains are moderate in regions 1 to 2 μm from the crack tip, and slip band behavior is not observed either within subgrains or by its effect on subgrain boundary structure. In regions ~3 μm from the crack tip containing fine carbide particles, dislocation loops were found. By the time of the conference, it is anticipated that comparison with an irradiated specimen will be completed and included.

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Effect Of Irradiation Temperature On Radiation Hardening Of Pure Copper And Copper-Based Alloy

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Low-temperature radiation embrittlement is one of the main negative consequences of neutron irradiation for pure copper and copper-based alloys. But currently available data on copper radiation embrittlement have been obtained at $T_{irr}=60-90^{\circ}\text{C}$. The systematic data on the effect of irradiation temperature in the range of radiation embrittlement ($50-200^{\circ}\text{C}$) are lacking.

This paper presents the results of two experiments on irradiation of pure copper and GlidCopAl25IG alloy in the RBT-6 reactor at irradiation temperatures of 80°C and 150°C . The irradiation dose range was $10^{-5} - 10^{-1}$ dpa. The comparison between the dose dependencies of materials hardening and embrittlement revealed that a rise in temperature causes hardening to drop and embrittlement to decrease. In pure copper and GlidCopAl25 alloy the level of radiation hardening at $T_{irr}= 80^{\circ}\text{C}$ in the dose range of $10^{-3} - 10^{-1}$ dpa is about 50 MPa higher than at $T_{irr}= 150^{\circ}\text{C}$. The study of the stress-strain curves of irradiated materials showed that the strain hardening coefficient decreases with a decrease in the irradiation temperature, therefore the uniform elongation is decreased.

TEM investigations of the microstructure of irradiated specimens showed that irradiation at 150°C produces small radiation defect complexes, stacking fault tetrahedron (SFT), $d_{aver}=2$ nm and dislocation loops, $d_{aver}=10$ nm. On the whole, the structure of the specimens irradiated at 80°C and 150°C is qualitatively similar. The density of complexes increases with the irradiation dose and is systematically higher at $T_{irr}= 80^{\circ}\text{C}$.

The microstructure data on the density and size of complexes in irradiated materials served as the basis for calculations of the level of radiation hardening, with the Orowan-Seeger model used. The authors used in their calculations the values of density and size of defect complexes obtained by the TEM investigations of irradiated specimens. The comparison between the experimentally observed and calculated radiation hardening revealed that for pure copper this model yields good agreement with the experiment. The agreement is somewhat worse for GlidCopAl25 alloy.

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12-58

Effect Of Bake-Out Regime On Recovery Of Properties And Microstructure Of Neutron-Irradiated Pure Copper And Copper-Based Alloys

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The intermediate bake-out regime is considered as an effective method for recovery of the workability of ITER high heat flux components. But the data on the effect of irradiation dose and temperature on the bake-out efficiency are rather scanty.

In this paper, we present the results of investigation into the effect the bake-out regime has on recovery of the mechanical properties, electrical conductivity and microstructure of pure oxygen-free copper MOB, CuCrZr IG alloy and Cu-B system alloys. The results of two experiments on irradiation performed in the SM-2 reactor at an irradiation temperature of 80°C and in the RBT-6 reactor at 150°C are described.

In the SM-2 reactor samples were irradiated at 80°C in the dose range of 10^{-3} - 10^{-1} dpa and in RBT-6 at 150°C in the dose range of 10^{-5} – 10^{-1} dpa. The irradiated samples were baked out in two regimes: at 350°C during 10 hours or at 250°C during 24 hours.

The effect of the bake-out on the yield and ultimate strength, total and uniform elongation, electrical resistivity and microstructure of irradiated samples was investigated. The bake-out is shown to efficiently recover the ductility of pure copper and copper alloys to a level of ~80% of the initial one. The dose

dependence of changes in the properties of MOB oxygen-free copper, CuCrZr IG alloy and Cu-B alloys after the bake-out procedure was obtained for the first time. The bake-out is demonstrated to be rather efficient both at $T_{irr}=80^{\circ}\text{C}$ and at $T_{irr}=150^{\circ}\text{C}$.

The comparison between the strain-stress curves of the irradiated samples and the samples baked out after irradiation revealed that the bake-out restores the strain-hardening coefficient of the irradiated samples and causes the uniform elongation to increase. Electric conductivity of irradiated materials is recovered by 10-25% after the bake-out. With an increase in the irradiation dose the recovery degree of electric conductivity drops, since in this case the part of electric conductivity controlled by the accumulation of transmutant Ni and Zn is increased.

The TEM investigations into the structure of irradiated MOB oxygen-free copper revealed that irradiation at $T_{irr}=150^{\circ}\text{C}$ causes small radiation defect clusters, stacking fault tetrahedra (of vacancy type) $d_{av}\sim 2$ nm and dislocation loops $d_{av}\sim 10$ nm (of interstitial type) to develop in the structure. The cluster density drops after the bake-out, with the dislocation loop density decreased by about a factor of ten. SFTs, which slightly grow in size after the bake-out, remain the prevailing hardening element in the structure of the baked-out samples.

The conclusion has been substantiated that the bake-out procedure holds good promises for ITER application.

12-59

Transport of Carbon Impurity Using $^{13}\text{CH}_4$ Gas Puffing in JT-60U

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Erosion and transport of carbon attract much attention in SOL plasma physics and critically important for the estimation of carbon erosion and tritium retention in co-deposited carbon layers. In particular tritium retention in the co-deposits layers is one of the most critical issues for fusion safety. Although it is well known that carbon erosion dominates in the outer divertor and deposition in the inner divertor, the carbon transport mechanism from the former to the latter is still not unclear. Recently we have conducted the $^{13}\text{CH}_4$ gas puffing experiments in order to understand the behavior of the carbon impurities generated from the outer divertor area as well as their long range transport in JT-60U. The total of 3×10^{23} $^{13}\text{CH}_4$ molecules were puffed into L-mode plasmas of $I_p = 1.0$ MA and $B_T = 2.5$ T from a local gas puff port mounted in the gap between the outer divertor tiles. The feature of magnetic configuration in the experiment was such that the minimum distance between the last closed flux surface and the first wall surface on the upper part of the vacuum vessel is about 4 cm expecting deposition of a large amount of ^{13}C atoms thorough the SOL plasma transport.

After gas puffing, several tiles were sampled from the divertor area and the first wall and analyzed by Quadrupole Secondary Ion Mass Spectrometry (SIMS). Although without the puffing the outer divertor tiles were mostly erosion dominated, a thick deposition layers were found on the outer divertor adjacent to the local gas puff port. Deposited layers were also found near the outer divertor leg and on the upper first wall exposed to SOL plasma. SIMS analysis has shown that, about 90% of the carbon atoms are found to be ^{13}C atoms in such thick deposition layers near the local gas puffing port. This indicates that a large part of carbon impurities eroded at the outer divertor do not directly travel long distance, but is promptly redeposited near the eroded place and long range transport is resulted by the repetition of erosion and prompt redeposition processes. Detail profiles of the ^{13}C on the tiles and the correlation between the profiles and plasma parameters will be described in the paper.

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12-61

Neutronics Calculations for Waste Characterisation in IFMIF

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The primary mission of the International Fusion Materials Test Facility (IFMIF) is to generate a materials irradiation database for the design, construction, licensing and safe operation of a fusion demonstration reactor (DEMO). This will require testing of qualifying materials in a high neutron fluence so as to accumulate significant amounts of damage. Structural materials, for example, will be exposed in the high flux region to irradiation levels of at least 20dpa/full power year. Neutrons, with energies of up to 55MeV, will be generated by accelerating 40MeV deuterons into a flowing liquid lithium target.

This paper describes the neutron transport calculations throughout the IFMIF facility and the activation of all materials within the target chamber, the chamber's steel liner and the surrounding concrete shielding.

The neutronics calculations were carried out using McDeLi, a modified version of MCNP4C2. This combines the FZK modelled neutron source with subsequent neutron transport using the MCNP algorithms.

Activation calculations were carried out using FISPACT-2005, which includes activation data for the highest energy neutrons. Details of the volumes and masses of various categories of waste are provided.

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12-62

Activation properties of Nb₃Sn, NbTi and GFRP irradiated with D-T neutrons

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Nb₃Sn, NbTi wires and glass fiber reinforced plastics (GFRP) are prospective candidate materials of the superconducting coil assembly for fusion reactors. These materials are activated by D-T neutrons penetrating through the reactor core. From the radioactive waste point of view, it is important to investigate the activation properties for these candidate materials. However, no experiment has been done to investigate the activation properties for these materials. Therefore, we have carried out the experimental investigation of the activation properties of Nb₃Sn, NbTi wires and GFRP materials.

The neutron irradiation experiment has been done by the FNS D-T neutron source of JAERI. We used complex multi-filaments wires of Nb₃Sn and NbTi produced by FURUKAWA-electric Ltd. and a commercial GFRP (G10CR) plate. These samples were irradiated up to the neutron fluence of 4×10^{19} n/m² corresponding to about one twentieth neutron fluence expected for ITER life time. After the cooling time, the dose rates and the emitted gamma-ray spectra were measured with a survey meter and a Ge detector, respectively.

Induced major radioactive nuclides are Nb-92m, Sn-117m, Sn-113 and Co-60 for Nb₃Sn, and Nb-92m, Sc-46 and Co-60 for NbTi. Also Na-24, Na-22, Sc-47, Co-57, As-74, Rb-83, Mn-54 and some unknown radioactive nuclides were observed from the irradiated GFRP. It is considered that those radioactive elements are due to the activation of the additive compounds in GFRP. The initial dose rates of the irradiated Nb₃Sn, NbTi and GFRP samples were 160 μSv/h, 120 μSv/h and 20 μSv/h, respectively. After 5 months from the irradiation, these dose rates were reduced to 2 - 4 μSv/h. Also, from the measured gamma-ray spectra, it was found that the final dose rates of Nb₃Sn and NbTi wires were dominated by Co-60 (5.27y) and the that of GFRP was dominated by Na-22 (2.60y).

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12-63

Evaluation Of Tritium Behavior In Concrete Materials

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Concrete walls used in a fusion reactor or a tritium handling facility has an important role to separate the atmosphere in a facility from environmental air, since the substantial tritium leakage is possible from a tritium system to the atmosphere of a tritium handling facility. Then, it is necessary from the viewpoint of safety control of tritium to quantify the tritium behavior in concrete used as the final barrier of a multi-confinement system of tritium. Although there are many reports on the behavior of tritiated water in concrete materials placed in water, the article about the behavior of tritiated water in concrete materials in the air with tritiated water vapor has been hardly reported.

Experiments to measure the transfer rate of tritium into columns made of cement paste were performed in this study. Only one face of a column was exposed to the air with vapor of tritiated water for certain days to several weeks and change of the tritium profile in the axial direction of column with time was observed from measurement of tritium trapped in pieces of cement paste, which had been cut off from the sample column. The standard size of a cement column in this study was 13mm in diameter and 100mm in length, and all surfaces of the column except one circular surface were sealed with the waterproofing paint and tape. Comparison of the experimental results with the estimated profile using adsorption isotherm of water on cement paste, diffusivity of water in cement matrix and isotope exchange properties in cement paste reported elsewhere by the present authors showed good agreement. It is ascertained from comparison in this study that the isotope exchange reaction gives a profound effect in transfer of tritiated water in cement materials because there is a large amount of the structural water to work as the isotope exchange capacity. Comparison of the penetration rate of tritium in concrete with that in cement paste was also performed in this study.

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Recycling of fusion reactor materials

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The inherent safety and environmental advantages of Fusion power in comparison with other other energy sources plays an important role in the public acceptance. No waste burden for future generations is therefore one of the main arguments to decide for fusion power. The waste issue has thus been studied in several documents in the final conclusion of which it is stated that there is no Permanent Disposal Waste if recycling is applied.

The paper describes the experiences gained in existing melting facilities on recycling and reuse of radioactive materials from present fission reactor decommissioning projects.

All steps of the recycling process were discussed with experts of existing melting facilities in Western Europe and the United States and the manufacturers of complex pieces for fusion reactors like blanket and divertor modules.

With this package of information the paper will highlight the difficulties and challenges to be faced to allow future recycling of fusion materials. Starting from components composition, function and expected activation, the feasibility is analyzed for every step of a recycling process: dismantling, melting, re-fabrication, etc. Evaluating the recycling option requires a detailed study of the overall material cycle occurring in a fusion plant during its exploitation: i.e. the different recycling paths, the timing aspects (recycling continuously during operation or at decommissioning time), using interim "decay" store or not and how long, etc. Alternative ways to close the material cycle loop are also analyzed in the presentation.

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Some Oxidation and Inflammation Characteristics of Compact Beryllium and Its Fine Particles

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The inflammation, combustion and detonation of metallic beryllium and its fine suspensions may occur at high temperatures and under specific conditions and should therefore be treated as a separate subject of inquiry relating to the safety of fusion reactors using beryllium.

The inflammation of metal particles is severely hampered by oxide films, forming a diffusion barrier on the surface of the particles as a result of preflame oxidation. In many cases, a preliminary destruction of the oxide film is necessary to initiate the inflammation. The inflammation of metals can be triggered not only by a thermal equilibrium disruption, but also by the diffusion of an oxidant.

Ambient temperature enabling the inflammation of fine Be particles is found to be dependent upon oxidant concentration. Where the hydrogen-oxygen ($O_2 < 20\%$) flame temperature is within 2600 K, particles of 30-35 μm in diameter practically do not ignite. For flames with lower free oxygen concentrations, the critical ambient temperature level is higher, gradually reaching beryllium $T_{\text{boil}} = 2750$ K. However, even at high oxygen concentrations of flame ($O_2 > 20\%$), there is no limit, in a strict sense, on the critical ambient temperature, as there is in the case of aluminium. In the temperature range 2600-2800 K, the percentage of ignited particles is no greater than 30%. In a dry atmosphere, all other conditions being equal, the general percentage of ignited particles is higher than in the presence of H_2O . In a hydrogen-oxygen atmosphere at 2900 K, the Be particle inflammation percentage is practically 100%.

The particles start to ignite at about 2380 K, if the oxygen partial pressure exceeds 400-600 kPa. At $P_{O_2} < 10$ kPa inflammation can even not occur at temperatures close to 2650 K.

In enriched combustible mixtures, critical flame temperature necessary to the inflammation of individual particles approaches the Be boiling temperature.

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12-66

Carbon and hydrogen behavior with formation of carbon deposition layer by hydrogen RF plasma

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Carbon deposition layers containing a large amount of tritium have been observed at large tokamaks where graphite materials are used as plasma facing components. Carbon atoms and hydrocarbon molecules generated by plasma-wall interaction at high-energy region are deposited on the wall surface at low-energy region. In order to clarify the tritium trapping mechanism to the deposition layer, it is important to investigate behaviors of tritium and carbon in low energy plasma of a few eV. In this study, carbon deposition layers were formed on quartz substrates by sputtering method using hydrogen RF plasma. Total pressure in a vacuum chamber was controlled to be 10 Pa. RF power was supplied to be 150W. The amount of carbon deposition layer and hydrogen retention in the layer were investigated as a function of mean residence time of hydrogen gas. Hydrocarbon concentration in the gas phase during plasma discharge was observed by quadrupole mass spectroscopy (QMS). Hydrogen retention in the layer was measured by thermal desorption method at argon atmosphere.

With increasing mean residence time of hydrogen gas, the total amount of carbon deposition layer increased. It is considered that carbon concentration in a chamber increased by an increase of mean residence time. Consequently, a deposition rate of carbon layer would increase. It was found that methane, ethane and acetylene were generated in the plasma. These hydrocarbon concentrations did not depend on mean residence time of hydrogen gas. When mean residence time of hydrogen gas was changed from 1.1 minute to 2.3 minute, hydrogen retention, H/C in the deposition layer decreased from 0.25 to 0.1. It is considered that the ratio of ion flux to carbon flux on the substrate surface decreased by increasing carbon concentration. As a result, the atomic ratio of hydrogen to carbon in the layer would decrease.

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12-67

Coolant Pipe Activation due to Sequential Reactions by Charged Particles

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Sequential reactions caused by charged particles that are produced via primary neutron reactions may generate non-negligible radioactivity in fusion material. Especially in coolant near fusion plasma, large amount of charged particles are produced by impinging neutrons. The reactions make the wall material further activated in addition to the radioactivity induced by direct neutron reactions. It is important to precisely estimate the radioactivity, because it dissolves into the coolant as corrosion product and enhance the dose rate around the coolant loop. A method was devised to calculate the radioactivity caused by the impinging effect on the basis of the ACT4 code that had been developed for the activation analysis of fusion reactor designs. The code had been recently revised to calculate the activation by the sequential charged particle reactions. An experiment simulating the neutron irradiation on the coolant and the pipe wall was analyzed to confirm the fitness of the method, and the enhancement of radioactivity in corrosion product due to the impinging effect was estimated for the cooling loop running through the first wall.

The experiment was performed with a 14 MeV fusion neutron source (FNS) facility in JAERI. Several sheets of laminated samples were stacked on a polyethylene slab, and the polyethylene surface was irradiated with 14 MeV neutrons. In the analysis of the experiment, the wall material was infinitively diluted in the coolant to simulate the charged particle generation. The result thus calculated was converted to the radioactivity in the full content pipe wall material. When the low activation ferritic steel F82H was supposed to be the pipe wall material, ⁵⁶Co ($T_{1/2} = 77.27$ days) is the influential radioactive nuclides produced by way of sequential ⁵⁶Fe(p,n) reaction. As a result of the analysis, the activity turned out to be about 50 times more produced by the impinging effect than that only in F82H. Since some of the dominant residual active nuclides emit only a little gamma ray, the gamma ray dose rate produced by ⁵⁶Co may account for several % of the total dose rate around the coolant pipe about 4 months after the reactor shut-down.

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Desorption Mechanism of Water Adsorbed on Metal Oxide Films

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From the viewpoint of tritium safety in fusion reactors, an effective method for tritium decontamination from vacuum chamber vessels and piping materials is required. The light irradiation to the surface of these materials is considered as one of the effective techniques. For the application of this technique, it is important to elucidate the desorption mechanism of water induced by the light irradiation. The present work aims to understand the desorption behavior of water adsorbed on metal oxide films, specifically those of titanium and iron. The titanium (Ti) was chosen to evaluate efficiency of its use as an additive component due to its photoelectric property, and the iron (Fe) to verify the behavior of base component of stainless steel. Furthermore, their alloy (Ti-Fe alloy) was inquired focusing on the effect of electron transfer between these two oxides.

The surface of Ti, Fe and their alloys was heated at 673 K for 2 hours under oxygen atmosphere of 10 Pa in order to oxidize the surface of the materials. Thereafter, water was adsorbed on the surface by water vapor exposure. After this pretreatment, the Nd:YAG laser with the wavelength of 230-500 nm was irradiated on the sample surface. The chemical form and the amount of particles desorbed by the laser irradiation were analyzed using time-of-flight (TOF) technique.

The water adsorbed on the surface was desorbed as H₂ and H₂O forms by the laser irradiation. The velocity distribution of H₂O was fitted only by a Maxwell-Boltzman distribution function or combination of a Maxwell-Boltzman and a modified Maxwell-Boltzman distribution functions. These results showed that the desorption was initiated by the thermal process or the combination of the thermal process and the photo-stimulated desorption (PSD) process. The amount of H₂O, which was desorbed from the oxide film of Ti-Fe alloy via the PSD process, was not described by the sum of those from the Ti and Fe oxides. This fact indicated the existence of PSD process caused by the electron transfer between these two metal oxides.

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12-69

Recycling and Clearance of Vanadium Alloys in Fusion Reactors

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Design of fusion reactors includes the development of low-activation materials. V–Cr–Ti alloys are among the candidate structural materials, with the scarce and costly V as the main component. Therefore, it is worth considering the alloy regeneration and refabrication as well as to avoid the disposal of the irradiated material as radioactive waste. However, to be able to safely reuse and handle such a material, it is necessary to bring its effective dose rate down to sufficiently low levels. Furthermore, in case of components far from the plasma, and then less exposed to neutron activation, the declassification of the materials to non-radioactive ones (clearance) may be reachable too.

Maximum radionuclides concentrations in the alloys allowing either clearance or hands-on refabrication within the nuclear industry were determined, using new IAEA Clearance Limits and recycling dose-rate limits.

Tritium retention in V-based alloys may be high due to high solubility of tritium in those materials. However, adequate detritiation techniques may be applied, in order to reduce tritium concentration in spent materials down to acceptably low levels.

The following results have been obtained:

- First wall and blanket structures: recycling may be a reachable goal for V-based alloys, provided that some activation products are eliminated.
- Less neutron-exposed structures: clearance is possible, but here also certain activation products must be separated.

In both cases, it turns out that the development of isotope chemical separation techniques is interesting and necessary for our purposes.

A suitable method for achieving the required substantial radioactivity reduction of activated V–Cr–Ti alloys is radiochemical extraction reprocessing. Such a technology, permitting to remove metallic activation products from spent materials, was developed and tested experimentally in Russia.

Concerning recycling of first wall and blanket components, based on the estimated element distribution factors in the extraction and re-extraction processes, and computations, it was shown that the alloy components may be purified from the activation products, using this technology, down to an effective contact dose rate of ~10 $\mu\text{Sv/h}$ in a cascade consisting of ~50 extraction stages. Such a radioactivity permits materials recycling in “hands-on” conditions.

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12-70

Activation characteristics of the materials in the fusion-driven subcritical system FDS-I

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The fusion-driven subcritical system (named FDS-I) was previously proposed as an intermediate step toward the final application of fusion energy. The reduced activation ferritic-martensitic steel (RAFMs. e.g. CLAM, the China Low Activation Martensitic steel) or 316ss is considered as an alternative structural material in the system. Liquid metal $\text{Li}_{17}\text{Pb}_{83}$ serves as the tritium breeder and one of the coolants to cool the blanket. The other coolant is helium gas to cool the first wall and the structure. The transmutation zones consist of $\text{Li}_{17}\text{Pb}_{83}$, long-lived Fission Product (^{99}Tc , ^{129}I , ^{135}Cs , etc.), Minor Actinides (^{237}Np , ^{241}Am , ^{243}Am , ^{244}Cm , etc.) and Pu etc. One of the main design principles is to maximize the transmutation rate and reduce the radioactivity and hazard levels of the FPs, MAs and Pu etc. and to minimize the potential hazard to environment and public of the activated materials, e.g. structural material, coolant, etc. The activation characteristics of the materials in FDS-I, including the loaded fuels/wastes (i.e. minor actinides and fission products), the supporting material (i.e. structural material) and the coolant etc., were calculated based on the three-dimensional (3D) geometry model to assess potential impact of system on safety and environment. Furthermore, the impact of the impurities in the materials on the activation characteristic was analyzed.

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12-71

Development of China Liquid Lithium-Lead Blanket and Its Material Technology

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A series of fusion demonstration reactors (named FDS series) have been designed and assessed in China which cover several types of liquid lithium-lead blankets including the RAFM steel-structured helium-cooled quasi-static lithium-lead tritium breeder (SLL) blanket, the RAFM steel-structured helium gas/liquid lithium-lead dual-cooled (DLL) blanket, the SiC/SiCf composite-structured high temperature lithium-lead (HTLL) blanket oriented to hydrogen production application and the austenitic stainless steel-structured helium gas/liquid lithium-lead dual-cooled high level waste transmutation (DWT) blanket etc, among which the RAFM-structured liquid lithium-lead blankets are the primary candidate because of their relatively mature technology base.

To demonstrate and validate the feasibility of the candidate blankets, the strategy for TBMs (test blanket modules) development has been proposed, which covers three-phases e.g. Out-of-pile experimental Mockup in liquid lithium-lead experimental loops, EAST-TBM (the Test Blanket Module for the Experimental Advanced Superconducting Tokamak EAST under construction) and ITER-TBM (the Chinese design named DFLL for ITER). The reference preliminary designs of three typical TBMs have been completed in wide collaboration with various institutions in China.

In this contribution, a design overview of FDS series demo reactors, lithium-lead demo blankets and relevant TBMs are presented as well as a summary of the ongoing and planned material R&D activities towards liquid lithium-lead blanket applications in China, including blanket-relevant technology experiments such as corrosion and MHD effects of liquid LiPb in lithium-lead experimental loops under construction, development of structural steels and MHD-reduced insulation coating and flow channel insert, and blanket mockup fabrication etc.

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12-72

Irradiation Effect in Zr-Based Metallic Glasses

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Since Zr-based metallic glasses have novel characteristics, such as extremely high mechanical strength and super-soft magnetic properties, they have a great potential for applications to functional materials. Although the metallic glasses can be attractive and useful in fusion devices, few studies have been reported on irradiation effects of them. The aim of this work is to clarify the structural and compositional changes of Zr-based metallic glasses under ion, electron and neutron irradiation.

Specimens used were $Zr_{55}Ni_5Al_{10}Cu_{30}$, $Cu_{55}Zr_{40}Al_5$ and $Ni_{60}Nb_{20}Zr_{20}$, prepared by single roller melt-spinning method, cut into a size of 10 x 5 x 0.02 mm. Metal ions such as Cu, Ag, Au were implanted at room temperature using a tandem accelerator with an energy range 0.5 to 1.5 MeV, to the dose range 4.7×10^{18} to $8.0 \times 10^{20}/m^2$. Electrons with 300 keV were irradiated at room temperature to the

dose range 50 to 100 kGy. Irradiation was also performed in Japan Materials Testing Reactor (JMTR) at 373K, 513K and 573K. After the irradiation, heat treatment was carried out for 30s in temperature range from 573 to 903 K, in vacuum of 6.6×10^{-5} Pa. Characterization of the irradiated surface layers was performed by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Rutherford Backscattering Spectrometry (RBS) and X-ray photoelectron spectroscopy (XPS).

Structure and composition of specimens were not changed by irradiation in the present experiment condition. Thin ZrO_2 layer was formed on the surface irradiated by ions and neutrons at higher doses, although no Zr oxides were detected for the electron irradiation. Because the projected ranges of the incident ions were far beyond the oxides layer, the formation of Zr oxide layer can be attributed to the collisional effects that lead to absorb oxygen from residual gas. Moreover, when the specimens irradiated by ions were annealed at crystallizing temperature, Zr_2Ni precipitated on the surface layer. In contrast, Zr_2Ni crystallization was suppressed in the specimen irradiated by electron at higher doses. These results will be discussed by surface analyses using XRD, XPS, RBS and SEM.

An Interactive Web-based Fusion Materials Properties Database

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The rejoining of ITER by the U.S. fusion program requires efficient material property data information exchange between various participants of the U.S. ITER Test Blanket Module (TBM) development program. Accessibility to the latest and up-to-date materials properties for ITER TBM development is essential. Internet technology is providing the means to develop such an efficient on-line fusion materials database. The combination of advanced Internet programming with Relational Database Management Software (RDBMS) provides an effective technology for interactive, scalable, and highly automated materials property database web site with Internet efficiency.

The architecture and the tools necessary for such an on-line RDBMS-based materials property database have been developed for the U.S. fusion materials community. The key feature of this RDBMS materials database web site is that archiving, publishing, and updating of content is performed with the involvement of the fusion materials property data community. Furthermore: (1) data is presented with a uniform standard in both graphical and tabular format; (2) original publications of hosted data is available in PDF format;

(3) built-in search engines can readily locate available data; (4) the materials database is scalable and expandable; (5) older data is never removed, new data is simply added to the database; (6) new data can be submitted over the Internet directly to the site by participating users; (7) quality assurance is performed with direct involvement of the data's author.

Another advantage of the RDBMS-based material property database is that equivalent design equations can be generated for all tabulated materials property data. The database provides an option to choose from a large number of empirical and/or physical based design equations for modeling materials properties. The materials data is categorized into thermo-physical, mechanical, chemical, and microstructural properties, which are readily accessed using both textual information and thumbnail sketches. The database is currently hosting data on low activation ferritics, with a focus on F82H. Efforts are well underway to provide material property data of other ITER TBM relevant materials, such as liquid and solid breeders and other advanced ferritics.

Keywords: Material Properties, Database, Web-based, RDBMS, F82H

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12-74

Development of 300 °C heat resistant boron-loaded resin for neutron shielding

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In National Centralized Tokamak, the JT-60 superconducting modification [1], the resin is installed between the vacuum vessel and the TF coil in the cryostat. The baking temperature for the wall conditioning will be planned to ~ 300 °C in the device. The neutron shielding resin which set up around the vacuum vessel need to the heatproof. So that, the boron-loaded resin with the heat resistance applicable to 300 °C has been newly developed as a neutron shielding material.

In the developed resin, boron carbide (B₄C) was loaded with phenol-based resin that has improved heatproof. The density of the boron-loaded resin is 1.8 g/cm³ and the main composition is B, C, H, N, and O. Hydrogen as a moderator of DD neutron (E_n = 2.45 MeV) and boron as an absorber of thermal neutron provides neutron shielding. The amount of boron was chosen to 5 wt% to absorb the thermal neutron estimated by 1D ANISN calculation.

Neutron penetration tests of the developed resin with baking and without baking have been performed using DD neutron sources at Fusion Neutronics Source facility in JAERI. In DD experiment, fast neutrons (E_n > 2 MeV) and thermal neutrons was detected by the ³²S(n,p)³²P reaction and ³²P(n,γ)³²P reaction, respectively. The fast neutron and the thermalized neutron attenuation rate at 10 cm thickness showed 8 % and 32 %. The neutron shielding performance of the developed resin after baking was almost same as that before baking.

In the outgas characteristics, the main outgas component from the resin at 150–300 °C was CO₂, NH₃, and H₂O. The weight of the resin (21.5 mg) has decreased by 1.1 %. Additionally, the 13 kinds of organic gases such as the pyridine have been observed by the amount of micro-g/g.

So, it can be concluded that a 300 °C heat-resistant neutron shielding resin is very attractive as the neutron shielding material.

[1] H. Tamai, et al, IAEA Fusion Energy Conference (2004), IAEA-CN-116/FT/P7-8.

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12-75

Materials selection and requirements for DEMO in a fast track development of fusion power

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This paper is concerned with the key aspects of materials selection issues and requirements for DEMO, as these have emerged from the combination of recent European studies of: (i) fusion power plant conceptual designs; (ii) the fast track development of the first generation of commercial fusion power plants.

The objective of the international fusion program is the creation of power plants that will have very attractive safety and environmental features and viable economics. Fusion power plant studies have shown that these objectives may be achieved in a variety of conceptual designs based on relatively near-term plasma physics together with blankets and divertors based on reduced activation martensitic/ferritic steels as the structural material in association with a variety of coolants, neutron multipliers, tritium generating materials and armors. Advanced materials are not required, though the development of these would bring further performance benefits. The urgent need to find global solutions to the provision of environmentally benign sources of power has led to the widespread acceptance of a 'fast track' approach to fusion development. Fast track studies have analyzed *inter alia* the requirements on DEMO(s) that would form the only step between the ITER/IFMIF generation of devices and the first generation of commercial plants.

This paper, based on technical work further developing the above studies, analyzes the materials selection issues and requirements for DEMO, and draws conclusions on the prioritization of R&D.

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12-76

Deformation and Damage of RAFM Steels under Thermomechanical Loading: A Challenge for Constitutive Equations

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Reduced activation ferritic martensitic (RAFM) steels as structural materials of fusion reactors will be subjected to complex thermomechanical loading and high irradiation doses. Correct modelling of their deterioration under these loading conditions is a precondition of a sufficiently reliable lifetime prediction procedure. Therefore a coupled deformation damage model taking into account the complex non-saturating cyclic softening of RAFM steels has been developed and successfully applied to describe the creep-fatigue behavior of F82H mod and EUROFER 97 under isothermal cyclic loading.

For verification the fitted model is applied to thermal fatigue tests performed on tube specimens. Previous evaluation of these tests showed that thermal fatigue loading leads to remarkably reduced lifetime in comparison to isothermal fatigue loading with the same mechanical strain range. In this evaluation changes in the stress range as well as actual test conditions, e.g. temperature gradients, were not considered. However, when applying the coupled deformation damage model changes in stresses between isothermal and thermal loadings will be automatically taken into account. For considering the actual test conditions the model has been implemented in the finite element code ABAQUS so that the thermal fatigue tests can be fully simulated by finite element calculations.

In the paper to be presented the thermal fatigue tests considered and how they were performed will be described. Thereafter the results of their simulations using the new coupled deformation damage model in combination with the finite element method will be presented and discussed.

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12-77

Multiaxial Fatigue Behavior of EUROFER 97: Experiments and Modeling

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First wall materials of a fusion reactor will be exposed to different complex loading conditions. One of these complex loading conditions is represented by multiaxial fatigue. For the assessment of the lifetime behavior of the ferritic martensitic steel EUROFER 97 under multiaxial fatigue conditions proportional and non-proportional multiaxial fatigue tests on tubular specimens has been performed under purely alternating strain-controlled loading. Both the load paths and the phase shifts were varied.

The first test series at room temperature has been performed with fixed directions of the principal stresses and strains. For this purpose a biaxial test facility was used, which generates the circumferential strain by a pressure difference of the surrounding media. A second test series has been conducted under alternating tension-torsion loading. In this case no fixed principal axis system results. In fact, rotating principal stresses and strains exists. In a further step, tests will be performed at elevated temperatures (500°C). The results obtained reveal the influence of the phase shift for different temperatures as well as the influence of test execution method (fixed or rotating principal stress system) for the same material.

Furthermore, the results will be used to verify and, if necessary, modify two approaches to lifetime prediction, which were developed and used successfully to describe the behavior of AISI 316 L (N) under non-proportional multiaxial loading.

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12-78

Statistical Assessment of Representativeness of Mechanical Testing Results To Substantiate The Guaranteed Level of Strength Properties of ITER Structural Materials

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ITER structural materials such as copper alloys for the divertor and first wall; steel 316 L(N) for the first wall operate under high thermomechanical stresses. According to the ASME requirements it is necessary to define the guaranteed level of strength properties for these materials at room temperature and in the operation temperature range (including the effect of neutron irradiation).

Sufficient data base on the properties of 316 L(N) steel and copper alloys such as CuCrZrIG and GlidCopAl25IG have been accumulated in the context of ITER R&D, which allows for the statistical analysis of the representativeness of these data. Sufficient data are available making it possible to compare the effect of slight difference in heat production technology on scattering in materials properties.

This paper describes an attempt of statistical analysis of the correlation between the results of mechanical tests for 316 L(N) steel and CuCrZrIG alloy obtained by several research groups in slightly different test conditions.

It is shown that with certain restrictions for the strain rate and material condition, most results agree with each other. Sufficiently large number of the test results for 316 L(N) steel allows us to establish with certainty the level of the minimum strength properties ($S_{m \min}$). As observed, $S_{m \min} - S_{m \text{ aver}} \sim 30$ MPa, i.e. the scattering in data is rather small.

Since the volume of data for CuCrZrIG is much less, there is an observed decrease in the $S_{m \min}$ value, as compared to $S_{m \text{ aver}}$.

It is worth noting that in case of irradiated material the problem with the guaranteed level of strength properties becomes more complicated. While strength properties of the irradiated materials are systematically higher and the number of samples is less, high scattering in the data causes a decrease in the $S_{m \min}$ value with 95% confidence.

The conclusion is possible that the accumulated database on the strength properties of ITER materials is statistically representative and different data sources provide reasonably close results. The database on irradiated materials is insufficient (only 2-3 tests for a test point), making it difficult to obtain high-confidence values for the minimal and average strength values.

Extending ITER Materials Design to Welded Joints

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This paper extends the current ITER materials properties documentation, prepared in anticipation of forthcoming construction and licensing phase, to weld metals. It also incorporates the needs of Test Blanket Modules for higher temperature materials properties.

Since the main structural material retained for ITER is type 316L(N)-IG, the paper is focused on weld metals and joining techniques retained for this steel. These are mainly materials developed and tested through 3 generations of French fast breeder reactors, and subsequently implemented in the European FBR and fusion programs, namely 16-8-2 and low and high temperature grades of 19-12-2 weld metals.

Materials properties data are analyzed according to the RCC-MR code procedures and design allowables are equally derived according to RCC-MR, but they remain also compatible with the ASME code. A particular attention is paid to the type of weld metal, to the type of welding and to the position of welding (flat, vertical, above head, etc.) and their influence on the materials properties data and design allowables.

The primary goal of this work is to produce comprehensive materials properties documentations that when combined with codification and inspection documents would satisfy ITER licensing needs. As a result, structural stability and capability of welded joints during manufacturing of ITER components and their subsequent service, including the effects of irradiation and eventual incidental or accidental situations, are also covered.

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High Heat Flux Performance of He-cooled Divertor Module for DEMO Reactor

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Further development of a helium-cooled divertor concept for fusion reactor like DEMO depends strongly on the progress in selection of suitable materials and technologies of their manufacturing and joining as well. Current design of divertor uses He-cooled thimble-like structure made of tungsten which is additionally covered by sacrificial tungsten tile. Each cooled thimble has to be connected with supporting unit made from ferritic steel.

The most suitable materials for mentioned divertor design and optimization of methods of their joining were experimentally selected on the previous stage of investigation. In particular, fast brazing at 1050°C of protective tungsten tile to cooled tungsten alloy thimble using rapidly quenched amorphous STEMET[®] alloy and following thermal cycling of joint at 14 MW/m² has demonstrated its reliability. Joining of tungsten thimble to ferritic steel supporting structure via mechanical conical interlock filled with a cast copper revealed satisfied properties (withstood 10 thermal cycles RT - 600°C with 10 MPa inner He pressure). Basing on mentioned experimental results the number of He-cooled thimble-like divertor modules having hexagonal protective tungsten tile was manufactured with the purpose of their high heat flux testing in DEMO expected environment.

Aiming to simulate DEMO relevant conditions unique closed He-cooling loop (10 MPa inlet pressure at 600°C) was specially built up. He-cooled divertor modules were integrated into He-loop and subjected to high heat flux on TSEFEY-M electron beam facility (Efremov Institute, Russia). The value of the heat flux absorbed by tested module was varied in the range 10 – 15 MW/m². The number of thermal cycles applied to each module was varied from 100 to 1000. To enhance heat transfer efficiency various cartridges (having an array of holes or slot array geometry) were inserted inside the thimbles.

Paper gives the main results of the high heat flux test and the first data evaluation. Properties of tungsten protective tile (tendency to cracking, DBTT, recrystallization), tile/thimble and thimble/steel joining areas before and after testing were compared. Authors discuss applicability of used materials to DEMO divertor. Nearest future plans in continuation of given work are also presented.

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12-84

Strengthen CVI-SiC Matrix in SiC/SiC Composites by SiC Nanowires

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SiC/SiC composites are one of the most attractive candidate structural materials for fusion because of their potential applications for high performance reactors and superior safety characteristics compared with metallic materials. Favorable features of SiC/SiC composites are the high temperature properties and the low activation characteristics. Chemical vapor infiltration (CVI) process is the leading process among several currently available ones, which allows for the production of radiation-resistant stoichiometric crystalline β phase SiC matrix and has other important advantages such as a modest processing temperature and a high flexibility. However, CVI matrix densification stops when the surface pores are closed, leaving a typical porosity of 10~15% in the materials. Such a porosity decreases the matrix cracking stress that limits the maximum applicable stress to composites when fatigue, creep rupture, and/or stress corrosion cracking are of main concerns. The purpose of this study is to further strengthen the matrix, and hence, the composite of CVI-SiC/SiC composites by

incorporating single crystal SiC nanowires in the composites. Single crystal SiC nanowires are well known to possess very high strength and are suggested as a good reinforcement material for ceramic matrix composites.

A recently developed CVI-SiC nanowire process was applied to grow SiC nanowires in the preforms of several plain-woven Tyranno-SA/SiC composites prior to the CVI matrix densifications. The volume fraction of the nanowires in the composites is 0, 1.6, 5.7, and 6.1%, respectively. The nanowires were CVI-coated with thin carbon layer as the nanowire/matrix interphase. The mechanical properties were and are to be evaluated using the simple three-point bending method for a comparison with previous observations on Tyranno-SA/SiC.

The present results showed positive relationship between the amount of the nanowire and the flexural stiffness, proportional limit stress, and ultimate flexural strength of the composites. The reinforcement efficiency of the nanowires is remarkable. The ultimate strength increased from 380 ± 113 to 750 ± 103 MPa for the composites with 0 and 6.1% of nanowires, respectively. Further efforts on the mechanical properties, microstructure inspections and the effects of the nanowires on the fracture (matrix cracking) behaviors are under going, and will be reported at the conference.

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Microstructure and Mechanical Property of Fiber/Matrix Interphase in SiC/SiC Composites after Irradiation

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The mechanical properties of continuous fiber-reinforced ceramic matrix composites depend on the properties of their constituents, especially fiber/matrix (F/M) interphase. SiC/SiC composites reinforced with less crystalline and non-stoichiometric SiC fibers have shown the interfacial debonding due to mismatch of swelling behavior of the fiber and β -SiC matrix after neutron irradiation. In contrast, due to highly-crystalline and near stoichiometric SiC fibers, advanced SiC/SiC composites have exhibited excellent irradiation stability in ultimate bend/tensile strength. However, the result of a slight loss of proportional limit stress (PLS) suggested degradation of F/M interphase. Therefore, irradiation effects on the interfacial role became one of the most important issues to be discussed. The purpose of this study is to clarify the microstructural stability and mechanical property changes in F/M interphase in SiC/SiC composites after ion/neutron irradiation.

The materials were advanced SiC/SiC composites reinforced with uni-directional, near-stoichiometric SiC fibers and fabricated by isothermal chemical vapor infiltration method. F/M interphase was single pyrolytic carbon (PyC) and (C/SiC)₅ multilayer. Neutron irradiation was conducted at JMTR. Neutron fluence was $1.0 \times 10^{25} \text{ n/m}^2$ ($\approx 1 \text{ dpa}$, $E > 0.1 \text{ MeV}$), and radiation temperature was 1073 and 1273K. The effect of irradiation on tensile and inter-laminar shear strength was evaluated by cyclic loading tensile and double notched specimen (DNS) test. Ion irradiation was carried out at DuET Facility in Kyoto University. Displacement damage level was up to 100dpa and irradiation temperature was up to 1673K. F/M interphases after the irradiation were observed by transmission electron microscopy (TEM).

From the tensile test, a slight loss of PLS was measured. And from the DNS test, downward tendency of inter-laminar shear strength was also confirmed. These results suggested degradation of F/M interphase. From microstructural analysis of PyC using TEM, the expansion of PyC layer in thickness was observed after ion irradiation. In high resolution TEM observation, the basal planes of the irradiated graphite-like carbon appeared to be chopped into small fragments and consequently amorphous-like structures were often observed in the irradiated region. This tendency was confirmed in the c-axis length. The detailed microstructural changes after ion/neutron irradiation will be discussed correlated with the mechanical properties.

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12-86

Efforts on Large Scale Production of NITE-SiC/SiC for Test Blanket Module of ITER

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The attractiveness of SiC/SiC for nuclear application is based on the excellent potentiality in chemical stability at very high temperature, inherent heat resistance and stability and low induced radio-activation property under neutron environment.

To indicate the feasibility of fusion reactor by utilizing newly developed NITE-SiC/SiC composite materials, R & D on NITE process with near net shape foaming having been carried out. In order to the establishment of large scale production of NITE-SiC/SiC, the pilot grade (PG) of NITE-SiC/SiC were fabricated and their baseline properties were evaluated. As the key elements, nano-powder fabrication and Tyranno-SA, SAK fabrication are extensively being developed.

SiC matrix formation at intra fiber bundles is depends on mixed slurry infiltrability into fiber bundles. In order to improve slurry infiltration, Tyranno-SAK fiber with 800 filaments per a bundle has been newly produced. Recently produced PG #3 NITE-SiC/SiC composites with Tyranno-SAK shows sound microstructure, low porosity and less fiber deformation compared to those of PG#1 and #2 with conventional Tyranno-SA, which has 1600 filaments per a bundle. And density and ultimate tensile strength of unidirectional PG#3 NITE-SiC/SiC are about 3.02 g/cm² and 397 MPa respectively. These values are almost same or higher than those of Lab. Scale NITE-SiC/SiC composites. The NITE-SiC/SiC composite of cylindrical shape was also fabricated by a near-net shape process called pseudo-HIP, which was a new type HIP using a carbon powder as the pressure transmitter.

The microstructure of NITE-SiC/SiC composites, such as fiber volume fraction, porosity and type of pores, can be controlled precisely. It makes it possible to produce Test Blanket Module (TBM) with proper thermal conductivity in response to the requirement of fusion reactor design. The trial to meet the different requirement for gas cooled solid blanket and dual cooling liquid blanket is on going and the results will be also presented.

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12-87

Mechanical Properties of Tyranno-SA/SiC Composite Prepared by the Whisker Growing Assisted CVI Process

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SiC_f/SiC composite is one of the typical continuous fiber reinforced ceramics. By using a long fiber to reinforce the ceramic matrix, the fracture toughness has been significantly improved. In addition to the thermo-mechanical advantages of SiC_f/SiC composites, a low induced activation by neutrons and a good irradiation resistance have also made them quite attractive for fusion reactor applications. Chemical vapor infiltration (CVI) is one of the main processes commonly used to fabricate SiC_f/SiC composites but it is a slow process with an inherent drawback of a substantial residual porosity. If the whisker growing process was applied prior to the conventional CVI process, called the whisker growing assisted process, dense SiC_f/SiC composite with a homogeneous microstructure could be obtained by dividing the large natural pores between the fibers or bundles by whiskers and then by effectively filling of the matrix through the modified pore structure during the process. Especially, if the number of the cycles of a whisker growth and the matrix filling was properly adjusted, the maximum size of the large voids would be decreased and the homogeneity of the voids distribution would be increased. This enhanced microstructure could result in improved properties of SiC composites.

In this study, SiC_f/SiC composites were made by the whisker growing assisted process using a plain weave fabric of Tyranno-SATM as a reinforced substrate. Ten layers of the fabric with a diameter of 50 mm were stacked as a green preform. Before the whisker growth, the preforms were coated with pyrolytic carbon using methane gas (CH₄). The mechanical properties such as flexural, elastic modulus, hardness, fracture toughness and interlaminar strength were measured and some results will be described with the thickness of a PyC interlayer. Additionally, the fracture behaviors of these composites will also be evaluated.

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Mechanical Properties of SiC/SiC Composite with Magnesium-Silicon Oxide Interphase

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Continuous SiC fiber reinforced SiC composites (SiC/SiC) are one of the candidate materials for fusion structural applications due to their excellent high temperature mechanical properties and low induced radioactivity after neutron irradiation. Conventional interphases between the fiber and the matrix, such as carbon and boron nitride, were degraded under neutron irradiation and oxidation environments. Therefore it is important to develop the interphase for excellent mechanical properties in SiC/SiC. Oxide interphases become a potential candidate to improve those properties since advanced SiC fibers have been produced, including Hi-Nicalon Type S and Tyranno SA which show the oxidation resistance and excellent performance under neutron irradiation as well as superior mechanical, thermal properties. In this study, we investigated the

magnesium-silicon-based oxide for the interphase of SiC/SiC due to thermal stability under the mild oxidizing environment and low induced radioactivity under 14-MeV neutron environment.

The magnesium-silicon-based oxide interphase was formed on the SiC fiber fabrics by alkoxide method. SiC fiber fabrics were dipped in the coating solution. After dipping, the fabrics were hydrolyzed at room temperature and heated in air to decompose the alkoxide. SiC/SiC composite was fabricated by the chemical vapor infiltration method using those fabrics. Microstructure of SiC/SiC composite was observed by SEM. The mechanical properties were evaluated by 3-point bending test. The SiC/SiC showed ductile failure behavior after the maximum load. The load did not become zero after the large deformation since the specimen was not turn off due to the tolerance of fibers. The fiber pull-out was observed in the composite. Those results indicate that the magnesium-silicon based oxide has a potential to good properties as an interphase of SiC/SiC.

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12-89

Reaction Sintering of Two-Dimensional Silicon Carbide Fiber-Reinforced Silicon Carbide Composite by Sheet Stacking Method

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Continuous silicon carbide fiber-reinforced silicon carbide (SiC/SiC) composite is one of the most attractive structural materials for future fusion reactors. Many researchers have studied the fabrication process of SiC/SiC composites. Present authors have explored a new fabrication process by hot-pressing using sheet stacking method which offers the ability to obtain dense composite and to simplify the fabrication process, and the dense SiC/SiC composite which showed non-brittle fracture behavior could be obtained. Bending strength and thermal conductivity of the composite were 240 MPa and 14 W/mK, respectively.

On the basis of this process, we have paid attention to reaction sintering which has some advantages such as a lower processing temperature and the possibility of densification and near-net shape of the composite with complex shape, therefore we tried to fabricate SiC/SiC composite by reaction sintering

using sheet stacking method in order to further increase mechanical and thermal properties of the composite and to obtain flexibility of manufacture process of SiC/SiC composites.

Green sheets containing β -SiC and carbon were prepared by doctor-blade method. The ratio of carbon/SiC was 0.5 - 0.7. Two-dimensional SiC fiber cloth was impregnated with the slurry containing β -SiC and carbon in vacuum and then dried. This impregnation process was repeated some times. The green sheets and SiC fiber cloths were stacked alternately, and then heat-treated at 350°C in air to decompose the binder and organic materials. The compact was set in a graphite crucible with Si and B powder, followed by reaction sintering at 1450°C for 2 h in vacuum.

The bulk density and bending strength of SiC/SiC composite in C/Si ratio of 0.6 was higher than that of the composite in C/Si ratio of 0.5 or 0.7, and the values were 2.9 g/cm³ or 200 MPa, respectively.

In this paper, fabrication process of two-dimensional fiber-reinforced SiC/SiC composites by reaction sintering using sheet stacking method is described and the details of properties of obtained composites will be discussed.

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12-90

Effect of Neutron Irradiation on Tensile Properties of Unidirectional Silicon Carbide Composites

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Silicon carbide fiber-reinforced silicon carbide matrix composites are promising materials for applications fusion reactors. The benefits of using the silicon carbide composites come from the lack of severe strength degradation by neutron irradiation known to date and the low activation/low decay heat properties, in addition to the intrinsic heat resistance of silicon carbide. Assuming the strength and fracture toughness retention for the silicon carbide matrices and fibers during neutron irradiation, integrity of the composites may be determined by the effect of irradiation on the properties of fiber-matrix interface.

In this work, model composites with the chemically vapor infiltrated silicon carbide matrices, uni-directional reinforcement by the near-stoichiometric SiC fibers, and varied interfacial structures were evaluated for tensile properties after neutron irradiation in High Flux Isotope Reactor to $0.8 - 7.7 \times 10^{25}$ n/m² at 573 – 1073K. The preliminary results suggested that the irradiated composites maintain the ultimate strength in many cases, but signs of the significant modification in the interfacial properties were also exhibited. The comprehensive results and details of the interfacial characterization will be presented. Additionally, a methodology of the composite design which incorporates the anticipated effects of neutron irradiation will be discussed.

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12-91

High temperature tests of 2D and 3D SiC_f/SiC composites

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SiC_f/SiC composites are promising materials because of their high temperature mechanical properties, their corrosion resistance and chemical stability. These properties together with the low activation profile of SiC make SiC_f/SiC composites favorable for application in future fusion reactors. However, the irradiation behavior of those composites is of concern, especially with respect to the reduction of thermal diffusivity but also with respect to mechanical strength and swelling. To improve on the thermal diffusivity, new 2D and 3D SiC_f/SiC composites have been developed in the frame of the European Fusion Development programme on advanced materials.

In this paper, the pre-irradiation characteristics of the new 2D and 3D SiC_f/SiC composites will be presented. In addition the paper will report on mechanical and physical properties at high temperatures of new 2D and 3D SiC_f/SiC composites. The test matrix comprises bending up to fracture, bending modulus, resonance modulus and thermal diffusivity. In addition tensile tests have been performed. Also preliminary results of bending creep at 1000°C will be shown. To enhance the deconvolution of irradiation effects from the normal sample-to-sample scattering, identical specimens will be tested before and after irradiation on bending- and resonance Young's modulus and thermal diffusivity.

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12-92

Material Compatibilities Studies between SiC and Solid Breeding Materials for High-Temperature Gas-Cooling Blanket System

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Silicon carbide (SiC) fiber-reinforced SiC-matrix composites (SiC/SiC composites) are considered as a structural material of a gas-cooling solid-blanket system which is expected to obtain a high-efficiency of energy convergent with high operating temperature. Material compatibility behavior between SiC/SiC composites and solid breeding materials is one of key material issues of the blanket system. Reaction of the structural materials and solid breeding materials depends on irradiation temperature, material composition and cooling gas atmosphere. From the view point of material composition, stoichiometry of the solid breeding materials will be changed by Li burning reaction under neutron irradiation. The purpose of this work is to clarify material composition effects on the compatibility behavior of solid breeding materials and SiC at high temperature region.

Li₂O, Li₂TiO₃, Li₄SiO₄, LiAlO₂ and Li₂ZrO₃ were used as tested solid breeding materials. These specimens were fabricated by sintering process as small disk shape tablets which had a diameter of 3mm. The Li compositions were changed from the stoichiometric composition to 5, 10, 15 and 20% less compositions by adjusting Li₂O compositions of raw materials. Monolithic beta-SiC fabricated by CVD and SiC/SiC composites fabricated by CVD were selected as structural material specimen. 3mm disk shape specimens were also prepared from the monolithic SiC and SiC/SiC composites. These disks were piled up and inserted in a He filled quartz tube and heated. The examined heat treatment temperatures were 800, 900, 1000 and 1100C. Holding times are 100h and 1000h. After the heat treatment, structural analysis at the interface region was carried out. In-situ observation of these materials reaction at high temperature region was also examined using a laser microscope with an infrared heater in Ar atmosphere. Results of analysis of the reaction products, reaction rate will be presented.

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12-93

Numerical Analysis of Mechanical Testing for Evaluating Shear Strength of SiC/SiC Composite Joints

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SiC/SiC composites are promising candidate materials for high heat flux components because of their potential for low-activation, low-afterheat and their high-temperature properties. For fabricating large or complex shaped parts of the composites, the technique of joining between simple geometrical shapes is considered to be an economical and useful method. Where, the shear strength of joints is one of the most basic and important mechanical properties to establish useful design database.

On the other hand, the strength of the bonded joint is largely influenced by the geometry of the joint and the test method for evaluating the strength. In order to study these influences, the level of stress and the order of the singularity in stress field are commonly employed for the relative evaluation of strength. Although detailed information on the stress field is provided, little information on the criteria of the fracture is obtained from these types of study. This comes from the fact that, the physics of failure itself is not explicitly modeled in these analyses. The interface element, which directly models the formation of the surface, may have potential capability not only to give insight into the criteria of the fracture but also to make the quantitative prediction of strength itself.

In this research, as examples of the most typical methods to determine the shear strength of SiC/SiC composite joints, the asymmetrical four point bending test of butt jointed composite, the tensile test of lap jointed composite and the compressive test of double-notched joint composite were analyzed by using finite element method with the interface element. Where, the interface potential was assumed to be a couple function of the opening and the shear deformations to describe a mixed mode of mode-I & II fracture behavior by one interface element. As the computational results, it was revealed that the strength in the asymmetrical bending test was controlled by both the bonding strength and the surface energy at the interface although the strength in the tensile test was governed by the surface energy.

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Mechanical Properties of LPS-SiC Ceramics with Al₂O₃-Y₂O₃-SiO₂ System

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Recently, SiC materials have been extensively studied for high temperature components in advanced energy system and advanced gas turbine because it has excellent high temperature strength, low coefficient of thermal expansion, good resistance to oxidation and good thermal and chemical stability etc. However, the brittle characteristics of SiC such as low fracture toughness and low strain-to fracture still impose a severe limitation on practical applications of SiC materials. For these reasons, SiC_f/SiC composites can be considered as a promising for various structural materials, because of their good fracture toughness compared with monolithic SiC ceramics. But, high temperature and pressure lead to the degradation of the reinforcing fiber during the hot pressing. Therefore, reduction of sintering temperature and pressure is key requirements for the fabrication of SiC_f/SiC composites by hot pressing method.

In the present work, monolithic Liquid Phase Sintered SiC (LPS-SiC) was fabricated by hot pressing method in vacuum atmosphere at 1800°C under 20MPa using Al₂O₃, Y₂O₃ and SiO₂ as sintering additives in order to low sintering temperature and sintering pressure. The composition of Al₂O₃ and Y₂O₃ was fixed, and the compositions of SiO₂ were differently applied in order to investigate influence of SiO₂ additives. The starting powder was high purity β-SiC nano-powder with an average particle size of 30nm. The characterization of LPS-SiC was investigated by means of SEM and three point bending test. Base on the composition of sintering additives-microstructure-mechanical property correlation, the compositions of sintering additives are discussed. Sintered density, flexural strength and elastic modulus of fabricated LPS-SiC increased with increasing the sintering temperature. Particularly, relative density of sintered body at 1800°C with the less content of SiO₂, specimen of AYSa-1800 was 95%. Also, flexural strength and elastic modulus were 587.5MPa and 216.3GPa, respectively. And Vickers hardness and fracture toughness were 2500 and 2MPa·m^{0.5}, respectively. It is superior values than that of LPS-SiC with Al₂O₃-Y₂O₃ system.

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12-95

A comprising steady-state creep model for the austenitic AISI 316 L(N) steel

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Among many other applications the 17Cr12Ni2Mo steel 316 L(N) is envisaged for ITER applications. Since creep data allowing statements to be made about the stress dependence of steady-state creep rate had been almost unavailable, a special long-term creep testing program at 550 °C and 600 °C was started in 1991. After an experimental period of about 10 years the creep tests have been either finished or aborted, and evaluated. Now this low-stress creep data not only allow for a much better long-term prediction of the reliability of 316 L(N) applications but also enable deformation modeling for a broader stress range.

The present work focuses mainly on the set-up of a steady-state creep model with help of rate-equations well known for different deformation mechanisms, such as diffusional flow, dislocation climb, and dislocation glide. In addition, the impact of microstructure and precipitation formation on steady-state creep is outlined and discussed. The resulting creep model consists of a summation of contributions for diffusion creep, power-law creep, and power-law breakdown (transition to pure dislocation glide). Most model parameters are either known material constants or could be directly deduced from the available data while a few free parameters had to be adjusted to the experiments. As a result, the creep model agrees well with experimental data for temperatures between 550 °C and 750 °C and for shear stresses down to 30 MPa. For very low stresses the model predicts far higher creep rates as usually extrapolated from tests performed at the medium stress range.

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12-96

Tensile Properties and Electrical Conductivity of Unirradiated and Irradiated Cu-Ni-Be

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High-strength, high-conductivity copper alloys are being considered for first wall heat sink, blanket, and divertor structural applications in fusion energy systems such as the International Thermonuclear Experimental Reactor (ITER). A particularly challenging application is the compression collar in the ITER blanket attachment, which requires high conductivity and the ability to withstand very high compressive loads. This demanding application produces stresses that are beyond the design stress limits of Cu-Cr-Zr or oxide dispersion strengthened copper. One potential candidate material for the compression collar is a high-strength, conductivity Cu-2%Ni-0.35%Be alloy.

The unirradiated tensile properties of two different heats of Brush-Wellman Hycon 3HP™ Cu-Ni-Be were measured over the temperature range of 20-500 °C for longitudinal and long transverse orientations. The room temperature electrical conductivity was also measured for both heats. Both heats exhibited a very good combination of strength and conductivity at room temperature. The strength remained relatively high at all test temperatures, with a yield strength of 420-520 MPa at 500 °C. However, low levels of ductility (<5% uniform elongation) were observed at test temperatures above 200-250 °C, due to flow localization adjacent to grain boundaries. Fission neutron irradiation to a dose of ~0.7 displacements per atom (dpa) at temperatures between 100 and 240 °C produced a slight increase in strength and a significant decrease in ductility. The measured tensile elongation increased with increasing irradiation temperature, with a uniform elongation of ~3.3% observed at 240 °C. The electrical conductivity decreased slightly following irradiation, due to the presence of defect clusters and Ni, Zn, Co transmutation products. The data indicate that Cu-Ni-Be alloys have irradiated tensile and electrical properties comparable or superior to Cu-Cr-Zr and oxide dispersion strengthened copper at temperatures <250 °C, and may be suitable for certain fusion energy structural applications.

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12-97

Influence of Mechanical Stresses on Radiation Swelling of an Austenitic Stainless Steel

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The influence of mechanical stresses on radiation swelling of the Russian austenitic stainless steel X16H15M2Г2Т (Fe-16Cr-15Ni-2Mo-2Mn-Ti) was studied. Gas-pressurized tubes constructed from this steel were irradiated at 740 K in the BN-600 fast breeder power reactor, under hoop stresses of 0, 100 and 200 MPa. This study is unique in that a very high dpa level was reached, being 108 dpa at a very high dpa rate. Swelling was measured and porosity characteristics were determined using the methods of hydrostatic weighing and electron microscopy.

It was established that swelling increased with increasing stress. Quantitative analysis of the cavity size distribution under stress of 200 MPa shows that the cavity concentration increased and the cavity spatial distribution became more homogeneous.

Analysis of the possible mechanisms of swelling rate enhancement under stress for these data allowed us to reveal that the dominating mechanism of the influence of stress on swelling in this austenitic steel is associated with the influence of stress on nucleation and growth of vacancy clusters. The implications of these findings for fusion application are discussed.

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12-98

The Synergistic Influence of Irradiation Temperature and Atomic Displacement Rate on Microstructural Evolution of Ion-Irradiated Model Austenitic Alloy Fe-15Cr-16Ni

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The dependence of void swelling on material and environmental variables is a concern for both fission and fusion reactors, with strong recent interest on the effects of dpa rate. A comprehensive experimental investigation of microstructural evolution has been conducted on Fe-15Cr-16Ni irradiated with 4.0 MeV nickel ions in the High Fluence Irradiation Facility of the University of Tokyo. Irradiations proceeded to dose levels ranging from ~0.2 to ~26 dpa at temperatures of 300, 400, 500, 600 and 700°C at displacement rates of 1×10^{-4} , 4×10^{-4} and 1×10^{-3} dpa/sec. This experiment is one of two companion experiments directed toward the study of the dependence of void swelling on displacement rate. The other already-published neutron irradiation experiment proceeded at seven different but substantially lower dpa rates in FFTF-MOTA at ~400°C. In both experiments the swelling was found at every irradiation condition studied to monotonically increase with decreases in dpa rate.

The microstructural evolution under ion irradiation was found to be very sensitive to the displacement rate at all temperatures examined. The earliest and most sensitive component of microstructure to both temperature and especially displacement rate was found to be the Frank loops. The second most sensitive component was found to be the void microstructure, which co-evolves with the loop and dislocation microstructure. At the higher dpa rates employed in the ion irradiation series it was possible to nucleate voids but they never grew quickly in the loop-dominated dislocation structure, while at the lower neutron-induced dpa rates the loops quickly unfaulted to form a dislocation network and voids which then accelerate in growth rate. The significance of these results is that the flux effect appears to be completely independent of irradiation temperature, in disagreement with most theoretical models which predict a shift in the swelling peak with increasing dpa rate, but with a reversal in the sign of the flux effect on the high side of the swelling peak. Another consequence of these results is that ion irradiation of simple model alloys will usually produce less swelling than neutron irradiation, primarily due to the higher dpa rates usually employed compared to that of neutron irradiations.

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12-99

Effect Of Irradiation Dose On Mechanical Properties And Fracture Character Of Cu//SS Joints For ITER

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Cu//SS type joints are basic for the heat-sink systems of the ITER high-heat flux components. By now, a number of technologies have been proposed for the production of such bimetallic structures, such as brazing, friction welding, HIP and cast-copper-to-steel. The last two mentioned technologies ensure sufficiently high mechanical properties and a high joint quality, when unirradiated. However, data on the irradiation resistance of these joints are scarce.

In this paper, the authors present the results of investigations into the irradiation resistance of GlidCopAl25/316L(N) and Cu-Cr-Zr/316L(N)-type joints produced by the HIP and cast-copper-to-steel (CC) technologies. Specimens of the joints were irradiated in the RBT-6 reactor in the dose range of 10^{-3} – 10^{-1} dpa at $T_{irr} = 150^\circ\text{C}$.

A significant growth of grains up to 300 μm (as compared to 50 μm in the optimized condition) was observed in the Cu-Cr-Zr alloy after the HIP procedure. The growth was even more significant in the copper part of the Cu-Cr-Zr/316L(N) joint produced by the cast-copper-to-steel technology. In joints of the GlidCopAl25/316L(N) HIP type no grain growth was observed.

The tensile stress-strain curves for irradiated and unirradiated joint specimens show deformation processes occurring in both the Cu and SS parts of the specimens.

Irradiation causes strengthening of the joints specimens (by about 150 MPa at the maximum dose), fracture occurs near the joint line. The uniform elongation drops from 7% in the initial state to 1-2 %. But the total elongation remains at the relatively high level of ~10%. SEM investigation revealed that fracture occurs only in the copper part of the specimens, and the ductile transcrystalline fracture predominates in the joints.

3D simulation indicates that the concentration of stresses and deformations in the copper layer adjacent to the joint line is responsible for this typical failure of the irradiated joints specimens.

The investigations performed make it possible to recommend joints of Cu-Cr-Zr//316L(N) (CC) and Cu-Cr-Zr//316L(N) (HIP) type produced by the cast-copper-to-steel and HIP technologies, respectively, for ITER applications. The radiation resistance of Cu-Cr-Zr//316L(N)-type joints specimens is similar to that of the base copper alloy.

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Novel Composite Heat Sink Material for the Divertor of future Fusion Reactors

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The use of CuCrZr as heat sink material limits the operation temperature of the divertor in fusion reactors to 350°C. For efficient energy production a higher operation temperature up to 550°C is required. A novel material has to be developed with a thermal conductivity of at least 200 Wm⁻¹K⁻¹ and sufficient strength under neutron irradiation.

We investigate SiC fibre reinforced copper as an additional layer at the highly loaded zone between plasma facing material (W) and heat sink (CuCrZr) of the divertor. Copper has a high thermal conductivity of 380 Wm⁻¹K⁻¹ but the strength and creep resistance at 550°C is very low. Therefore copper is reinforced with SiC long fibres (SCS6, Specialty Materials) having excellent high temperature strength.

The fibres were galvanically coated with an 80-µm-thick copper layer as matrix. Hot isostatic pressing at 650°C was applied to form the composite material. With push-out tests the interface between fibres and matrix was characterised. A

flat-ended punch of 100 µm in diameter pushed single fibres out of the matrix. The calculated interfacial shear strength was approximately 6 MPa indicating a very weak bonding between fibres and matrix.

An additional 100-nm-thin titanium interlayer deposited by magnetron sputtering led to a higher adhesion of the SiC fibres in the copper matrix. Titanium reacted with the carbon surface of the fibre to TiC and formed with copper the alloy Cu₄Ti during heat treatment. With this chemical bonding the interfacial shear strength increased one order of magnitude to 70 MPa.

As a next step the thermo-mechanical fatigue (TMF) behaviour of the material will be investigated experimentally under near service cycle loading conditions. During the discontinuous plasma burning in tokamak type fusion reactors the plasma facing component as well as the composite material itself is exposed to complex thermo-mechanical load cycles. Macroscopic thermal stresses are caused by thermal gradients and by thermal mismatch between plasma facing material (W) and heat sink (CuCrZr). In microscopic scale TMF-cycles result from the different coefficients of thermal expansion between SiC fibre and Cu matrix.

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12-101

Investigation of CuCrZr Alloys using Positron Annihilation Spectroscopy

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The influence of heat treatment (HT) on precipitate sizes and densities in CuCrZr (Outokumpu) alloy has previously been investigated by transmission electron microscopy (TEM). In the present work we investigate the effect of HT on the defect structure in non-irradiated CuCrZr as well as in the alloy neutron irradiated after HT, using positron annihilation spectroscopy (PAS). Results obtained by PAS were compared with the results achieved by TEM. In the PAS measurements of non-irradiated alloys a well-defined long defect lifetime τ_2 was observed with basically constant value 176 ± 8 ps, but with an intensity I_2 that exhibited strong dependency on the HT. Thus, a clear influence of the applied HT on the defect microstructure of the CuCrZr alloy was observed. The measured defect lifetime τ_2 is associated with the creation of the vacancy-type defects at the precipitate matrix interface, believed to arise from lattice mismatch between matrix and precipitates. The PAS measurements on non-irradiated material were used as a starting point for the following measurements on neutron irradiated CuCrZr samples. The irradiations by fission neutrons were performed in research reactors BR-2 Mol, Belgium and Budapest, Hungary. On the prime aged (PA) sample, the dose dependence was investigated at two different irradiation temperatures T_{irr} , i.e. 50°C (10^{-3} , 10^{-2} , 10^{-1} dpa) and 350°C (0.1, 0.2, 0.3 dpa). Also PA samples, neutron irradiated to a dose of 3×10^{-1} dpa at the irradiation temperatures 50°C , 150°C , 200°C , 300°C and 350°C , were investigated. In the temperature range up to 300°C only one well-defined defect lifetime of 178 ± 1 ps was observed. At $T_{irr} = 350^\circ\text{C}$, it decreased slightly to 170 ± 1 ps. This is very similar to pure Cu, except that no voids were observed at the highest irradiation temperatures.

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12-102

Characterization of 12Cr18Ni10Ti Stainless Steel Irradiated at Low Displacement Rates in BN-350 Reactor

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Void swelling of austenitic stainless steels irradiated in fast reactors is influenced by a variety of factors, such as composition, dpa level, temperature, and dpa rate. As recent studies have shown it is the composition and especially the dpa rate that strongly determine the low temperature boundary of swelling, often at low dpa rates occurring at temperatures below those observed at higher dpa rates.

This paper presents results of investigation of a hexagonal blanket assembly designated N-214(1) that was irradiated in the BN-350 fast reactor up to a maximum dose of 12.3 dpa at low dpa rates of only $\leq 3.8 \times 10^{-8}$ dpa/sec. The assembly was constructed from austenitic stainless steel 12Cr18Ni10Ti which is the Soviet analogue AISI 321. Experimental techniques employed were metallography, immersion density, Vickers microhardness and electron microscopy. Specimens were removed from five altitudes on the assembly to reveal details of microstructure and properties at different doses in the temperature interval $281\text{--}430^\circ\text{C}$.

At a low dose/temperature combination of 0.65 dpa and 281°C this steel exhibits significant void swelling as compared with only rare single voids observed in irradiated steel 08Cr16Ni11Mo3 at 12.3 dpa and 281°C from an earlier study on another assembly N-214(2) irradiated at higher dpa rates. The swelling values are higher for all altitudes compared to assembly N-214(2), although the dpa doses and dpa rates were everywhere smaller. These results support the assumption that the lower temperature boundary of swelling for 12Cr18Ni10Ti steel in this assembly is caused not only by its composition but also by the lower dpa rates involved.

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12-103

Swelling and Microstructure of Cold-worked Austenitic Stainless Steel ChS-68 after High Dose Neutron Irradiation

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Due to a unique set of physical-mechanical properties chromium-nickel stainless steels are widely used in the nuclear industry. Based on their performance in fast reactors these steels are also being considered for use in fusion reactors.

The austenitic steel ChS-68 (0.06C-16Cr-15Ni-2Mo-2Mn-Ti-V-B) was developed in SSC RF A.A. Bochvar All-Russia Research Institute of Inorganic Materials (VNIINM) as an alternative to the austenitic steel EI-847 (0.06-16Cr-15Ni-3Mo-Nb) used as a pin cladding structural material in Russian fast reactors. Use of ChS-68 in the cold-worked condition for pin cladding in the BN-600 fast reactor has allowed an increase of fuel burn-up from 9 up to 12%., with maximum dose for pin cladding increased from 70 up to 94 dpa.

Prior to use of modified versions of this type of steel for higher exposure in either fission or fusion environments, it is necessary to understand the mechanisms by which the microstructure is altered by irradiation at reactor-relevant temperatures. In this paper the results of microstructural investigation of ChS-68 CW pin cladding irradiated to a maximum dose of 84 dpa in the BN-600 fast reactor are presented.

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12-104

Hold-Time Effects on the Fatigue Life of Copper and Copper Alloys for Fusion Applications

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Copper and its alloys are of prime interest for high heat flux applications in near-term fusion systems such as ITER. These systems must operate in a pulsed mode to allow for plasma heating and compression and subsequent refueling. This means that the component temperatures and mechanical loading conditions will fluctuate making fatigue a major materials failure mode. The first wall materials, for which copper alloys are the prime candidate, will be most affected by this process. Several studies of the fatigue response of copper and copper alloys indicate that they have marginal, but acceptable fatigue endurance properties under continuous cycling conditions. However, the most recent studies of the fatigue performance which include hold time effects indicate that the hold process can substantially degrade the material fatigue life. This reduction in fatigue life is found to be most dramatic in the long life, high cycle fatigue regime where fatigue lives can be reduced by a factor of more than three. It is also found that the reduction in fatigue life requires only a short hold period of 10s, much shorter than the anticipated on/off cycles in ITER of 1000s/100s.

Current experiments on OFHC Cu and CuCrZr show that the mode of fatigue crack initiation changes from one dominated by persistent slip band (PSB) formation and linking in continuously cycled systems to one where grain boundary cracking is more predominate in hold-time and longer life tests. An analysis of the fatigue loading performance with hold times also indicates that copper and its alloys show a large, up to 15%, relaxation in stress during the hold period at constant applied strain at room temperature. This temperature, 0.22 T_m, is well below temperatures typically associated with thermally-induced creep and stress relaxation. This paper examines the underlying microstructural aspects that control both the stress relaxation process and the changes in fatigue crack initiation and early growth. This information is critical for assessing the impact of cyclic loading with extended hold periods on fatigue failure modes.

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12-105

**Microstructure of Austenitic Stainless Steel
EC 316LN Irradiated in SINQ Target-4 up
to 20 dpa and 1700 appm He at $\leq 430^\circ\text{C}$** *X. Jia¹, M. Grosse¹, and Y. Dai¹*

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Austenitic stainless steel AISI 316L has been used widely in heavy irradiation areas of various fusion reactor research facilities and will be the material for the first wall of ITER. It has been also applied in a number of spallation targets. The helium-to-dpa ratio is relatively high in fusion cases, about 10 appm He/dpa, while in spallation cases is much higher, about 100 appm He/dpa. Helium induced embrittlement effects in AISI 316L were intensively studied in the last two decades but mostly at temperatures above 450°C. The knowledge about helium effects in austenitic steels at lower temperatures, strongly needed for ITER and spallation targets as well, is still very limited. In the present work, the

microstructure of solution annealed EC 316LN after irradiated in SINQ Target-4 up to 20 dpa and 1800 appm He at $\leq 430^\circ\text{C}$ has been investigated with transmission electron microscopy (TEM) and small angle neutron scattering (SANS) techniques. The TEM results indicate that high-density small defect clusters and large frank loops were introduced by the irradiation. High-density tiny helium bubbles with a mean size of about 1 nm were observed in the sample irradiated at about 430°C to 19.6 dpa and no large voids were detected. The SANS analysis confirms essentially the TEM results. The results will be compared with our previous observations on the same material irradiated at low doses in SINQ Target-3 and those of AISI 304 irradiated with 750 MeV protons, and with those martensitic steels from the same irradiation as well.

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12-106

The Influence of Cold-work Level on the Irradiation Creep of AISI 316 Stainless Steel Irradiated as Pressurized Tubes in the EBR-II Fast Reactor

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Development of dimensional change correlations for both fission and fusion service requires either a very large amount of relevant data covering all potential material and environmental conditions, or application of derived insight on broadly-applicable principles to a more limited amount of data. Previous studies have shown that irradiation creep is much less directly responsive to material and environmental variables, other than being directly dependent on the stress level, with most of its sensitivities at higher exposure levels arising directly and proportionately from the sensitivities of void swelling.

To better define the sensitivities of irradiation creep before the onset of swelling and its relationship to void swelling after swelling commences, previously unanalyzed and unpublished data are being examined to test the generality of earlier proposed relationships. The current effort focus is on the dependence of irradiation creep on temperature, stress level and cold-work level.

In an earlier study on titanium-modified 316 stainless steel irradiated in FFTF it was shown that at relatively low irradiation temperatures (400-500°C) irradiation creep was linear with stress and relatively independent of cold-work level.

In order to test the generality of these conclusions unpublished experiments conducted in the EBR-II fast reactor on pressurized tubes of AISI 316 stainless steel have been analyzed. The EBR-II tubes have different advantages and disadvantages compared to the FFTF tubes, allowing confirmation of assumptions employed and conclusions reached in the FFTF study. It was observed that while the onset of swelling is dependent on the cold-work level, the irradiation creep is not dependent on cold-work but is nevertheless strongly dependent on the swelling. It was also shown that the creep rate is linearly proportional to the stress and that stress enhances swelling. Stress levels that exceed the yield stress upon reaching full temperature cause the tube to acquire permanent diametral deformation but the swelling-creep relationship is retained thereafter. Swelling strains were shown to be isotropically distributed, although precipitation-induced strains are not necessarily distributed isotropically, especially for cold-worked steel.

Based on the FFTF and EBR-II studies, as well as other sources, it appears safe to assume that the creep compliance component of irradiation creep can be considered to be linear with stress, independent of irradiation temperature and relatively independent of cold-work level, especially for lower temperature applications such as those anticipated for ITER.

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Plenary Session 14 – Integration of Experiments and Multiscale Models to Solve the Grand Challenges of Fusion Materials and the Materials Design Interface

14.1

Integration of Experiments and Modeling in Fusion Materials Research

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The role of modeling and computer simulation in materials science and engineering is getting more and more important supported partly by the rapid advance of computer power. Research on fusion reactor materials is no exception. Understanding of elemental processes not accessible by other means has extended dramatically in recent years by computer simulation. Designing fusion demo reactors requires a large material database corresponding to each component material in the reactor exposed to a variety of environment. In order to construct such material database, a huge test matrix is required and it will be an impossible task under a reasonable resource and within a reasonable time frame. In order to fill the gap not covered by the matrix, quantitative prediction based on theory and modeling is essential. In this paper, importance of combining experiments and modeling will be emphasized. Dislocation-obstacle interaction and 1-dimensional motion of defect clusters are major topics in this paper. Both HVEM *in situ* experiments and MD simulation results will be presented.

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14.2

Bridges between Materials and Design

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The strong relation between conceptual development and detailed design of in-vessel components on the one hand and materials choice and development on the other hand is addressed. The development of Helium cooled breeder blankets or divertor concepts will serve as examples. From the various aspects of “material design interface” three will be discussed in detail.

Firstly, developing new “design concepts” implies specific requirements on materials with respect to operating temperature window and physical and mechanical properties. Materials need to be pre-selected and further optimized. The development of tungsten alloys for Helium cooled divertors will be used as an example. Other limitations include technological issues such as joints or coatings. Vice versa, considering, secondly, a “well developed and well characterized material”, the challenge for the designer is to develop an optimum process or equipment. Early feedback and close cooperation between engineers and material scientists is needed to avoid delays of a project. This will be exemplified by the use of RAFM steels like F82H or EUROFER for TBMs (Test Blanket Modules).

Thirdly, and as the essential part, the application and the development of “design criteria” will be outlined. The introduction of new ideas and new rules into existing code frameworks is multifunctional: (i) it might be mandatory to guarantee safe operation and for the protection of capital investment, (ii) it is an additional source to improve performance in an efficient and economic way. Design for accidental conditions with ITER TBMs requires the application of the “plastic path” of the ITER structural design criteria (SDC). Breeder blankets are operated cyclically in the “high temperature regime”. Therefore, fatigue-creep interaction is a severe concern. The introduction of “visco-plastic paths” might help to solve some issues. To develop new design rules might sound tedious, but is a challenge to experts in the materials community. In essence, it needs knowledge and deep understanding of material mechanical behavior and microstructure to identify failure modes, cause and progress of damage. Finally, any approach to develop advanced design methodologies needs sophisticated modeling, from “micro” to “macro” scale, and experimental verification and validation. As most prominent examples the challenge to include fracture mechanical rules in the ITER SDC will be discussed.

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Parallel Oral Session 15A – Materials – Design Interface

15A.1

Challenges of Structures and Materials Research in the Aerospace Industry

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The aerospace industry continues to evolve as systems and platforms grow in size and complexity. Air and space is no longer the sole environment as network centric systems pervade ground, sea, underwater, and cyberspace domains. In this expanded vision of aerospace, materials science continues to be an enabler of systems and platforms but research scientists and engineers must adapt to changing conditions within industry. Most notably is the influence of systems engineering in the design and decision making process. Second is the need to develop integrated solutions that present broad capabilities rather than achieve specific missions. And finally, emerging technologies such as nano engineered materials, structurally adaptive materials, and integrated smart materials themselves pose significant technical hurdles in fabrication, scale up, modeling and analysis

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15A.2

In-Pile Performance of Pebble Bed Assemblies and Implications for the HCPB Blanket Concept

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Developing the European Helium Cooled Pebble-Bed (HCPB) blanket requires a neutron irradiation programme to assess the material behaviour at relevant temperature levels and thermal-mechanical loads. Studying the irradiation performance of the breeding blanket materials such as Eurofer, lithium ceramics and beryllium separately, prevented to learn about their interaction in a component. To investigate the effect of neutron irradiation on the thermal-mechanical behaviour of the ceramic pebble-bed, a number of sub-assemblies representative for the HCPP concept have been irradiated in the HFR Petten. The assemblies consist of a ceramic breeder bed sandwiched between beryllium pebble beds separated by Eurofer floating plates. The irradiation assembly contained four of such stacks with different breeder materials and temperatures to enhance the design basis for the DEMO breeder concept.

For the design and interpretation of this complex irradiation experiment an advanced thermo-mechanical model has been developed. The model includes the non-linear elasticity, compaction, and creep compaction of the ceramic and beryllium pebble beds. The strong effect of the Beryllium bed compaction on thermal conductivity in the blanket affects the model predictions considerably. The results of these calculations are critical for a safety assessment of the in-pile operation of the experiment and provide a better understanding of the in-pile behaviour. At the same time the blanket and TBM designs will need such evaluation tool.

This paper reports the in-pile behaviour of the HCPB Pebble Bed Assembly, during 300 Full Power Days. The damage doses accumulated for the Eurofer structure range up to 3 dpa. In addition the detailed thermo-mechanical analyses using the model will be presented and compared with the in-pile experimental results. The strategy for post-irradiation examination that has been developed from the experimental and model results will be discussed. The impact of the results on HCPB blanket and TBM designs will be addressed. Besides materials performance and interaction issues, useful insight is obtained for the design of TBM instrumentation.

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15A.3

Large-Scale Finite Element Modeling of the Thermo-Mechanical Behavior of the Dual Coolant US-ITER TBM Incorporating Damage Evolution

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Within the framework of VISTA (Virtual International Structural Test Assembly), we analyze normal and off-normal thermo-mechanical performance of the U.S. Dual Coolant Lead Lithium (DCLL) ITER Test Blanket Module (TBM) by carrying out simulation studies to evaluate a range of potential failure paths. VISTA's unique approach lies in modeling the effects of thermal and mechanical loads on failure modes of irradiated structural materials in which damage evolves as a function of operational time. First damage functions are developed to reflect property degradation as a result of neutron irradiation and aging. These damage functions are then incorporated into large-scale finite element models of full 3-dimensional geometry. We develop material models that describe rate-

dependent plasticity, thermal and irradiation creep, volumetric swelling, and fracture toughness of F82H. The developed hierarchical constitutive laws are based on multi-scale integration of materials theory, models, simulations and experiment. The response of the U.S. DCLL ITER-TBM is evaluated using the developed damage functions for F82H. Detailed coupled thermal-mechanical response of a multi-pass 8 MPa pressure helium coolant through the First Wall, internal support structures and the back plate is simulated to assess the effects of 3-D geometric features on the structural performance of the TBM. Off-normal operation is analyzed by simulating high pressure helium coolant leak into the DCLL TBM. It is determined that the internal coolant channels of the FW results in high stress concentrations along the joints between the FW and internal support structures. Optimization studies are performed to alleviate the effects of stress concentration on failure modes.

Keywords: damage functions, VISTA, constitutive equations, failure path, true stress-strain, F82H.

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15A.4

Materials and Design Interface of In-Vessel Components for Fusion Reactors

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The paper presents interactions between materials and design development for in-vessel components of fusion reactors based on experiences of R&Ds on the divertor plates for ITER and for DEMO plants in JAEA (formerly JAERI). To satisfy design requirements, it has been developed new armor materials and joining techniques as well as correlations, such as a fatigue lifetime evaluation method.

The ITER divertor mockups have been developed and tested in high heat flux test facilities in JAEA. The divertor structure consists of a cooling structure made of copper alloy and of armor tiles made of carbon-based materials or tungsten. These materials are metallurgically bonded together. In R&D activities of the ITER divertor, multi-dimensional (1D, 2D and 3D) carbon fiber composite materials with high thermal conductivity of 400 - 600 W/m/K and a silver-free braze technique were developed to satisfy the ITER design

requirement. To evaluate the durability of the divertor structure, thermal fatigue experiments of the divertor mockups have been carried out in our high heat flux test facilities. As a result, it was found that the thermal fatigue lifetime of the ITER divertor structure was mainly limited by the stress/strain concentration of the copper cooling tube rather than the separation of the bonded interface. In addition, a numerical simulation based on a finite element method has also been made to establish a fatigue lifetime evaluation method for the divertor structure, and could successfully predict the thermal fatigue lifetime of the divertor mockups.

For DEMO divertor design in JAEA, a reduced-activation-ferritic-martensitic (RAFM) steel, F82H, is a primary candidate structural material from a viewpoint of heavy neutron irradiation in the DEMO plant. High heat flux experiments of divertor mockups have energetically been performed to clarify the durability and the thermo-mechanical performance of the DEMO divertor structure made of F82H. Based on the lifetime evaluation method described above, correlations for thermal fatigue lifetime of the F82H divertor structure has been being developed in JAEA.

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Parallel Oral Session 15B – Blanket Engineering - II

15B.1

Correlation Between Tritium Release and Thermal Annealing of Irradiation Damages in Neutron-Irradiated Li_2SiO_3

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In blanket systems for D-T fusion reactors, energetic tritium is produced in the tritium breeding materials. For establishment of the tritium recovery system, it is a critical issue to elucidate the chemical behavior of tritium produced in the materials, such as its existing states and release behavior. In our previous studies, we have suggested that the thermal annealing processes of the irradiation damages played an important role in the tritium release processes of lithium-bearing ceramics, such as Li_2O , Li_4SiO_4 , and LiAlO_2 .

In the present study, we investigated correlation between tritium release and thermal annealing behaviors for neutron-irradiated Li_2SiO_3 . The sample was irradiated with the thermal neutrons in the research reactor of Kyoto University. Annihilation processes of the irradiation damages were studied by the ESR (Electron Spin Resonance) method. In the annealing experiments, the Li_2SiO_3 sample was heated stepwise up from R.T. to 648 K at intervals of 25 K for 5 min. Just after each heating treatment, the sample was immediately cooled with liquid nitrogen and then ESR measurement was performed.

Extrinsic ESR peaks resulting from the irradiation damages were observed for the neutron-irradiated sample. This suggested that various irradiation damages were produced in the sample by neutron irradiation. The peak intensities began to decrease from 448 K and finally disappeared around 573 K. From the ESR spectra of sample annealed at each temperature, E' -center, which is expected to correlate with tritium release, was supposed to exist from the viewpoint of the previous studies. In the presentation, the annealing processes of E' -center and its correlation with tritium release behavior will be discussed in detail.

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15B.2

A SIMS Study of the Distribution of Tritium in Neutron-Irradiated Beryllium Pebbles

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A key issue of beryllium as a neutron multiplier in the blanket of future fusion reactors is tritium retention. In order to develop reliable models to predict tritium kinetics in beryllium in the typical operating conditions of the material in the blanket, a detailed experimental characterization of microscopic diffusion phenomena is necessary.

A study of tritium spatial distribution in neutron irradiated beryllium pebbles from the BERYLLIUM irradiation (2 mm diameter, 480 atoms of ^4He and 7 of ^3H per million atoms of beryllium) by means of Secondary Ion Mass Spectrometry (SIMS) is presented. Samples in different conditions (non-irradiated, at end of irradiation and after thermal ramp annealing up to different temperatures) are examined. By mapping and line scanning performed on a planar section of the irradiated pebble by means of a Cs^+ primary ion beam with a spatial resolution of 1 micron, tritium is detected both in atomic and molecular form ($^3\text{H}_2$): atomic tritium is dissolved in the lattice, whilst molecular tritium is contained in helium bubbles or small clusters in the vicinity of dislocations. The concentration of both forms is decreasing in the periphery of the pebble; in the central part molecular tritium is uniformly distributed, whilst the concentration of atomic tritium decreases periodically in the vicinity of grain boundaries. Tritium peaks are also observed correspondingly to large grain boundary bubbles. SIMS results are in agreement with Transmission Electron Microscopy observations of bubble formation in the same material and confirm recent improvements of the theory of tritium diffusion and precipitation into bubbles.

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15B.3

An Overview of Recent Progress in Studying Redox Control in FLiBe Using Dissolved Beryllium

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Formation of TF due to neutron irradiation of FLiBe is a problem in that it is expected to lead to rapid corrosion of structural materials in a FLiBe-based blanket system. Beryllium has been suggested as a redox agent based on the thermodynamics of it reacting with HF--the reaction of beryllium with HF to form beryllium fluoride and hydrogen gas is spontaneous at the temperatures of

interest. Unlike when using other redox agents such as cerium, accumulation of excess beryllium fluoride in the salt is not a problem, since it is one of the major constituents of FLiBe. Over the last couple of years, an extensive experimental program has been undertaken at the STAR facility of the Idaho National Laboratory within the JUPITER-II joint Japan-US project with the mission to study the feasibility of beryllium redox in FLiBe. Using multiple experimental methods which will be described, it has been discovered that beryllium dissolves rapidly and to a high extent in molten FLiBe. Additionally, it has been found that this dissolved beryllium reacts readily with HF that is bubbled into the salt. Kinetic data collected over a range of flow rates and concentrations will be presented. A comparison between the data and various versions of a kinetic model will be presented as well. When experimental results are compared with kinetic models, both mechanistic issues and reaction rates can be elucidated. The strategy for using atypical reaction conditions for these tests to predict redox behavior in typical fusion system conditions will be explained.

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15B.4

Swelling, Mechanical Properties and Microstructure of Beryllium Irradiated at 200°C up to Extremely High Neutron Doses

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At present beryllium is considered as main neutron multiplier material for the DEMO fusion reactor blanket. In the fusion reactor beryllium will be placed in a high energy neutrons field and exposed to significant radiation damage. Therefore since the 14 MeV high flux neutrons source was not built for the present it is actually continued to investigate of the beryllium state after irradiation in a nuclear reactor under maximal fast neutrons doses. Analysis of such experimental results gives some possibility for prediction of beryllium behavior in fusion conditions.

In the work there are presented the results of investigation of neutron irradiation influence in the SM reactor at 200°C up to doses of $(0.7-13) \cdot 10^{22} \text{ cm}^{-2}$ ($E > 0.1 \text{ MeV}$) on swelling, mechanical properties (tensile and compression tests,

microhardness) and microstructure of four Russian beryllium grades (TE-56, TE-30, TIP, DIP) manufactured by hot extrusion (HE) and by hot isostatic pressing (HIP).

Dose dependencies of swelling and mechanical properties of irradiated beryllium are presented. Monotonic increase of swelling with growth of neutron dose takes place. Swelling of beryllium at maximal investigated neutron doses is not more than 4 %. Mechanical tensile and compression tests lead to absolutely brittle destruction of all irradiated specimens. Decrease of strength of irradiated beryllium in comparison with initial state takes place however stabilization of its level occurs at maximal investigated neutron doses. Microhardness of irradiated beryllium monotonically increases with growth of neutron dose. It is noted that dependence of swelling and mechanical properties radiation changes of beryllium grades from manufacture technology is not meaning.

Voids which are result of manufacture technology are in initial beryllium microstructure. Under neutron irradiation accumulation of gas atoms (helium, tritium) in beryllium occurs that leads to evolution of this voids, in particular increase of diameter and volume density of voids takes place. At the same time formation of gas atoms clusters which gradually evaluate to very small gas filled bubbles occurs. Superposition of this processes in microstructure determines as a result swelling and radiation mechanical properties changes of beryllium.

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15B.5

Recent Results on Beryllium and Beryllides in Japan

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Recently, several R&D programs of beryllium for fusion are being promoted in Japan and the community of beryllium study is growing up. In the R&D area of beryllium for solid breeding blanket, major subjects are beryllide application, lifetime evaluation of neutron multiplier, impurity effect in beryllium on irradiation behavior, recycling of irradiated beryllium and so on. As for neutron multiplier materials, recent R&D has been focused on Be alloys, in particular Be-Ti alloys as an advanced option, because of better properties, such as compatibility with structural materials and oxidation resistance at higher temperature. Therefore, recent results on beryllide R&D will be mainly presented in this conference.

Phase diagrams of binary system of Be-Ti and Be-V were experimentally established to understand basic metallurgical behavior for manufacturing technology development. The basic properties such as physical, chemical and mechanical properties for stoichiometric Be₁₂Ti fabricated by HIP and Be-Ti alloy with α Be phase fabricated by an arc melting process have been studied and some advantages against beryllium were made clear. Especially, both oxidation and steam interaction were about 1/1000 smaller than those of beryllium. These results suggested us the possibility to realize mixed packing with tritium breeder, to reduce a risk of LOCA, and to realize high efficient blanket. As to the compatibility between Be-Ti alloys and F82H, the growth rate of the reaction layer for the Be-Ti alloys decreased with increasing the Ti content in the Be-Ti alloy. Furthermore, Be-Ti alloys were irradiated with various accelerators and testing reactors. Then tritium inventory and irradiation effects have been evaluated.

In order to enhance the R&D activities, the R&D network consisted of industries, universities and laboratories in all Japan has been organized. Many collaboration and information exchange strongly promote the R&D and some projects for commercial application have been launched from these activities.

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Parallel Oral Session 16A – Multiscale Modeling of Radiation Effects

16A.1

Computational Modeling of Material Failure

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Some recent approaches to the computational modeling of material failure will be discussed. One approach involves incorporating a model of the failure process into the material constitutive relation so that, for a suitable imposed deformation history, the stress carrying capacity of the material vanishes and new free surface is created. In a contrasting approach, the deformation and fracture properties of the material are specified separately. In particular, the deformation properties of the material are embodied in a volumetric constitutive relation while the fracture properties are specified in a surface constitutive relation. Regardless of the formulation, predictions of fracture require a length scale to enter the analysis, if only from dimensional considerations, and there are various ways physically relevant length scales can be included in each of these formulations. Application of one or both of these approaches to various fracture phenomena will be illustrated including: the ductile-brittle transition; specimen size effects; and effects of material microstructure on the predicted fracture behavior. The strengths and weaknesses of various modeling approaches will be discussed.

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16A.2

An Atomic-Scale-Simulation Study of Hardening Due to Copper Precipitates in Iron

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Formation of nm-scale copper-rich precipitates is a common radiation effect in reactor structural ferritic steels. Due to their small size and uncertain structure and composition, the properties of these precipitates and their interaction with other crystal defects are difficult to describe. The interaction of dislocations with copper precipitates is particularly important because it is the basis of hardening effects, yet the details of the strengthening are mostly unknown because of the atomic-scale nature of the mechanisms involved. This paper presents results of extensive atomic-scale computer simulation of interactions between copper precipitates and dislocations in iron. We consider both edge and screw dislocations with Burgers vector $\frac{1}{2}\langle 111 \rangle$ gliding under applied stress and interacting with precipitates with size up to 6nm. Precipitates with copper concentration from 50 to 100 at.% and containing up to 5 at.% of vacancies have been modeled. The simulations have been performed for temperatures up to 600K and a range of applied strain rates. We report results for the effects of the variables on the critical obstacle stress and describe the different mechanisms revealed by the simulations. The results suggest possible explanations of some experimental data on irradiated Fe-Cu alloys.

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16A.3

Modeling of Helium Bubble Formation in BCC Metals

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High rates of insoluble helium are introduced into fusion materials during irradiation. High concentrations of helium in materials play a significant role on microstructural changes, such as the formation of helium bubbles, which enhances void swelling and produces surface roughening and intergranular embrittlement. For further development of helium-resistant materials, the nucleation and growth mechanisms of helium bubbles as well as the role of material's component on their formation should be clarified. In the present study, atomistic simulations and thermodynamical evaluation were performed to provide the energetics and formation kinetics of helium bubbles in bcc Fe.

The formation free energy of helium bubbles that we evaluated using the thermodynamics of lattice defects provided the binding energy of a point defect to a helium bubble in Fe matrix. The evaluated binding energies depended much on the helium pressures of bubbles, rather than bubble size. Both the helium and self-interstitial atom (SIA) binding energies decreased with increasing helium pressures, while vacancy binding energy showed an increasing function of pressures. These trends are still valid even for atomistic-size helium-vacancy clusters which were evaluated by our previous molecular

dynamics (MD) and molecular static (MS) simulations [1]. These relationships indicate that the helium pressure of thermally-stable helium bubbles ranges within the limited values depending on temperatures.

The helium binding energy of thermally-stable helium bubbles in bcc Fe matrix is relatively greater than that of dislocations [2] or grain boundaries [3]. Namely, helium is more strongly bound to matrix bubbles than to dislocations or grain boundaries, indicating that helium atoms can be widely dispersed over bcc matrix even at high temperature. This strong He-trapping of matrix bubbles in bcc metals could be different from the case of fcc metals.

Our MD&MS simulations also showed the stable configuration and corresponding displacement fields around helium-vacancy clusters in Fe. The displacement fields become more asymmetry for the clusters of higher pressures. From the displacement fields thus obtained and appropriately-evaluated existing probabilities of mobile point defects in the field, point defect capture radius was evaluated. The capture radius strongly depends, not only on cluster size, but also on helium bubble pressures.

References

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16A.4

Systematic Group-Specific Trends for Point Defects in BCC Transition Metals: An Ab-initio Study

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Point defects play a crucial part in the microstructural evolution of metals and alloys under neutron irradiation in a fusion or an advanced fission power plants. Recently, there has been renewed interest worldwide in structural materials with high irradiation resistance, particularly in alloys based on the bcc transition metals (TM) including vanadium, tungsten and iron.

Using density functional theory, we have carried out a systematic study of point defects in all the bcc TMs. We found that the <111> crowdion is the most stable self-interstitial atom (SIA) configuration in all the elements of groups VB and VIB of the periodic table. This is fundamentally different to the case of α -Fe, where owing to the presence of magnetism the <110> dumbbell forms the most stable SIA configuration. We also show that the formation energy of the double <111> crowdion is the lowest among di-SIAs configurations and that a stable Frenkel pair can be formed between a <110> dumbbell and a vacancy in the non-magnetic bcc TMs. These findings are at variance with the original empirical calculations by Johnson that predicted the <110> dumbbell configuration to be the lowest energy configuration in all the bcc TMs. The structure of the di-interstitial configuration predicted by density functional calculations makes it possible to resolve the controversy about the interpretation of X-ray Huang diffuse scattering in bcc-Mo. Finally, by using the nudged elastic band method, we find very small migration energies of the <111> SIAs defects in the bcc TMs of groups VB and VIB. The predicted low energy barrier for crowdion motion explains the observed group-specific trends of the low-temperature isochronal resistivity recovery curves for irradiated bcc TMs.

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16A.5

Multiscale Modeling of He Transport and Fate in Irradiated Nanostructured Ferritic Alloys

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We describe the development and application of a multiscale model of the transport and fate of He in irradiated nanostructured ferritic alloys (NFA) for fusion first-wall and blanket structural applications. Since the upper operating temperature for conventional ferritic steels is $\sim 550^\circ\text{C}$, a more economically attractive fusion power system may be feasible if NFA can be developed to improve creep strength by introducing a high particle density to impede dislocation motion and provide distributed He bubble nucleation sites and vacancy-interstitial recombination centers. Key characteristics of NFA are 1) a high density ($\sim 10^{24} \text{ m}^{-3}$) of small ($\sim 3 \text{ nm}$ diameter) Ti-Y-O clusters, 2) fine to ultra-fine crystallite grain sizes and 3) high dislocation densities. The size and number density of these features can be modified by appropriate thermo-mechanical treatments.

We employ molecular dynamics (MD) simulations to assess the binding and migration energies of He and defects with each other and at various trapping sites such as coherent precipitate interfaces, dislocation jogs and representative grain boundaries. Kinetic Lattice Monte Carlo (KLMC) simulations are used to determine migration mechanisms and diffusion coefficients of substitutional and interstitial He. KLMC is also used to model He and vacancy clustering on precipitate interfaces, on dislocation lines and in grain boundaries. The effects of radiation induced vacancies and self-interstitial atoms as well as ballistic re-solutioning are modeled in detail. The MD and KLMC simulations provide critical information for rate theory and cluster dynamics models that follow point defect and He transport and partitioning to, and recycling between, matrix cavities, precipitates, dislocations and grain boundaries. The effects of irradiation variables like the irradiation temperature, dpa dose and dose rate and He/dpa ratio are examined. Model predictions are compared to experimental observations primarily from He-implanter studies on conventional steels, NFA, and other model alloys.

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Parallel Oral Session 16B – High Heat Flux Plasma Facing Materials - II

16B.1

Development of Candidate Plasma Facing Materials for Steady State Operation of the EAST Device

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EAST is a superconducting tokamak, now under construction in ASIPP as a National project, which will operate in high power and steady state, and impose severe requirements on PFM. To meet the requirements of EAST device, one kind of doped graphite GBST1308 with SiC gradient coatings, and 2-3mm thick tungsten were chosen as candidate PFM.

In recent years, a series of doped graphite has been developed in China. Detailed investigation of the composition and full characterization of the microstructure of doped graphite were carried out and now still in progress.

These research activities mainly concentrated on multi-element (B, Si, Ti) doped graphite with SiC coatings. Investigations on the applicability of doped graphite as PFC under high heat flux, and further evaluation under HT-7 limiter plasma irradiation have been carefully investigated. These results have demonstrated that new carbon armored PFC will be an attractive choice making them competitive with other candidate PFC.

Tungsten is now under development being considered as one of the promising candidate materials for the EAST divertor plate, several joining methods have been developed, 2mm thick tungsten realized by vacuum plasma spray, brazed or directly by blast compound to copper alloy with adaptive interlayer, a modeling for residual stress analysis has been established and high heat flux experiments have also been finished, a small mock-up possess good performance, a large module is now under way. In the above experiments, the mock-ups were examined with respect to the temperature distribution, changes of surface morphology, surface atomic composition and etc.

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16B.2

The JET ITER-like Wall Project

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Currently, the primary ITER materials choice is a full beryllium main wall with CFC (carbon fibre composite) at the divertor strike points and tungsten on the baffles and dome. There is however a concern that tritium retention may still be unacceptably high due to chemical erosion of the CFC near the strike points and transport of hydrocarbons to shadowed areas. ITER therefore still considers a full tungsten divertor to be a backup solution, which carries its own risks of melt damage and plasma contamination.

Neither the ITER reference combination of first wall and divertor materials nor its backup have ever been simultaneously tested in a tokamak. The lessons we

can learn from such an experiment in JET may be decisive for preparing ITER plasma scenarios compatible with a metallic wall, and reducing the risk related to wall and divertor materials in ITER.

The feasibility of an experiment at JET with tungsten in the divertor and beryllium as wall material has been assessed, together with the best options for such a project (W-coated versus W-bulk divertor tiles, Be-limiter or complete Be-wall configuration), costs and time schedule. In a dedicated workshop at JET in October 2004, the following strategy was agreed:

- To replace, where possible, all exposed CFC components of the main chamber of JET with bulk beryllium tiles.
- Initiate R&D for an inertially cooled W-bulk tile design for use in areas near the strike point and to fully characterize W-coating technologies for the CFC divertor tiles.

On that basis, a project has been set-up to design, manufacture and test all the necessary components in view of their installation in a dedicated shutdown in 2008. This paper gives an overview of the present status of the ITER-like wall project and of the future developments. In particular, the on-going R&D for the tungsten divertor (both W-bulk and W-coated tiles) and the main issues linked to the procurement of beryllium tiles are described.

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16B.3

High Heat Flux Facility GLADIS – Operational Characteristics and Results of W7-X Pre-Series Target Tests

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A new ion beam facility for the testing of actively cooled plasma facing components (PFCs) under high heat fluxes is now in operation at IPP Garching. The facility GLADIS (Garching Large Divertor Sample Test Facility) is equipped with two 1 MW power ion sources. Each individual source generates heat loads between 5 and 65 MW/m² at target position. The set-up with water-cooled RF ion sources having a central beam diameter of 70 mm allows effective testing of large components, which can reach a maximum length up to 2 m and a cooling water consumption up to 8.5 l/s. The high perpendicular heat flux density allows an inclination of the target to the beam and thus an enlargement of the heated surface up to 200 mm for a heat flux of 15 MW/m² in the standard operating regime. Therewith the facility has the potential capability for testing of full scale ITER divertor targets.

The installed diagnostics (50 Hz real-time IR camera, CCD camera, pyrometers, water calorimetry and up to 40 fast data acquisition channels for the instrumentation of mock-ups) allow the measurements of the thermal response and the spatial and temporal temperature distributions of tested components.

In the beginning of operation, the ion beam was characterized by calorimetric beam profile measurements.

On the basis of these characterization results, screening and cycling heat load tests up to 15 MW/m² will be performed on the actively cooled WENDELSTEIN 7-X pre-series target elements. The purpose is to confirm the design, the manufacturing route and the acceptance criteria for the production of nearly 950 elements made of copper-chromium-zirconium alloy heat sinks covered with CFC NB31 tiles.

The paper discusses the results of the thermal and thermo-mechanical evaluation and the consequences for the successful series production.

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16B.4

Development of Ultra-Fine Grained Tungsten Alloys and Their Mechanical Properties for Fusion Applications

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Tungsten (W) is superior to other materials in many respects, including high melting points, low thermal expansion coefficients, low sputtering yield and excellent compatibility with liquid metals. However, W and its alloys exhibit serious embrittlement in several regimes; i.e., low-temperature embrittlement, recrystallization embrittlement and radiation embrittlement. Therefore, the authors have been making efforts to achieve simultaneous improvements in the resistance to these types of embrittlement from viewpoints that the most effective microstructure consists of fine grains and finely dispersed particles, the finer the better. In this paper, our current status of the progress will be presented.

Our recent study on low temperature embrittlement in W showed that plastic working after HIP significantly improves the room temperature ductility and the beneficial effect of plastic working becomes prominent with decreasing grain size from 2.0 to 0.6 μm. This suggests that more marked ductility improvement will be achieved by fabricating consolidated bodies with very fine grains less than 0.6 μm in diameter and giving a sufficient degree of plastic working. Therefore, it is at first presented in this paper that consolidated bodies of W-(0.3-0.7) wt%TiC with ultra-fine grains as small as 0.1–0.5 μm in diameter and high relative densities of 99% and higher are successfully fabricated by advanced techniques. Effects of additions of alloying elements on the fabrication and mechanical properties of the consolidated bodies are described. Efforts to achieve a sufficient degree of plastic working by hot forging and rolling for such ultra-fine grained consolidated bodies are also presented.

16B.5

Thermal Stability, Nano Structure, and Chemical Erosion of Metal-Doped Carbon Films

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The plasma-facing walls of thermonuclear fusion devices are subject to erosion, implantation of hydrogen isotopes and re-deposition of eroded material. The major disadvantage of carbon as plasma-facing material is its intense chemical reactivity with hydrogen (and oxygen), resulting in high erosion yields due to chemical erosion and the ability to trap large amounts of hydrogen in re-deposited layers. The chemical erosion yield can be reduced by dopants and the amount of trapped hydrogen could vary with the doping.

Investigations on metal-doped carbon films provide information on the potential of reduction of the chemical erosion by doping and on the erosion behaviour of deposited mixed layers in fusion devices using both, carbon divertors and metal walls.

In this study the preparation and characterization of nano-dispersed metal-doped carbon films are presented with special emphasis on the thermal stability of the

dopant phase and its crystallinity and distribution. In addition, the chemical erosion of these film was determined.

Carbon films doped with Ti, V, W, and Zr of different concentrations (0-20 at%) were produced by means of magnetron sputtering, which distributes metal atoms homogeneously in the carbon matrix. As characterizing techniques x-ray diffraction (XRD), MeV ion beam analysis (IBA), and extended x-ray absorption fine structure (EXAFS) were used. The effects of thermal annealing to temperatures of 400-1500 K on the phase, crystallinity and distribution of the dopant were determined. The three techniques deliver complementary information on different length scales of the crystallization and diffusion. Already heating to 1100 K transforms the initial amorphous films to crystalline ones, in which all four metals are in carbide state with crystallites on the nanometer scale. Diffusion of more than 50 nm is not observed even for heating at 1500 K.

The chemical erosion of these carbide-doped carbon films (pre-heated to 1100 K) was studied in dependence on the dopant type and concentration. A drastic reduction of the erosion yield by one order of magnitude was observed even for concentrations of less than 10 at% of the metal atoms.

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Poster Session 17

Microstructure and Mechanical Properties of Ausformed F82H

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After normalizing and tempering heat treatments, microstructure of F82H includes many precipitates and some grain boundaries are heavily decorated with relatively large $M_{23}C_6$ carbides formed during tempering. Normalized F82H contains high number density of dislocations due to martensitic transformation but most of them will disappear during the tempering heat treatment. In conventional tempering procedure, loss of dislocations starts at lower temperature (at about 773 K) before $M_{23}C_6$ precipitates form at higher temperature. Hot rolling of super-cooled austenite (ausforming) induces martensitic transformation at high temperature and uniform carbide formation in grains with high number density of dislocations. The purpose of this study is to improve the ductility of F82H by modified carbide distribution.

Plates of F82H were kept at 1423 K for 5400 s (90 min) and cooled down to 1023 K, followed by hot rolling. After six passes of hot rolling at 1023 K, the starting thickness 32 mm was reduced down to 16 mm. For the first experiment, process temperature was chosen to be the same temperature as the ordinary tempering condition. Plates were further kept at 1023 K for 1800 s (30 min) after hot rolling, followed by air-cooling.

Vickers hardness (Hv 176) and yield strength (408 MPa) of the processed plates show that present ausformed material is softer than ordinary F82H tempered at 1023 K. Elongation in tensile tests was 22%, which is the highest value ever obtained with F82H. In spite of its high elongation and low strength, ductile-brittle-transition-temperature (DBTT) was 338 K, which is much worse than that of ordinary one. Transmission electron microscopy revealed no lath structure, suggesting no remaining austenite after ausforming. Furthermore, martensite formed during ausforming was almost completely annealed to ferrite. Coarse precipitates at the ferrite grain boundary and in grains were observed.

Tempering under high dislocation density introduced by ausforming is highly accelerated. Plates hot rolled at lower temperatures (973, 923 and 873 K) are also prepared, and better DBTT values are being obtained. Further details on these specimens will be presented at the conference.

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17-2

Effects of Microstructure on Fatigue Fracture Mechanism of Reduced Activation Ferritic/Martensitic Steel

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During the past years Reduced Activation Ferritic/Martensitic steels (RAF/Ms) have been developed that satisfy not only the criteria of reduced long-term activation but also significantly improved impact and fracture toughness properties. The loading of structural materials in fusion reactors is, besides the plasma surface interactions, a combined effect of high fluxes and neutron irradiation. Next step fusion devices can be characterized by plasma burn and off-burn periods generating thermal cycling. Depending on the pulse lengths, the operating conditions, and the thermal conductivity, these oscillating temperature gradients will cause elastic and elastic-plastic cyclic deformation giving rise to (creep-) fatigue in structural first wall and blanket components. Therefore, investigation of fatigue is essential to reactor design. And also, fatigue testing after irradiation will be carried out in hot cells with remote control system. Considering limited ability of specimen manipulation in the cells, the specimen and the test method need to be simple for operation. The existing data bases of RAF/M steel provide baseline data set including post-irradiation fatigue data. However, to perform the accurate fatigue lifetime assessment for ITER-TBM and beyond utilizing the existing data base, the mechanical understanding of fatigue fracture is mandatory.

It has been previously reported by co-authors that dislocation cell structure was developed on low cycle fatigued RAF/M steel, and led the fatigue crack to develop along prior austenitic grain boundary. In this work, the low cycle fatigue properties of RAF/M steels and its fracture mechanisms were examined based on the detailed microstructure analyses. Extraction replica method was used to examine precipitation morphology in detail. Fracture surfaces and crack initiation site were investigated by scanning electron microscope (SEM). Transmission electron microscopy (TEM) was also applied to clarify the microstructural features of fatigue behavior.

It is also important to understand the specimen size effects on microstructure bases, as the small size specimen were used for irradiation experiments as for the limitation of irradiation volume. The low cycle fatigue tests were carried out with cylindrical and hourglass type specimen with several dimensions. Microscopic analyses were carried out to clarify the dimension and geometry effects on fracture mechanism.

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17-3

Change of Microstructure and Mechanical Properties of Modified 9%Cr Martensitic Steel during Creep

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Change of microstructure and mechanical properties during a creep deformation for martensitic modified 9Cr-Mo steel has been studied. Interrupted creep testing was carried out at 600°C under a constant applied stress, using specimens of 6 mm in gauge diameter and 30 mm in gauge length. After testing, the longitudinal cross section of the specimens was observed metallographically by a 200 kV transmission electron microscope (TEM) equipped with an energy dispersive spectrometer (EDS) to determine the coarsening of the precipitates and the growth of martensite lath width during a creep deformation. Tensile properties were measured using AIS 2000 (indentation-typed tensile test system) and Vickers hardness measurements were also performed at a load of 200 g.

The average size of the precipitates was about 55 nm in the as-tempered state. It linearly increased up to 85 nm with the creep exposure time. On the other hand the growth rate of the lath width was constant until a tertiary creep, but the growth rate of the lath width was accelerated during the tertiary creep. The growth behavior of the lath width was consistent with the creep deformation. It shows that the change of the martensite lath width may represent a material softening during a creep deformation. The decrease of the matrix yield strength by the material softening occurred during the creep deformation. Matrix yield strength abruptly decreased at the early stage of creep. The decrease of the matrix yield strength was gradually saturated. When the yield strength of the matrix decreased to about 90% of initial matrix yield strength by the creep deformation, the specimen was ruptured.

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17-4

Reduction of toroidal magnetic field ripple with ferritic steel armors in JT-60U

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Modification for reducing toroidal magnetic field ripple with ferritic steel armors started at Jan. 2005 in JT-60U to reduce the fast ion loss which causes degradation of heating efficiency and heat load on radio frequency (RF) wave launchers in neutral beam (NB) heated discharges with large volume plasma.

Instead of graphite armors in a vacuum vessel, ferritic steel armors will be installed at a part of outboard just inside toroidal magnetic field coils. Distribution of toroidal magnetic field was calculated in detail with 3-D magnetic field calculation code FEMAG. Maximum ripple of toroidal magnetic field can be reduced from 2% to 1% at the surface of large volume plasma. Reduction of fast ion loss was evaluated with Monte-Carlo orbit following code OFMC. Ripple trapped loss and banana drift loss will be reduced to 65 % and 67%, respectively.

Supports of armors have been reinforced for electromagnetic force and acceleration load on ferritic steel armors at plasma disruptions. No limitation in plasma operation due to structural strength of vacuum vessel was confirmed by detail analysis of double wall structure.

Reduced-activation ferritic steel F82H had been confirmed its applicability for high temperature plasma in JFT-2M[1]. Therefore, 8Cr-2W-0.2V heat resisting steel plates, of which concentration limits for the active elements was relaxed from those in F82H for using in JT-60U, was manufactured from 2.6 ton ingots made by 20 ton vacuum induction melting. Plates were heat-treated at 1273 K C for 30 min and air-cooled (normalizing), and then tempered at 1023 K for 90 min followed by air-cooling.

Metallographic examination shows clear tempered martensitic microstructure. Mechanical property at room temperature was measured. Ultimate tensile strength and 0.2% yield strength are 643 MPa and 480 MPa, respectively. Magnetic and thermal properties will be presented in the conference.

[1] K. Tsuzuki et al., Nucl. Fusion, 43(10), 1288 (2003)

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17-5

Mechanical Characterization and Modeling of Brazed Joints of Refractory Alloys

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Within the scope of the European fusion power plant study for development of a concept for a He-cooled divertor, a transition joint between tungsten-based alloys and oxide dispersion-strengthened ferritic steels has been considered. In order to comply with the environmental conditions of the application, the usage of refractory alloys (W-1%La₂O₃ and EUROFER 97) is required. However joints of dissimilar materials suffer from a mismatch in coefficients of thermal expansion. The components of the joint are exposed to mechanical and cyclic thermal loads which give rise to development of high stresses and could possibly lead to fatigue and creep of the materials.

A mechanical characterization and modeling of a joint of refractory alloys is carried out. Preferable joining technique is high-temperature brazing at 1120-1180°C with Ni-based amorphous foils and brazing pastes as filler material. A temperature range between RT to 700°C is defined as operating condition for the materials. A set of finite-element computations has been performed to investigate the joint behavior and characterize it under that combined loading. The analyses are divided into steps to include the residual stresses in the joint as well as to consider the changing thermal load due to operating mode. The calculated stress distribution after a certain amount of load steps at different temperature levels considers different failure modes.

In order to verify the calculated data and provide a deeper understanding of the failure modes observed, mechanical characterization experiments, including tensile, shear, and isothermal fatigue, in the temperature range of RT-650°C are carried out at the Institute for Materials Research II, Forschungszentrum Karlsruhe, Germany.

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17-6

The Effects of Specimen Size on the Cleavage Fracture Toughness of Eurofer97: A Single Variable Experiment

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Eurofer97 is a precursor to the primary candidate martensitic steel for ITER test blanket modules (TBMs). Use of ferritic-martensitic alloys requires adequate defect tolerant margins to protect against fast fracture. A Master Curve (MC) method is being developed to provide a highly efficient means of acquiring and applying fracture toughness information. The MC method enables the use of small to very small specimens required in irradiation experiments. The method assumes that a master toughness-temperature curve, $K_{Jc}(T-T_0)$, exists that can be indexed to an absolute temperature scale by a reference temperature T_0 . Neutron irradiation elevates T_0 (embrittlement). It is also known that other factors such as specimen size and geometry have a strong influence on T_0 . Such effects must be accounted for in measuring toughness with small specimens and application of toughness data to assessing failure limits of fusion structures. Size dependence derives from both the statistical and constraint loss effects. It has been previously shown that critical-stress/critical stressed volume micromechanical local fracture in low alloy pressure vessel steels and a martensitic steel F82H, that is similar to Eurofer97, can be used to treat size effects. Here we present the results of measurements of the K_{Jc} at -142°C using 3 point bend specimens taken from the middle of a single plate of Eurofer97 with independent variation specimen dimensions designed to systematically probe statistical and constraint loss related size effects. The T_0 derived for various specimen configurations for the database composed of 96 points is analyzed using the standard test method in ASTM E1921. These results are compared to a model based analysis of this database described in a companion paper.

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17-7

Precipitation in Ion-Irradiated Reduced-Activation Ferritic/Martensitic Steels

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It was previously reported that reduced-activation ferritic/martensitic steels (RAFs), such as F82H-IEA and JLF-1, showed a variety of changes in its mechanical property after neutron irradiation at 573K up to 5dpa, and have possible correlation with precipitation. The effects of irradiation on precipitation were also reported previously.

In this study, irradiation effects on precipitation were investigated in detail utilizing ion irradiation in which irradiation condition could be controlled with high accuracy. F82H IEA heat, JLF-1 HFIR heat, and aged F82H-IEA (873K x 30k hr) were used for experiments. The specimens were irradiated at DuET facility, Inst. of Advanced Energy, Kyoto University up to 10 dpa at 573K with 6.4MeV Fe³⁺ ion.

Cross sectional TEM thin film specimens of ion irradiated region were made utilizing focused ion beam (FIB) processor with micro-sampling system at JAERI. These thin film specimens were made to contain both irradiated region and non-irradiated region beneath irradiated region. Size distribution and aspect ratio of precipitates were analyzed on each region. It turned out that the finer precipitates were dominant in irradiated region of F82H compared to that in non-irradiated region, but fewer and larger precipitates were dominant in irradiated region of JLF-1. These results confirmed the presence of irradiation effects on precipitate evolution even at 573K, which was observed in neutron irradiated RAFs.

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17-8

Long Term Stability of Finely Dispersed TaC Particle during Tempering 8%Cr-2%W Martensitic Steel

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Precipitation behavior of 8%Cr-2%W martensitic steel during tempering at 740 °C has been studied. Both Cr₂₃C₆ and TaC of an NaCl type have been confirmed at an early stage of tempering by TEM and XRD. Cr₂₃C₆ is coarse, about several tens nm. On the other hand, TaC is very fine, about 10 nm, when tempering up to 10 h. Particle size of TaC is gradually increases, however the size keeps below 20 nm even after tempering for 1000 h. This may suggest that TaC particles grow very slowly at lower temperatures, i.e. the actual service conditions. Chemical composition of TaC is roughly constant, 70Ta-13Cr-9Fe-8W in at%, and the aspect ratio keeps constant, 1.6, during tempering up to 1000 h. After the saturation of the precipitation of TaC, the time exponent for the growing TaC is about 0.1, the value of which is much lower than the theoretical value, i.e. 1/5 or 1/3. This fact is re-confirmed by the measurement of the X-ray half integrated breadth of (111) TaC peak. The reason is discussed in terms of the interactions between TaC particles and dislocations during tempering.

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17-9

Analysis of 300°C neutron irradiation response of Eurofer97 and F82H plate

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NRG has launched an extensive 300°C irradiation campaign with sodium-filled irradiation capsules, of which 7 contained tensile specimens and 4 contained KLST impact specimens. Hundreds of tests were performed on specimens with dose levels ranging from 0.2 to 9 dpa. In this paper, the yield stress hardening at various test temperatures is carefully studied for all capsules and compared to the available literature data. Furthermore, the impact results for Eurofer97 are compared with those for F82H from previous and current programs. 300°C low cycle fatigue data have been produced as well and are compared to F82H data.

The first conclusion must be that the entire irradiation series has been extremely consistent, and therefore the results are presented with high confidence. The yield stress hardening with dose for all tested materials is identical and follows a square root trend up to 3 dpa. Above this dose level the hardening starts to level off.

The impact results very clearly show that the tensile hardening does not have a 1:1 correlation with the shift in DBTT. Where the hardening is identical, the shifts of the various Eurofer97 and F82H heats show considerable variation. It is still evident that Eurofer97 is convincingly better than F82H in terms of impact response. The 25 mm thick plate F82H of IEA heat 9753 had much higher scatter than any other plate material. A re-normalisation and temper treatment was applied to optimise for impact toughness, which also cured the inhomogeneity, but the post-irradiation DBTT shift was not positively affected.

The LCF results show that irradiation at 300°C makes the fatigue life shorten at high strains, most likely due to localised deformations. In the low strain region, the fatigue life is extended by irradiation. Eurofer97 has better fatigue properties in the unirradiated condition but after irradiation F82H is superior.

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17-10

Progress in Development of China Low Activation Martensitic Steel for Fusion Application

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The RAFMs (Reduced Activation Ferritic/Martensitic steels) are considered as the primary candidate structural material for the fusion Demo reactor and the first fusion power station in the future, and a lot of work has been done on EUROFER97, JLF-1, F82H, 9Cr2WVTa in Europe, Japan and USA etc.. A series of R&D activities on structural material CLAM (China Low Activation Martensitic steel) and related technology for liquid LiPb blanket are being carried out in ASIPP (Institute of Plasma Physics, Chinese Academy of Sciences) in the framework of the Knowledge Innovation Program of Chinese Academy of Sciences and National Natural Science Foundation. The studies based on small ingots of a few kilograms have been done in wide collaboration with other institutes and universities in China for years. Recently several ingots of about 20kg (named ASIPP-FDS-heats) have been produced. Melting of large scale ingots for 100kg to one ton is planned, too. This contribution presents a summary of the status of the activities which mainly covers smelting of the steels and controlling of its chemical compositions, testing of physical and mechanical properties, HIP joining, testing of the properties of Al-based coating, corrosion behavior in liquid lithium-lead eutectic and irradiation effect by plasma in the superconducting tokamak HT-7 etc.. Besides, a lot of simulation and analysis on activation characteristics of CLAM steel in various neutron environments of reactor designs have been done including the analysis on impurities' contribution to total dose rate level and also compared with those of the other RAFMs under the same irradiation condition. In addition, a network-based database management code for RAFM steels has been developed.

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17-11

Effect of Dislocation Density on Hydrogen Retention Property of F82H

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A reduced activation ferritic-martensitic steel, F82H, is one of the candidates of the first wall and blanket structural material for fusion reactor. Hydrogen isotope behavior in the material, (such as tritium inventory, permeation and recycling), is affected by the density of defects, the trapping sites for hydrogen. And the density is increased with neutron irradiation damage. In this study, the effect of the defect density on hydrogen retention property of F82H (IEA heat) with various heat treatment has been investigated. Three different tempering conditions were employed as follows.

- (Std) ; Normalized at 1313 K for 0.5 h and tempered at 1023 K for 1 h
- (HT) ; Normalized at 1313 K for 0.5 h and tempered at 1073 K for 1.5 h
- (NT) ; Normalized at 1313 K for 0.5 h and not tempered

The densities of dislocations obtained by TEM observation were $4 \times 10^{13} /m^2$, $1 \times 10^{14} /m^2$ and $2 \times 10^{14} /m^2$ for HT, Std and NT samples, respectively. After the irradiation of the hydrogen beam, 1 keV H_3^+ (i.e. 333eV per H) with a flux of $1 \times 10^{20} H/m^2s$ and a fluence of $1 \times 10^{23} H/m^2$, the samples were heated from room temperature to 773 K to measure the released hydrogen molecules by using QMS.

The experimental result shows that hydrogen retention of F82H increases with density of dislocation in the specimen. Also the similar tendency is found for the temperature where hydrogen desorption becomes maximum. The experimental difference of hydrogen retention between samples is about 20 %. A model to derive the amount of trapping sites from the hydrogen retention data was applied and the difference of the trapping sites is estimated about 50 %.

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17-13

Dynamic Strain Ageing Behavior on Tensile and Low Cycle Fatigue Properties of JLF-1 Steel in Vacuum

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Development of reactor materials and blankets is a critical issue for early realization of fusion energy. A reduced-activation ferritic/martensitic steel, JLF-1, is considered as one of the candidate alloys for the first wall application of fusion reactor. In the low cycle fatigue (LCF) test at 673 K and 873 K in a vacuum, serration was observed on the stress-strain hysteresis curves, which is considered as the dynamic strain ageing (DSA) effect. Since these temperature ranges belong to the operation temperature for fusion reactors, the DSA behavior on JLF-1 was investigated.

The DSA is the phenomenon of interaction between diffusing solute atoms and mobile dislocations during plastic deformation. When the deformation rate is so slow, the mobile dislocation can be pinned by diffusible atoms. The well-known manifestation of this phenomenon is the repeated appearance of serration, load drop and other discontinuities in the stress-strain curves obtained in tensile deformation.

Although the serration on the stress-strain curve can be explained well by DSA, there are still some issues unclear, such as why the onset position of serration change with number of cycles. In this paper, the tensile properties of JLF-1 steel were studied from room temperature to 873K at difference strain rates in a vacuum condition using engineering size cylinder specimens with 8 mm in diameter. The LCF test was also done at 673 K and 873 K. So the DSA behavior on the stress-strain hysteresis curve of LCF can be compared with that of tensile test. The DSA on yield strength and ultimate tensile strength (UTS) of JLF-1 is investigated. The fractography of tensile specimen is observed in a scanning electron microscopy (SEM).

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17-14

**On the Effects of Irradiation Induced
Displacement Damage and Helium on the Yield
Stress Changes and Hardening and Non-hardening
Embrittlement of 8Cr Tempered Martensitic Steels:
Compilation and Analysis of Existing Data**

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Data on irradiation hardening and embrittlement of 8-10Cr normalized and tempered martensitic steels (TMS) alloys has been compiled from the literature, including results from neutron, spallation proton (SP) and He-ion (HI) irradiations. Simple, phenomenological-empirical fitting models were used to assess the dose (displacement-per-atom, dpa), irradiation temperature (T_i) and test temperature (T_t) dependence of yield stress changes ($\Delta\sigma_y$), as well as the corresponding dependence of sub-sized Charpy V-notch impact test transition temperature shifts (ΔT_c). The $\Delta\sigma_y$ are generally similar for SP and neutron irradiations, with very high and low helium to dpa ratios, respectively. Further, the $\Delta\sigma_y$ trends were found to be remarkably consistent with the T_i and dpa hardening-dependence of low alloy steels irradiated at much lower doses. The similar T_i and (low) dose dependence of $\Delta\sigma_y$ and ΔT_c , as well as an analysis of paired ΔT_c - $\Delta\sigma_y$ datasets, show that embrittlement is typically dominated by a hardening mechanism below about 400°C. However, the corresponding hardening-Charpy shift coefficient, $C_c = \Delta T_c / \Delta\sigma_y \approx 0.38 \pm 0.18^\circ\text{C}/\text{MPa}$ is lower than that for the fracture toughness

reference temperature, T_0 , with $\Delta T_c / \Delta\sigma_y \approx 0.6 \pm 0.1^\circ\text{C}/\text{MPa}$, indicating that sub-sized Charpy tests provide *non-conservative* estimates of embrittlement. The C_c increases at $T_i > 400^\circ\text{C}$, and $\Delta T_c > 0$ are sometimes observed in association with $\Delta\sigma_y \leq 0$, indicative of a non-hardening embrittlement (NHE) contribution. Analysis of limited data on embrittlement due to thermal aging supports this conclusion, and we hypothesize that the NHE regime may be shifted to lower temperatures by radiation enhanced diffusion. Possible effects of helium on embrittlement for T_i between 300 and 400°C are also assessed based on observed trends in C_c . The available data is limited, scattered, and potentially confounded. However, collectively the database suggests that there is a minimal NHE due to helium up to a several hundred appm. However, a contribution of helium to NHE appears to emerge at higher helium concentrations, estimated to be more than 500 to 600 appm. This is accompanied by a transition from transgranular cleavage (TGC) to intergranular fracture (IGF). IGF generally occurs only at high $\Delta\sigma_y$. Synergistic combinations of large $\Delta\sigma_y$ and severe NHE due to helium weakening of grain boundaries could lead to very large ΔT in first wall and blanket structures at fusion spectrum dose levels above 50 to 75 dpa and in SP irradiations at much lower doses.

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17-15

Microstructural evolution of a heavily neutron-irradiated ODS ferritic steel (MA957) at elevated temperature

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Oxide Dispersion Strengthened (ODS) ferritic steels have high resistance to radiation damage and superior long-term thermal-mechanical strengths at high temperature, offering a promise of high performance blanket material for advanced fusion energy system as well as core material for advanced fission and transmutation ones. Development of some nuclear energy generating systems has been proposed and supported intensively under several international collaboration programs (Generation IV International Forum (GIF), Advanced Fuel Cycle Initiative (AFCI), International Nuclear Energy Research Initiative (I-NERI) etc).

Current research issue on ODS ferritic steels is considered to be poverty of experience and understanding on their practical neutron-irradiation behaviors at the temperature higher than 600C.

In this research, a MA957, most familiar but primitive 14CrODS ferritic steel contained the highly textured-anisotropic grain structures, was irradiated at 500-700C to fast fluences ranging from 19.8 to 20.8 x 10²⁶ n/m² (E > 0.1MeV) in the experimental fast reactor JOYO. The dose achieved varied from 99 to 104 dpa. TEM observation and micro-hardness measurement were carried out to clarify the irradiation effects on microstructural evolution of 14CrODS ferritic steel at elevated temperature and high dose.

Microstructural examination revealed that all of the highly textured- anisotropic grain structures, following heavy irradiation at the temperature above 600C, have not changed. In addition, large regions in all specimens have retained high dislocation density, contained negligible cavitation. Details on microstructural analyses and micro-hardness measurements would be discussed in this conference.

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17-16

The Effects of Consolidation Temperature, Strength and Microstructure on Fracture Toughness of Nanostructured Ferritic Alloys

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The fracture toughness of nanostructured ferritic alloys (NFA) was assessed. Fe-14Cr-3W-0.5Ti rapidly solidified powders were attritor milled with 0.25% Y₂O₃ and consolidated by HIPing at 850 (nominal), 1000 and 1150°C. This processing produces alloys with very fine grain size and a high density of nm scale dispersions of Y-Ti-O solute clusters and slightly larger Y₂Ti₂O₇ oxides. The scale of these nanofeatures coarsens with increasing consolidation temperature, resulting in a range of alloy strength levels, which have previously been shown to be consistent with the overall microstructure. The ultimate objective of this study is to systematically assess the effects of consolidation temperature, strength and microstructural variations on the constitutive properties and fracture toughness of NFAs, including extrusion versus HIP consolidation paths. Thus we will carry out a complete mechanical and microstructural characterization of these alloys, including K_{Jc}(T) and K_{Jd}(T) using fatigue pre-cracked specimens and the temperature and strain rate dependent tensile properties. The micro-nano-structure will be characterized by TEM, 3D atom probe and SANS. However, due to delays in acquiring the consolidated alloys, interim fracture testing was carried out with sub-sized B=1.65, W=3.3 and L = 18 mm slit-notched bend bars with a flank angle of 0°. The alloys dynamically tested over a range of temperatures to assess K_{pc}(T), where ρ is the notch root radius of ≈ 75μm. Hardness measurements were also carried out at both room temperature hardness and -198°C. The database will be used to develop micromechanical model of K_{Jc}(T) in the cleavage transition within the general framework of the local critical stress-critical volume concept. The model is used to guide the design of alloys with an optimized combination of strength and toughness.

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17-17

Fracture toughness and Tensile Properties of Nano Structured Ferritic Steel 12YWT

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The oxide dispersion-strengthened (ODS) steels are being developed and investigated for fission and fusion application in Japan, Europe, and the United States. In this paper, the fracture toughness and tensile properties of an ODS steel with nominal composition of Fe-12Cr-2.5W-0.4Ti-0.25Y₂O₃ (designated 12YWT) were investigated and compared to commercial RAFM steels. For 12YWT steel, particles were estimated to be only few nm in diameter at a number density of $1-2 \times 10^{23} \text{ m}^{-3}$.

Small, 1.6-mm thick and 3.2-mm wide, 3-point bend specimens were used for fracture toughness characterization of this steel. Specimens were fatigue pre-cracked to initial crack length (a) to width (W) ratio of 0.5 and tested quasi-statically in the temperature range from -50°C to 100°C. Specimens tested up to 50°C exhibited elastic-plastic cleavage fracture that was typical for the transition region in ferritic steels. Specimens tested at 100°C exhibited ductile stable crack growth. In these cases, the J-intergal at the onset of stable crack growth (J_{Ic}) was determined from the J-R curves. Their equivalent values in terms of stress intensity, K_{Ic} , were about 100 MPa $\sqrt{\text{m}}$. This study showed that oxide dispersion resulted in significant decreases in the toughness properties compared to commercial RAFM steels, although appreciable level of toughness was still retained.

Tensile tests were performed at different temperatures from room temperature to 800°C. As expected, this material exhibited very high yield strength, ~1300 MPa, at room temperature. For comparison, yield strength of commercial RAFM steel is about 550 MPa. Yield strength of 12YWT decreases as test temperature increases and at 800°C yield strength is about 323 MPa.

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17-18

First Results On The Characterization Of The Advanced EU Reference RAFM ODS-EUROFER Steel

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In advanced Blanket concepts a replacement of presently considered RAFM steels by suitable ODS alloys would allow a substantial increase of the operating temperature from ~550°C to about 650°C or even more. RAFM-ODS steels of the first generation showed good tensile and creep properties but poor impact behaviour. A breakthrough has been achieved in overcoming the poor high temperature ductility and ductile-to-brittle-transition temperature (DBTT) of first generation RAFM-ODS and commercial ferritic ODS alloys. Selecting a specific production route for Eurofer-0.3wt.-% Y₂O₃-ODS steel which included rolling and appropriate thermal treatments, DBTT could be shifted from values between +60 and +100°C for hiped ODS-Eurofer of the first generation to values between -40 and -80°C. This production route was chosen to produce 50kg of a European reference batch of 6 and 16 mm plates and 12.5 and 20 mm extruded rods. First investigations show that the hardening and tempering behaviour as well as the microstructure of the rolled plates are very similar to that of the first batch. From this it can be expected that also the mechanical properties of the EU reference batch will be very similar. Results of tensile, creep and impact tests will be presented showing that this ODS-Eurofer steel offers not only attractive high temperature strength, but also greatly improved high temperature ductility and DBTT. Microstructural investigations will correlate microstructure and mechanical properties. With the production of the EU reference RAFM ODS steel in principle the scalability from laboratory to larger industrial batches has been successfully demonstrated.

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Characterization of microstructural evolution in nanostructured ferritic alloys using positron annihilation

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Positron annihilation lifetime spectroscopy (PALS) and orbital electron momentum spectra (OEMS) data are coupled with small angle neutron scattering (SANS) observations for defect and nanocluster (NC) evolution in nano-dispersion-strengthened ferritic alloys (NFAs). In this study, NFAs were created by mechanically alloying (MA) Fe-14Cr-3W-0.4Ti-0.25Y₂O₃ (wt%) powders followed by hot isostatic press (HIP) consolidation at 850, 1000 and 1150°C. Additionally MA957 (Fe-14Cr-0.3Mo-0.9Ti-0.30Y₂O₃), which was also found to have NCs by SANS, was characterized in both the as-extruded form as well as after thermal annealing. In the control alloys, which do not contain yttrium and do not contain NCs according to SANS measurements, the positrons primarily annihilate in the matrix and matrix features like dislocations or small solute clusters. A small fraction of the positrons annihilate at large vacancy clusters or gas bubbles. In the case of the yttrium containing alloys, which are shown to contain a high density of NCs by SANS, up to ≈ 50% of the positrons annihilate at non-magnetic features characteristic of Y-Ti-O NCs and, perhaps, smaller vacancy cluster-bubble type features. During thermal annealing, the fraction of positrons annihilating in the Fe matrix increases with increasing annealing temperature, corresponding to microstructural recovery and precipitate coarsening.

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Solid State and Resistance Joining Technologies for Fusion Energy Systems

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An advanced class of nanostructured ferritic alloys (NFAs) that provide excellent resistance to high temperature thermal aging, neutron irradiation damage, and high-temperature creep is being developed for fusion reactor structural applications to allow increased operating temperatures. NFAs are dispersion strengthened with a high density of stable nano-size atomic clusters (NC) and application of these materials requires advanced joining technologies that retain properties similar to base materials. DDL OMNI Engineering under a Small Business Technology Transfer Research (STTR) grant from the Department of Energy (DOE) has investigated the feasibility of applying advanced solid-state and resistance joining technologies to these alloys. Partners include the Edison Welding Institute and Dr. Robert Odette at the University of California, Santa Barbara. The joining technologies evaluated included friction stir welding (FSW) and electro-spark deposition (ESD) processes. The base material used in joining experiments was MA957 that is dispersion strengthened with a high density of Y-Ti-O NC, provided by D. Gelles of the Pacific Northwest National Laboratory. Preservation of MA957 NC is essential for maintaining high-temperature mechanical properties. Phase I demonstrations indicate the MA957 alloy was successfully joined using both ESD and FSW processes based on results of metallographic, microhardness and tensile tests. The effects of FSW and ESD processes on MA957 microstructure were assessed using small angle neutron scattering (SANS). FSW achieved excellent joint efficiency in transverse tensile testing and microhardness testing showed a uniform hardness across the weld region with little scatter, similar to the base material. The ESD process produced a microstructure that consisted of a uniform matrix of extremely small-scale weld deposits and intermittent porosity and voids. ESD welds displayed a lower joint efficiency in transverse tensile testing compared to FSW welds, and microhardness testing showed a hardness decrease in the deposited material. SANS results indicated the NC were still present in FSW welds, but that the NC were damaged in ESD welds. These results appear to correlate with the reduction in mechanical properties across ESD welds. Both processes show promise in joining MA957, with FSW displaying better mechanical properties and NC preservation. This project was sponsored by the DOE under contract DE-FG02-04ER86181.

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17-21

Effect of Irradiation on the Microstructure and the Mechanical Properties of Oxide Dispersion Strengthened EUROFER97 Steel

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The oxide dispersion strengthening (ODS) is an efficient approach to improve the strength of the ferritic/martensitic (F/M) steels at high temperature and a good resistance to swelling on irradiation for the future fusion reactor. This would allow overcoming the operation limit of 550°C of the EUROFER97 base material, whose chemical composition is 8.9 wt. % Cr, 1.1 wt. % W, 0.47 wt. % Mn, 0.2 wt. % V, 0.14 wt. % Ta and 0.11 wt. % C and Fe for the balance. It is found that the addition of 0.3% yttria particles increases the mechanical strength of the matrix material by about 40-50% but to the expense of the material's ductility. The materials strength as measured up to 700°C is maintained with fair ductility. Two types of EUROFER97 ODS steels were irradiated in the PIREX facility with 590 MeV protons. Irradiation doses range from 0.3 to 2 dpa at room temperature and at 350°C. Tensile tests are performed and the obtained results are related to the strength of the unirradiated ones. It appears that after irradiation ODS EUROFER97 shows only a slight change in the mechanical properties. Microstructure of the irradiated samples is analyzed in transmission electron microscopy using the bright field, dark field and weak beam conditions. The presence of voids, dislocation loops are observed in the samples irradiated at 350°C, whereas very small sized defects with sizes from 2 to 3 nm are observed on the samples irradiated at room temperature. The dispersed yttria particles are found to be stable and undissolved on irradiation over all the temperature range and irradiated dose. The relationship between the defects density to dispersoid is measured and it is attempted to relate the effect of the density to the mechanical strength.

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17-22

Water Corrosion Resistance of ODS Ferritic – Martensitic Steel Tubes

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The oxide dispersion strengthened (ODS) ferritic-martensitic steels have superior radiation resistance, and they allow to increase the operation temperature up to around 973K due to their superior creep strength. Those advantages of ODS steels promotes to be applied to the coolant outlet pipe in blanket system of DEMO fusion reactor as well as long-life cladding tubes in the advanced fast reactor fuel elements. The 9Cr martensitic ODS steels have been mainly studied for their excellent radiation resistance. On the other hand, higher chromium 12Cr ferritic ODS steels are expected to exhibit further resistance to water corrosion and high temperature oxidation. As for the high temperature oxidation, it was demonstrated in the previous paper that 9Cr and 12Cr ODS ferritic-martensitic steels have superior resistance, comparing to the conventional 12Cr ferritic steel. However, water corrosion data of ODS ferritic-martensitic steels are very limited. In this study, thus, water corrosion test was conducted for ODS ferritic-martensitic steels, comparing to conventional austenitic stainless steel and ferritic-martensitic stainless steel.

The two types of ODS steel tube were tested: 9Cr-ODS martensite (9Cr-2W-0.13C-0.2Ti-0.35Y₂O₃) and 12Cr-ODS ferrite (12Cr-2W-0.05C-0.3Ti-0.25Y₂O₃). For a comparison, typical fast reactor cladding of austenitic stainless steel (PNC316) and ferritic-martensitic stainless steel (PNC-FMS) were included in this test, where PNC316 is the fuel cladding material for the prototype fast breeder reactor MONJU. Test temperature is 333 K and duration is up to 1,000 h under degassed water environment (containing 1ppm chlorine). The water corrosion test was conducted with varying pH control of 8.4, 10 and 12. Weight loss of each specimen was estimated from amount of the dissolved iron and chromium in the water at before and after test.

In 9Cr-ODS martensite and 12Cr-ODS ferrite, the weight loss due to water corrosion is significantly small and there is no clear difference with PNC-FMS and PNC316. The metallic gloss is kept and no pitting is observed on the surface of all test specimens. In addition, the effect of pH on the water corrosion resistance of ODS ferritic-martensitic steels were discussed from the results of corrosion test in caustic water environment (pH10, 12).

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17-23

Thermal Aging Embrittlement of High Cr Oxide Dispersion Strengthened Steels

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The effects of thermal aging on the mechanical properties are one of critical issues for application of oxide dispersion strengthened (ODS) steels to advanced nuclear reactor systems, which will be serviced at high temperatures for a long time. In this work, microstructural characteristics and mechanical property changes were investigated by using TEM, microhardness and small punch (SP) tests. The materials used were five kinds of ODS steels produced by varying Cr contents from 13 to 22 wt. % but keeping yttria contents within 0.36-0.38 wt. %. Small punch (SP)

specimens were sampled from the extruded rod in such way that the axis direction is parallel to longitudinal or transverse-direction with respective to the extruded direction. Specimens were thermally aged at temperatures between 693 and 773 K up to 1000 hours. Ductile to brittle transition behavior of SP tests was strongly affected by not only the Cr and Al contents, but the specimen sampling orientation, showing greater susceptibility in transverse-direction. Furthermore, SP-ductile to brittle transition temperature (SP-DBTT) and microhardness of thermally aged ODS steels were significantly increased as a function of Cr contents, aging time and temperature. The shift of SP-DBTT in 16 Cr and 19 Cr ODS steel, aged at 693 K for 322 hours was approximately 50 and 73 K, respectively. The embrittlement mechanisms of ODS steels were discussed in terms of the formation of very fine (<1~2 nm) chromium-rich ferrite.

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17-24

Low Cycle Fatigue Properties of ODS Ferritic-martensitic Steels at High Temperature

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The oxide dispersion strengthened (ODS) ferritic-martensitic steels with superior creep strength over 973K are expected as prospective materials not only for the long-life cladding tubes of the advanced fast reactor fuel elements but also for structural materials of blanket systems of DEMO fusion reactor. For ODS ferritic-martensitic steels, the extensive efforts have been made to study the high temperature low cycle fatigue properties.

Two types of specimens were prepared: 9Cr-ODS martensite and 12Cr-ODS ferrite. Both were manufactured by means of hot-extrusion of the mechanically alloyed powders, and were normalized-and-tempered (1323 K x1h and 1073 Kx1h) for 9Cr-ODS martensite and annealed (1523 Kx1h) for 12Cr-ODS ferrite. Basic chemical compositions are 9Cr-0.14C-2W-0.2Ti-0.35Y₂O₃-0.09Ex.O and 12Cr-0.04C-2W-0.25Ti-0.23Y₂O₃-0.05Ex.O. The round bar type specimens were machined from the extruded bars in the dimension of 5 mm in diameter and 10 mm in gauge length. The longitudinal direction of the specimen is parallel to the hot-extruded direction.

The strain-controlled low cycle fatigue tests were conducted at 873 K, 923 K, 973 K and 1023 K. The strain control was completely reversed using a triangular wave form. The total strain ranges were controlled from 0.5 % to 1.5 % with strain rate of 0.1%/s. Corresponding plastic strain covers 0.01 % to 1 %. During the tests, the ODS ferritic-martensitic steels contain relatively low level of plastic strain, because they have large elastic strain region due to their higher yield strength, compared with conventional 12 Cr ferritic-martensitic steels. No noticeable cyclic hardening or softening was observed during the tests. An investigation of the hysteresis loops reveals that all of specimens ideally deformed in an elastic-plastic manner. The strain-life curves were formulated for the number of cycles to failure vs. controlled strain range, based on the Manson-Coffin laws. It was shown that 9Cr ODS martensite and 12Cr ODS ferrite exhibited similar fatigue behaviour, and both were demonstrated to be superior to the conventional 12Cr ferritic-martensitic steels.

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17-25

Effects of Neutron Irradiation on the Mechanical Properties of High-Cr Oxide Dispersion Strengthened Ferritic Steels

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Oxide dispersion strengthened (ODS) steels have been considered to be effective to increase the thermal efficiency of water-cooled solid breeder blanket of fusion reactor, because they showed a high strength at higher temperatures than 823K. High-Cr ODS steels have been developed to improve corrosion resistance in the super critical pressurized water. Irradiation embrittlement caused by the Fe-Cr phase decomposition under neutron irradiation is a critical issue for high-Cr steels. In this work, the effects of neutron irradiation on the mechanical properties of the ODS steels have been investigated.

Various ODS steels which contain 14 ~ 22 wt% chromium, were produced by mechanical alloying method. In order to investigate the susceptibility to the hardening and embrittlement induced by neutron irradiation, the ODS steels were irradiated in JMTR at 673 and 873K up to the neutron dose of 5×10^{20} n/cm². Tensile tests and miniaturized Charpy V notch (MCVN) impact tests were performed before and after irradiation. The microstructure observations were also conducted by TEM.

High-Cr ODS steels showed a significant hardening after the irradiation at 673K, while no effect was observed after the irradiation at 873K. It should be noticed that the significant irradiation hardening was not accompanied by the reduction of total elongation. This behavior is similar to that observed for the 9Cr ODS steel irradiated in HFIR at 573K to 3 dpa. As for the effect of Cr concentration of the irradiation hardening, the hardening became larger with increasing Cr concentration. The mechanism of the irradiation effects on high-Cr ODS steels will be discussed based on the TEM microstructure observation.

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17-26

Pre- and Post-Deformation Microstructures of the Oxide Dispersion Strengthened Ferritic Steels before and after Neutron Irradiation

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Pre- and post-deformation microstructures of oxide dispersion strengthened (ODS) ferritic steels were investigated in order to identify the effects of oxide particle morphology on the tensile-deformation behavior before and after neutron irradiation.

The materials used for the present study were the K1 (19Cr-Ti-Y₂O₃) and K4 (19Cr-Ti-Al-Y₂O₃) ODS steels developed by Kyoto University under the collaboration with JNC and KOBELCO as cross-cutting materials for the application to fusion reactor and Generation IV concept advanced reactors.

Tensile tests revealed that the yield stress of the K1 and K4 were about 1100MPa and 850MPa at room temperature, respectively. Orowan stresses were calculated from the diameter and number density of oxide particles obtained from transmission electron microscopy. The difference in the Orowan stress between the K1 and K4 was well-consistent with that in the yield stresses measured. Energy dispersive X-ray analysis indicated that the chemical compositions of the oxides were different between the K1 and K4; the former includes Y-Ti oxides and the latter includes Y-Al oxide as well as Y-Ti oxides. This suggests that adequate contents of aluminum, titanium and Y₂O₃ should be determined for an optimization of mechanical properties.

The tensile deformation microstructures were also examined for the ODS steels. The microstructure after deformations at temperatures below 600°C consisted of dense dislocations pinned at the dispersed oxide particles. On the other hand, the microstructure after deformations at 800°C consisted of low density of tangled dislocations, indicating that the moving dislocations could overcome the oxides during the deformation at 800°C. Irradiation effects on the tensile deformation microstructure are also discussed.

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17-27

Tritium distribution measurement of the tile gap of JT-60U

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In TFTR, significant amount of tritium retention was found in the tile gaps and then tritium retention in gaps between plasma-facing tiles becomes one of major safety concerns in a fusion reactor because of the difficulty of its removal. It was also found that the tritium was retained in the redeposited layers originated from plasma facing carbon tiles, which was the first indication of close correlation between erosion, migration and redeposition of carbon wall materials and tritium incorporation in the redeposited layers.

In the present work, we have measured the tritium distribution on side surfaces of the plasma facing carbon tiles, facing the gap between the tiles, used in the JT-60U by means of Tritium Imaging Plate Technique (TIPT), which gives a high-resolution 2-dimensional tritium profiles. The samples were graphite and CFC tiles respectively taken from the main chamber and divertor region of JT-60U. Since JT-60U is a D-D discharge machine, tritium is produced by D-D nuclear reactions, having 1 MeV initial energy. In the previous work we have found that tritium distribution on the plasma-facing surface is predominantly determined by the implantation of high energetic tritium ion (triton), and nearly half of totally generated tritium was retained in all plasma facing tiles with the depth of more than 1 μ m. Compared to the plasma facing surface, the tritium concentrations on both toroidal and poloidal sides surfaces were quite low for most of the divertor tiles. Probably because the tile gaps are not directly facing to the plasma and high energetic triton cannot impinge. Hence, tritium incorporated in the redeposited layers on the gap side surface must be originated from thermalized / neutralized one. However, for the outer dome wing tile, significant amount of tritium was found on the bottom side of that was facing to the outer pumping slot. SEM observation showed that the side was covered by thick carbon redeposited layers. This is the first clear indication of tritium codeposition in D-D discharge machine. In the presentation, detailed tritium distribution profiles and comparison with other devices will be discussed.

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17-28

Tritium Permeation Barrier for the First Wall of a Fusion Reactor

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In the case of reduced activation steel as a structural material, recent investigations of blanket concepts from the European Power Plant Conceptual Study (PPCS) indicate that a fraction of the particle flux impinging onto the first wall of a blanket module from the plasma can diffuse through the structural material into the coolant [1]. This will lead to tritium contamination of the coolant. For this reason we suggest, that a tritium permeation barrier may be required for the plasma-facing wall of a blanket module.

The usual materials approach for tritium diffusion barriers, oxide ceramics, is incompatible with the requirements for first wall materials concerning erosion by physical sputtering. A tungsten coating is proposed as plasma-facing material [2]. Either this tungsten coating turns out to be sufficient to suppress tritium permeation into the structural material, or a double-layered coating is required, with a tungsten coating facing the plasma and an intermediate diffusion barrier between the top tungsten coating and the structural material. In the latter case, the permeation reduction performance of the intermediate barrier coating will be different from the usually investigated configuration due to the absence of surface effects on the ceramic. Tritium will enter the barrier coating in atomic form instead of dissociating on its surface.

We investigated the permeation barrier performance of such combinations of thin coatings: Coatings of tungsten and alumina with thicknesses in the μ m range were deposited on EUROFER 97 steel. The following combinations were examined: Bare EUROFER, EUROFER with a tungsten coating, EUROFER with an alumina coating, and the combination EUROFER-alumina-tungsten. The coatings were applied by vacuum arc deposition for alumina and by magnetron sputtering for tungsten. While a 1 μ m tungsten coating reduces the permeated flux by roughly 1 order of magnitude, a 1 μ m alumina coating leads to a reduction of about three orders of magnitude. The deposition of tungsten on top of an alumina coating in turn reduces its barrier performance by about a factor of two.

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17-29

Performance of a hydrogen sensor in Pb-16Li

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In the HCLL (Helium Cooled Lithium Lead) TBM (Test Blanket Module) for ITER, a correct and reliable management of tritium is of basic importance, both for safety and fuel cycle reasons. To develop a sensor for measurements of hydrogen (and its isotopes) concentration in liquid Pb-16Li, a permeating capsule is being developed. Niobium was initially chosen as constructing material. The experimental results, however, both in gas phase and in liquid metal, showed a permeating flux much lower than predicted, most probably due to the formation of an oxide layer. The sensor could operate neither in dynamic nor in static mode in this way. To overcome this problem, another permeable material (Fe Armco) has been chosen.

The new sensor has been constructed and tested in the LEDI device in ENEA Brasimone. The testing temperature was 350-550 °C and the external hydrogen pressure between 200 and 1100 mbar. Results of testing have shown that the sensor cannot be operated in equilibrium mode yet, at least at the present conditions, because of the very long time necessary to reach the hydrogen pressure equilibrium. In spite of these results the equilibrium mode has not to be definitively excluded: in fact, by means of a substantial optimisation of the sensor geometry the pressurisation rate could be fast enough to reach equilibrium pressure in a relatively short time. In this frame, the preliminary designs of different possible solutions have been developed and the good results, in terms of performances of these optimised sensors, have been demonstrated by several simulations performed with an ad-hoc developed code.

Present experimental results are however positive, since they have shown that the Fe sensor can be successfully operated in dynamic mode, since it is possible to link, at each temperature, the permeation fluxes to the hydrogen test pressure by means of a simple power equation: then, hydrogen concentration in the liquid metal is determined by the Sieverts' law. The Fe-based sensor quickly reaches the steady-state value of the permeating flux and therefore has a response fast enough to follow rapid changes of the hydrogen concentration in the liquid metal.

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17-30

Reaction Rate of Be with Fluorine Ion for Flibe Redox Control

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Flibe is one of the most promising liquid blanket materials for fusion reactors. Tritium is generated in a form of tritium fluoride there. Tritium fluoride is so corrosive to materials that it is supposed to be transformed to an elementary form of T₂. Therefore, it is the most important thing to know how fast tritium generated in the Flibe blanket is converted to the elementary form. The Flibe/Chemistry study of JUPITER-II task 1-1-A is being carried out as a collaboration work between Japan and US since 2001. In order to estimate the Redox reaction rate of elementary Be with HF dissolved in Flibe, three governing equations of a HF material balance equation, a Be resolution rate equation and an impurity reaction rate equation were simultaneously solved. The major impurity assumed here was an iron ion, because about 100 ppm iron ion was observed in purified Flibe by ICP-mass spectrometer. The first-order and second-order reaction rate equations were compared based on predicting the kinetic behavior of Be dissolved with HF. The saturated Be concentration and the linear dissolving rate constant were determined based on fitting the numerical curve to experimental ones. Using the dissolution rate and the reaction rate constant determined experimentally, the tritium concentration changes in a Flibe blanket was estimated for the application to a fusion reactor system, e.g. FFHR-2. It was found that the rate constant is enough high that tritium generated in a Flibe blanket of the FFHR-2 is sufficiently fast converted to the elementary form.

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17-31

Measurement of tritium trapped in the irradiation defects produced by high energy proton and spallation neutron in SS316

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For the accurate evaluation of tritium transport not only in the DT fusion devices but also the transmutation facilities, which exposed to higher neutron irradiation conditions, irradiation effect on tritium trapping in SS316 has been investigated by means of spallation neutron source irradiation. The specimens were the SS316 disks (3mm ϕ , 0.2mm^t, 13mg) irradiated by mixture beam of spallation neutron and 580MeV proton up to 5.0 ~5.9 dpa at 363 or 433 K. The experiment was carried out by measuring tritium generated by the spallation reaction in the SS316 specimens by the thermal desorption method (TDS) with a constant ramp rate of temperature in the constant helium-hydrogen (~2%) mixture carrier gas flow. Tritium measurement was carried out by real time tritium concentration measurement by an ion chamber and tritium collection by water bubblers after oxidation of tritium by the oxidation catalysts.

As the result, the ion chamber measurements indicate that tritium release behavior does not depend on the irradiation temperature, i.e., tritium release starts above 523 K and tritium release shows peak at about 673 K, and additional tritium release is not observed above 673 K. On the other hand, results of tritium collection by the water bubblers revealed that residual tritium in the SS316 specimen was less than 10% of calculated produced tritium amount for each specimen. Those experimental results indicate that residual tritium in the SS316 specimen is attributed to the trapped tritium in the irradiation defects, and contribution of mobile tritium is small. Additionally, the tritium transport analysis based on the one-dimensional diffusion model also has been performed not only for the TDS experiment but also for the irradiation and storage period. The results indicate that there exists irradiation defects acts as hydrogen isotopes trap site of about 250appm with trap formation energy of about 0.7 eV in the irradiated SS316, with which both of the residual tritium amount and TDS behavior of tritium in/from specimen can be well explained.

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17-32

Behavior of Hydrogen Isotopes Irradiated in LiAlO₂

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For predicting the tritium recovery process, behavior of hydrogen isotopes in the blanket breeding materials should be clarified. In this paper, a systematic experiment was devoted to LiAlO₂, one of the candidate tritium breeders. In-situ Fourier transform infrared spectroscopy (FT-IR) was performed during 3 keV deuterium ion irradiation at room temperature, and the thermal desorption spectroscopy (TDS) was conducted to evaluate the deuterium desorption process after the ion irradiation. The existence state and the thermal stability of the irradiated deuterium were investigated as a fundamental study.

In the IR spectra obtained during the deuterium ion irradiation, a broad peak was observed at wave number of 2750-2400 cm⁻¹ corresponding to overlapped peaks from the stretching vibrations of O-Ds in multiple existence states. The shape of the peak depended on the ion fluence, due to the accumulation of the radiation damage, such as the formation of lattice defects. The LiOD, which is a main chemical form of deuterium in Li₂O under the same condition, was not found. In the TDS experiment after the ion irradiation, D₂O and D₂ were desorbed. The ratio of D₂/D₂O amounts was dependent on the ion fluence and the desorption temperature. It was also observed that the shape of the IR peak was changed during the TDS experiment. These facts indicated that the deuterium near the surface existing in various chemical forms was desorbed in different manners because of the radiation effect. A model to describe these behaviors of irradiated deuterium in LiAlO₂ was proposed based on the experimental results.

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17-33

He-O Glow Discharge at Elevated Temperatures for the Removal of the Codeposited C/H Layer

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The buildup of tritium inventory in the layer formed by the codeposition of eroded carbon and tritium from the plasma presents the ITER fusion reactor with one of its greatest challenges. A relatively fast and effective way to remove this layer once formed is a necessity. In this study we examine the combination of a He-O glow discharge with heating as a possible removal technique. The experiments were performed using a tile removed from the TFTR reactor prior to the tritium campaign. Samples approximately 1 cm by 1 cm by 2 mm were cut from a relatively large area of the tile containing a uniform codeposited layer of deuterium and carbon. SEM was used to generate micrographs of each of the samples. Individual samples were then exposed to a He-O glow discharge while being heated. Sample exposures included the following conditions: 1 hour at 373 K, 443 K, and 513 K; and 4 hours at 443 K. After the exposure, the samples were returned for SEM observation of the same areas examined prior to the exposure. Comparison of the before and after images revealed that the amount of the codeposited layer removed was significantly less than 1 μm . Removal rates this low would suggest that He-O glow discharge with heating is insufficient to remove the thick layers predicted for ITER in a timely fashion. The only condition that might still suggest He-O glow with heating was a viable technique for tritium inventory reduction would be if the technique happened to preferentially remove the deuterium in the sample and leave the carbon. To examine this possibility, $\text{D}(\text{}^3\text{He},\text{p})\text{}^4\text{He}$ nuclear reaction profiling was performed on the exposed surface of each sample. The results revealed a slight reduction in the deuterium content in a near-surface region less than one micron thick, and no reduction at depths greater than one micron. The final conclusion of this work is that He-O glow discharge even when combined with heating is not sufficient to remove the thick codeposited layers expected for the ITER fusion reactor.

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17-34

Thermal release rate of tritium trapped in bulk and plasma exposed surfaces of carbon specimens obtained from JET divertor

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Tritium co-deposition with carbon in the colder remote areas of the JET vessel immobilises a substantial amount of the fuel and increases the tritium inventory of the machine. A well defined physical or chemical description of these co-deposits is still not available. Therefore they are reported in the literature as a carbonaceous amorphous C:H layer, and unfortunately sometimes as a "diamond-like films" referring to their sp^3 hybridisation.

From the present study it appears that the gas-solid reactions taking place during the thermal treatment of carbon samples involves various tritiated hydrocarbon species having one or two carbon atoms in their structure. As the deuterium and tritium content for such compounds is very high e.g. D/C~0.75, such small chain hydrocarbons, like methyl ($-\text{CH}_3$) or ethyl ($-\text{CH}_2\text{CH}_3$), must exhibit mainly a sp^3 hybridisation.

The thermo-desorption measurements have shown that under a stream of He containing 0.1% H_2 , tritium is released only at temperatures above 400°C. While, after heating at 1100°C, full combustion measurements confirmed that there is no more tritium remaining in the sample.

Finally, the thermal response of the samples was also investigated by laser irradiation and can be explained adequately in terms of co-deposition. In the absence of any co-deposition the uniformly good thermal conductivity of the bare tile allows an easy conduction of the heat from the surface. A co-deposited layer has poor thermal conductivity and also poor thermal contacts with the substrate and this does not allow efficient dissipation of the heat induced by the laser beam.

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17-35

Efficiency of tritium removal techniques in castellated structures

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The use of carbon materials in the high flux area of fusion devices, including ITER divertor for the first phase, is seriously hampered by the formation of co-deposits of carbon and the plasma fuel which are found to migrate to remote areas not reached by the fusion plasma. This problem has triggered the development of a series of in-situ cleaning techniques aimed at the removal of these co-deposits or at least to their tritium content. Among these, the high temperature oxidation and the glow discharge-driven erosion or isotope interchange have been considered for ITER operation (1). On the other hand, the possibility of trapping of these co-deposits in gaps such as those existing between the tiles of the plasma facing components (PFCs) or within the castellated structure of the divertor modules poses an extra constraint to the available cleaning technique, even for the nominal PFCs. In this work, the problem of carbon film removal from the gap existing in castellated structures such as that of the macro brush design has been addressed. A metallic structure with gaps of 1mm and 1cm depth, in which bottom a carbon-film layer has been deposited, was exposed to several cleaning techniques. In particular hydrogen and nitrogen diluted glow discharges, on one hand, and high temperature oxygen exposure have been investigated. In the first case, the impact of glow discharge parameters and gas mixture on the cleaning efficiency has been investigated by mass spectrometry and thermal desorption spectroscopy. In the oxidation experiments, the effect of temperature and gas pressure has been investigated with the same diagnostics. Finally, conclusions about the design of tiles and divertor modules in ITER from the point of view of tritium removal requirements will be presented.

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17-36

Applicability of Pd-Cu Alloy to Self-Developing Gas Chromatography of Hydrogen Isotopes

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To efficiently recover tritium in hydrogen isotopes from exhaust gases of D-T fusion devices such as ITER is one of key issues to keep safety and to reuse as fuel gas. A new separation technique (SDGC: self-developing gas chromatography) of hydrogen isotope mixtures has been recently developed, which is based on utilization of the isotope effects in the absorption and desorption process of hydrogen isotopes. In this new technique, Pd-Pt alloys were applied as a promising candidate of column materials. However, a practical use of such alloys requires reduction of the amount of these metals because of expensive materials. To solve this problem, it is necessary to seek an alternative of platinum and to reduce the amount of palladium. From this viewpoint, we have examined thermodynamic properties of hydride and deuteride of Pd-Cu alloy, and separation tests were also carried out using this alloy.

Absorption and desorption isotherms of hydrogen and deuterium were measured using Pd-Cu alloys having different composition of copper (4, 8, 10, 15 and 25 at.%) in a temperature range from 323 to 453K. Thermodynamic properties such as ΔH° and ΔS° of hydride and deuteride were evaluated from the temperature dependence of isotherms for each Pd-Cu alloy. In addition, ratios of an equilibrium dissociation pressure of deuteride to that of hydride were examined in the same temperature and copper composition, and it was seen that they were dependent on not only temperature but also the composition of copper: they decreased with increasing copper composition as well as temperature. On the other hand, separation tests of H₂-D₂ mixtures were carried out in a temperature range from 288 to 304K by using Pd-25at.%Cu alloy powder as the column material. It was seen from the observed chromatograms that it is possible to separate hydrogen isotopes by Pd-Cu alloys, indicating that Pd-Cu alloy can be used as alternative of Pd-Pt alloy.

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17-37

Tritium Control Design of the HCPB Blanket Concept

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The Tritium control is one of the most important issues in the design of a fusion blanket: under this term it is intended not only all the processes and systems connected to the recovery of the Tritium produced in the breeding material, but also all the features required to minimise the permeation of the Tritium into the main coolant or to reduce the Tritium inventory in the materials for the implication in the safety due to the potential release in the environment. The paper presents the Tritium control design for the Helium Cooled Pebble Bed (HCPB) blanket concept proposed in 2001 as reference European DEMO solid breeder blanket.

In the HCPB the tritium is produced by neutron reaction in a lithiated ternary ceramics (Li_4SiO_4 or Li_2TiO_3). The breeding materials, in form of pebbles (diameter <1 mm), is purged by a low pressure flow of Helium that extracts the tritium from the pebble beds. An addition of H_2 to the purge flow (reference value 0.1%) contributes to facilitate the extraction of T from the pebbles. The outlet gas mixture (mainly He + HT + H_2) is processed outside the reactor; the tritium removal process considered in the HCPB Design is based on a combination of cold traps and molecular sieves (operating in Temperature Swing Adsorption configuration), for the liquid and gaseous T forms, respectively. The gaseous stream is then processed to recover the fuel for the thermonuclear reaction.

The second objective of the design is to minimize the quantity of T that contaminates the main coolant flow. Sources of Tritium are the permeation inside the blanket module from the purge loop and the impingement of Tritium ions in the FW and hence into the cooling channels. A Coolant Purification System is used to process continuously a by-pass stream of the Helium Coolant Loops keeping the level of T in it under a design value (~0.8 Pa) that is considered sufficient low to keep the potential losses of T in the steam generator below a safety acceptable level. Furthermore, the overall inventory of the T in the blanket materials should be minimised to avoid the risk of T release during accident.

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17-38

HTO Electrolysis Method by Using Proton Exchange Membrane Fuel Cell

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The system of proton exchange membrane fuel cell (PEMFC) can also be applied as the electrolyzer of HTO bred in the solid breeder blanket of a D-T fusion reactor, although the PEMFC may be the effective power source of a future motor vehicle. The bred tritium in the solid breeder blanket will be recovered by the purging operation using the helium gas mixed with H_2 or H_2O , and it has been recognized in the recent studies that not a little amount of bred tritium is released to the purge gas in the chemical form of HTO even when helium gas with H_2 is used as the blanket purge gas. We consider that application of the PEMFC system for conversion of HTO to HT is reasonable because it is preferable to transfer bred tritium to the main fuel system in the chemical form of HT. Accordingly, it is important to understand the performance of the electrolysis system using the PEMFC in the tritium atmosphere and to quantify the tritium inventory in the system.

The mass transfer performance of H_2 , HT, H_2O , HTO and He in the proton exchange membrane (PEM) placed in the blanket purge gas atmosphere is discussed in this report using the mass transfer coefficients and adsorption isotherm of water reported by the present authors elsewhere. The tritium inventory in the PEM system used as the HTO electrolyzer is also discussed, where the amount of tritium both in adsorbed water and structural water in the membrane taken up through adsorption and isotope exchange reaction is estimated.

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Measurement System for In-pile Tritium Monitoring from Li_2TiO_3 Ceramics at WWRK

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220-days irradiation of Li_2TiO_3 ceramics with 95% enrichment by isotope 6Li was carried out at WWRK reactor. One of the study goals was to examine tritium release behavior during Li burn-up. To achieve this goal three types of ceramics samples were examined simultaneously: one — under constant temperature of 650C, and two (pebbles and pellets) — within temperature change ranges from 500 to 900C.

Three experimental ampoules and three-channel tritium measurement system “Sakura” has been merged with stationary WWRK’s multi-purpose loop facility. “Sakura” consists of three analytical systems (mass-analyzers) and automated measurement system. Each analytical system consists of hydrogen Pd-Ag filter with heater and temperature control system, vacuum system and mass-spectrometer. Automated measurement system consists of controlled oscillator CO (frequency synthesizer) —source of operating frequency for omegatron-type mass-spectrometers, relay multiplexer, and digital voltmeter linked by serial interface (RS-232) with controlling computer. This computer controls CO and multiplexer and reads measured values from voltmeter using serial port COM.

Second remote computer is provided for remote control of measurement system using Ethernet link and remote administrating software. Both computers are linked in local network by UTP-5 cable connection (up to 100 meters long) using TCP/IP protocol. Remote administering allows to control computer from a safe distance: run and stop measurement program, display operative screen content at remote computer, save and exchange data-files.

At Pd-Ag filter operational temperatures tritium and its compounds, which are produced by ceramics under irradiation, easily penetrate into the analytical system and its flows were measured there. Described measurement system was able to provide reliable operation along whole duration of irradiation. Lithium ceramics has been extracted after dismantling and cutting in a ‘hot’ cell of the irradiation ampoules. The studies of residual tritium in irradiated samples are carrying out. Flows of tritium release from ceramics and its temperature during the reactor campaigns with different conditions are represented in the paper.

The work main result is justification of burn-up in 6Li reaching up to 20% of Li_2TiO_3 ceramics with 95% enrichment by 6Li .

The work is fulfilled in the framework of ISTC K-578 Project under funding in JAERI (Japan).

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17-40

Erbium oxide as a new promising tritium permeation barrier

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Among several approaches on how to control tritium permeation and accumulation in the first wall of a fusion reactor, the application of a permeation barrier in the form of a coating looks feasible. For instance, alumina coatings have been studied for decades and are recognized to be suitable since a few microns of a crystalline α -alumina layer is capable of reducing the permeability a fusion relevant material, e.g. EUROFER 97, up to a factor of 10^3 and more. But crystalline alumina also suffers from some problems such as advanced requirements for its formation.

Recently, we focused our attention on another crystalline coating, erbium oxide, which possesses some advantages over alumina in terms of the coating's production. While erbia has demonstrated a permeation reduction factor about two times lower than alumina, it can easily form the required crystalline structure revealing no crack formation being deposited on steels like EUROFER 97 and further tested on thermal loads, is stable with respect to aggressive environments like liquid lithium (therefore, Pb-17Li as well), keeps its electrical properties being irradiated with neutrons etc. Of course, some aspects of the erbia application as a diffusion barrier need to be found out, such as affecting neutronics of the blanket.

Here we discuss the applicability of erbia as the tritium permeation barrier for different blanket concepts providing experimental results on the suppression of hydrogen permeation by using thin erbia coatings deposited with various techniques. The latter is of special concern since the possibility to cover complex surfaces with erbia could be a serious profit over other barrier materials, e.g. alumina.

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17-41

On the mechanism of the disproportionation of ZrCo hydrides

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Among the getter materials the interalloy ZrCo has been selected by the ITER team as reference material for the storage of hydrogen isotopes at the Tritium Plant because of its good getter properties, which are comparable to those of uranium. However the fact that under certain conditions, presence of simultaneous high partial pressure of H_2 and high temperature, ZrCo can lose its activity or during repeated hydrogen (or hydrogen isotopes) absorption-desorption heat cycles has been a matter of concern. Under thermal cycling the getter hydride material tends to disproportionate, i.e. convert into ZrH_2 and $ZrCo_2$, and thus show significant performance degradation in its gettering properties. A detailed investigation to quantify the conditions leading to disproportionation and, in particular, re-proportionation of ZrCo was therefore undertaken. To investigate the disproportionation of ZrCo, reaction vessels of quartz and ceramic were used equipped with systems to evacuate the vessel, handle hydrogen isotopes and analyze the gases. X-ray diffraction analysis of the solid material was used to identify the phases involved. In addition, thermal techniques were employed to study the release characteristics of the getter as a function of temperature. A mechanism of disproportionation is discussed in the present paper trying to give a qualitative explanation based on crystallographic considerations. In parallel, an attempt was made to learn more about the mechanism of re-proportionation.

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Neutron Elastic Recoil Detection for Hydrogen Isotope Analysis in Fusion Materials

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Hydrogen isotopes show complicated behavior on the surface of plasma facing components (PFC) in fusion devices, and the study is important for the design of the fuel recycling, plasma control, tritium inventory, etc. In the case carbon-based materials as PFC, co-deposited layers of carbon compounds are formed with the thickness of several tens of micrometers on PFC surfaces due to plasma-wall interactions. Since these layers retain a lot of hydrogen isotopes more than the bulk of PFC, it is necessary to reveal hydrogen isotope distributions in the layer. However, conventional ion beam analyses for hydrogen isotopes were not appropriate in such depth region. In this study, we propose the Neutron Elastic Recoil Detection Analysis (NERDA) using a neutron beam to extend the analyzing depth of hydrogen isotopes up to several hundreds micrometers.

The 14.1 MeV neutrons produced by $T(d,n)^4He$ reactions with 350 keV deuterons at the target of the 0° beam line of the Fusion Neutron Source were collimated with a through-hole of 20 mm in diameter. The collimated neutrons, *i.e.* a neutron beam, entered a sample from the normal direction. Emitted particles from the sample were measured using a ΔE -E counter telescope detector positioned at the detection angle of 25° with the solid angle of 1.8×10^{-2} sr. Typical fluence of incident neutrons was 4.0×10^{13} neutrons/m², which was monitored with a ²³⁸U fission chamber located behind the target chamber.

The proof-of-principle experiment was performed using a standard sample of deuterated polyethylene film containing a known concentration of deuterium with the thickness of 100 μ m. The depth resolution was evaluated to be 99 μ m corresponding to 12 % of the maximum probing depth of 801 μ m for the sample. For a carbon-based PFC sample the depth resolution was expected to be 61 μ m, which was enough to reveal hydrogen isotope distributions of co-deposited layers. In further work, we will apply NERDA to analyses of hydrogen isotope distributions in PFCs of JT-60U and TFTR.

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Characteristics on tritium release during plasma operation on JT-60U

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During DD plasma discharges, tritium produced by the discharges is accumulated in-vessel components. Simultaneously, however, the plasma heating or the impinging of high energy particles causes the detritiation of the in-vessel components. Since it is crucial to ensure tritium safety and the process control on a fusion reactor with a carbon based first wall and the detritiation during the discharge helps this, an optimization for the detritiation of the in-vessel components during plasma operation on JT-60U is pursued. The recent modification of the control system on JT-60U, protracting the pulses of heated plasma discharges from 15 sec. to max. 65 sec. and its heating from 10 sec. to max. 30 sec., contributes to study on the detritiation of the in-vessel components.

(1) Comparison of detritiation capability during plasma operation including He glow discharges, He Taylor discharges, and various kinds of plasma heating, suggests that longer pulses of Radio Frequency plasma heating such as ECH and LH, injecting approximately max. 16 MJ respectively, or those combination, provides the most efficient detritiation of the in-vessel component in the recent campaign. During such discharges, the tritium exhaust was larger than the production though the optimization would be still needed for the detritiation. He glow discharges after the experimental campaign on those days also provided effective detritiation consuming modest power of 300 V and 6 A.

(2) NB heated DD plasma discharge, injecting max. 350 MJ including the negative ion based NB, added in-vessel tritium shot by shot with a few ten percent of the tritium produced by discharge. The DD plasma discharges heated with the longer power negative ion based NB enhance the exhaust of the in-vessel tritium. Simultaneously, a temperature rise of the divertor tiles leads the detritiation, where it critically depends on the temperature. NB heated HH plasmas have also detritiation capability, since they do not produce tritium and consume high power to enhance detritiation due to the poor neutralization efficiency.

(3) The combination of longer pulses of high power RF heating and He glow discharges, therefore, could provide the much efficient detritiation approach during DD plasma operation.

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17-44

Pulsed-laser ablation of co-deposits on JT-60 graphite tile

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Pulsed-laser ablation (PLA) is considered to be one of the effective methods to remove tritium co-deposited with carbon in ITER and D-T fusion reactors. In order to optimize laser-irradiation condition to remove tritium from the co-deposits, we have investigated PLA of two graphite targets; (a) the co-deposits on JT-60 graphite tiles exposed to hydrogen plasmas and (b) an isotropic-graphite target irradiated by 10 keV-H⁺ or 8 keV-D⁺ ions with a fluence of 10²² ions/m².

Two fourth harmonics of Nd:YAG laser ($\lambda = 266$ nm) systems, a ns laser (Continuum Minilite II, $\tau \sim 4$ ns, $E_L < 4$ mJ/pulse, $I_L < 3 \times 10^9$ W/cm², repetition rate: 10 Hz) and a ps laser (Continuum Custom PY61C-10, 20 ps, < 3 mJ/pulse, $< 3 \times 10^{11}$ W/cm², 10 Hz), and an ArF excimer laser (Lambda Physik OPTex, 193 nm, ~ 8 ns, < 13 mJ/pulse, $< 2 \times 10^9$ W/cm², 5 Hz) were used to investigate dependence of PLA on wavelength, pulse width, and intensity of the lasers. Visible light emissions, emitted ions, and desorbed gases were measured by a spectrometer, a time-of-flight mass spectrometer (TOFMS), and a quadrupole mass spectrometer (QMS), respectively.

With the ps-266 nm laser irradiation of the D-implanted graphite target, two different ablation regime were clearly distinguished, i.e. a weakly-ablated (WA) region ($I_L = 0.5 - 8 \times 10^{10}$ W/cm²) and a strongly-ablated (SA) region ($I_L > 8 \times 10^{10}$ W/cm²) above the laser intensity of ablation threshold ($I_L = 0.5 \times 10^{10}$ W/cm²). In the WA region, large-size carbon-cluster ions C_n⁺ were emitted, and visible emission spectra showed only C₂ Swan band, C₃ and CI emissions. In the SA, a large amount of C⁺ ions was emitted, and the emissions of CI, CII, and CIII lines were dominant. QMS observations showed that desorption of D₂ molecules were appreciable in the WA region compared with that in the SA region. When the ns-266 nm or 193 nm ArF laser was irradiated, only the WA region was observed. Details of the ablation mechanism of the JT-60 graphite tiles and the H- or D-implanted graphite will be discussed, and an optimal condition for hydrogen removal will be proposed.

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17-45

Tritium recovery from isotropic graphite and CFC used to plasma facing material

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Development of an effective measurement procedure to evaluate the amount of tritium remained in the plasma facing material is one of the key issues from viewpoint of fuel recycling or radioactive safety for establishment of the nuclear fusion reactor. In the tritium releasing operations after D-D discharge experiment of JT-60U, it has been gradually cleared that tritium remains not only in the re-deposited layer on the tile surface but also in the tile bulk. Therefore, the suitable releasing methods of tritium in the bulk of tile should be developed. In this study, tritium thermal release operation with dry gas purge, isotope exchange with hydrogen or water vapor and combustion with oxygen were performed for the isotropic graphite tile and Carbon Fiber Composite (CFC) tile used for JT-60U. Obvious re-deposited layers were not found on the surface of any sample tiles.

It was observed in this study that all the tritium retained in the both tiles were not released by dry gas purge and following hydrogen gas purge in one day even at so high temperature. About 20 % of tritium was remained in the tile after hydrogen gas purge at 800 °C for one day and about 1.5 % of tritium was remained in the tile after hydrogen gas purge at higher temperature as 1200 °C. Therefore, combustion of the tile with oxygen was required to release all the tritium still remained in the tile. It was also observed that the practical combustion temperature should be around 800 °C when releasing period is limited in one day though both tiles began to combust at the temperature around 600 °C. The grain of isotropic graphite tile combusted in the manner as that all grain size was reduced equally. On the other hand, the fiber of the CFC combusted in the manner as that porosity of fiber was reduced keeping the diameter of fiber was almost constant. It is concluded that complete combustion of tile is required to recover all tritium exist in the grain whether the grain exist in the plasma facing side of the tile or not.

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17-46

In-reactor vacuum RIED experiment on sapphire

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The possibility that ceramic insulators will suffer RIED (Radiation Induced Electrical Degradation) has led to recommendations which seriously restrict their use in ITER (limited temperature, voltage, radiation field). With a few notable exceptions, due to the extreme difficulty associated with in-reactor electrical studies to date most of the experimental RIED data giving rise to these recommendations have been obtained from particle accelerator experiments.

However the controversial nature and potential importance of RIED not only for ITER but also for other future fusion devices, means that the need for a versatile experiment in a nuclear environment is urgent. To meet this necessity an experimental rig to enable in-reactor experiments to measure online electrical conductivity of ceramic insulating materials in vacuum at controlled temperatures has been designed and constructed. Despite the number and length of cables involved, the system allows conductivities as low as 10^{-14} S/m to be independently measured for 3 sets of samples at different temperatures, in a pumped vacuum in the 10^{-3} mbar range.

Experiments are now underway in the BR2 reactor at SCK•CEN Mol, irradiating sapphire samples at 75 to 90 Gy/s, and temperatures between 300 and 400 C. The reactor is run at low power (1.8 MW) as a compromise between nuclear heating, temperature control, and dose rate. A special channel is used to enable the rig to be inserted and withdrawn during reactor operation, thus allowing measurements with and without irradiation to be made as required. The first results up to 10^{-4} dpa indicate a permanent volume degradation several orders of magnitude above the initial conductivity value. The paper will report on the rig design, development, and testing, together with the full results obtained in the present reactor irradiation cycle.

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17-47

Long Term Stability of Erbium Oxide Coatings

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A liquid lithium blanket is an attractive blanket design because of its high effectiveness, continuous operation and its compact system. However, there is a serious problem called MHD pressure drop, which is Lorentz force opposite direction to the lithium flow. To solve this problem, it is suggested to construct insulate coatings on inner pipe walls. The coatings must have high resistance to reduce MHD pressure drop, and also have high chemical stability in liquid lithium, which has high reduction activity at high temperature. Several ceramic materials were suggested from the point of chemical stability and investigated with bulk materials by exposure to liquid lithium, aluminum nitride, yttrium oxide and erbium oxide would be preferable candidate materials for the coating. In this study, erbium oxide coatings were fabricated by arc source plasma deposition, and the specimens were exposed into liquid lithium.

Specimens were erbium oxide coatings fabricated by arc source plasma deposition, at R.T. and at 700 C. The coatings were exposed at 500 to 700 C, for 100 and 1000 hours. After the immersion, residual lithium on the surface of the specimens was removed with vacuum distillation.

In the exposure of 100 hours, the specimens which were fabricated at 700 C had less damage in 500 to 700 C exposures, while the specimens which were fabricated at R.T. had more damage and many part of the coatings were peeled off in 500 to 600 C exposures. On the other hand, the specimens fabricated R.T. showed better compatibility in the exposure at 700 C. It is considered that crystallinity of the coatings fabricated at R.T. changed by annealing as that of the coatings which were fabricated at 700 C, and the specimens became more stable than that fabricated at R.T.

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17-48

Chemical Shift of Characteristic X-Ray Wavelength in Silicon-Containing Ceramics due to Neutron Irradiation

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Several ceramic materials will be applied into fusion reactors as structural and functional components to sustain fusion plasma under very severe environment such as intense radiation, high temperature, high heat load. Properties of ceramics will be influenced by irradiation of intense fast neutrons, which produce crystalline defects and transmutation products into materials. It is known that volume increase, reduction of thermal diffusivity, reduction of mechanical properties, degradation of electric properties are induced. Therefore, evaluation of defects induced into crystals or non-crystals are highly requested with use of variety of evaluation methods.

In the present study, change in peak wavelength of characteristic X-ray emitted from materials was tried to measure by so-called electron probe microanalysis. Characteristic wavelength of silicon in neutron-irradiated and non-irradiated ceramics was precisely measured to detect any change in materials induced by

neutron irradiation. The merit of this method is its applicability for materials independent of crystallinity. Wavelength reflects atomic coordination of a specific ion/atom in materials.

Wavelength of silicon K or K characteristic X-ray emitted from materials was measured by electron probe microanalysis, using WDX (Oxford Instruments, Microspec WDX-450) equipped onto a scanning electron microscopy (Hitachi, S-3500H). Accelerating voltage of electron beam was 20kV. Crystal used to disperse X-ray of silicon was PET (Pentaerythritol, $2d=2.575\text{nm}$). Specimens used in this study were neutron-irradiated and non-irradiated single crystal Si, SiO₂ glass, quartz, polycrystalline Si₃N₄ and polycrystalline SiC. Measurement was conducted successively during monitoring temperature of PET.

Detected wavelength was continuously shortened with increasing temperature of PET. Chemical shift of peaks from metallic silicon was observed for unirradiated materials, for longer wavelength in case of Si₃N₄, Quartz and SiO₂ glass. Chemical shift of SiO₂ glass due to neutron irradiation reduced but that of quartz increased compared from these of unirradiated ones. Chemical shift of SiC increased depending on irradiation condition.

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Physical Property Changes of Crystalline and Non-Crystalline SiO₂ due to Neutron Irradiation and Recovery by Subsequent Annealing

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Silicon dioxide (SiO₂) are expected to be used in fusion reactors as functional materials such as optical fibers for plasma diagnostics in non-crystalline form, and can be applied as insulators, windows in crystalline form. Many aspects on optical property change of fibers were recently mentioned, but less attention was paid for physical property change due to neutron irradiation.

In this study, physical property change of crystalline and non-crystalline forms of high purity SiO₂ due to neutron irradiation and recovery of physical properties by subsequent annealing were examined.

A set of rectangular specimen (2 x 4 x 25 mm) and disc (10 mm in diameter and 2 mm in thickness) were neutron-irradiated in the Japan Materials Testing Reactor up to a fluence of 3.0×10^{23} n/m² ($E_n > 0.1$ MeV) at irradiation temperature of 140°C, and 6.9×10^{23} n/m² at 300°C. Specimens were high purity crystalline

SiO₂ (α -quartz, SEIKO Denshi. Ltd.) and non-crystalline SiO₂ (silica glass, T4040, Toshiba Ceramics Co.). After neutron irradiation, the specimens were isochronally annealed for 1 h in vacuum from 100 to 1000°C. Macroscopic length change, thermal diffusivity change, lattice parameter change of the specimens due to neutron irradiation and due to isochronal annealing were measured.

Non-crystalline SiO₂ shrunk about 0.8% in macroscopic length after both irradiations. The length started to increase (recovery) beyond 500°C, and gradually increased with increasing annealing temperature. After 1000°C annealing, it mostly recovered. The macroscopic length of crystalline SiO₂ expanded anisotropic manner, larger expansion along a-axis than c-axis, due to the neutron irradiation. The amount of expansion was greater after the higher fluence irradiation. Macroscopic length of crystalline SiO₂ started to reduce (recovery) due to the annealing over 500°C. They were almost completely recovered at 800°C in the case of lower fluence irradiation specimen, but at 1000°C in the case of higher fluence irradiation specimen. The change in lattice parameter of the crystalline SiO₂ due to irradiation and isochronal annealing showed the same trend as that of macroscopic length change. Thermal diffusivity of specimens reduced ~10% in the case of non-crystalline SiO₂ and 60~70% in the case of crystalline SiO₂. Both cases thermal diffusivity increased by the annealing beyond 500°C, and mostly recovered after annealing at 1000°C.

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17-50

Thermally induced emf in unirradiated MI cables

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The large amount of mineral insulated (MI) cables that will be used in ITER together with the quite low voltage signals that must be carried in some cases, implies that a careful analysis of all the possible sources of noise or signal drift is of paramount importance. An example of MI cable use for a critical device is the equilibrium pick-up diagnostic coils. The coil output signal is fed to an integrator, so any variation of EMF caused by radiation and/or thermal effects is added to the real signal, and moreover can rapidly saturate the integrator if its too high.

Several candidate MI cables for use in ITER with different central conductor, sheath and insulating materials have been examined. Measurements were made for different thermal configurations without radiation. This has separation of the thermal effects from RIEMF (Radiation Induced Electromotive Force). Because thermal gradients are inherent in all irradiation set-ups, previous measurements have included both radiation and thermal effects making the results more difficult to explain.

Our results indicate that thermal EMF (TIEMF) can be larger than RIEMF when measuring the voltage along the central conductor of MI cables. It is shown that voltage maxima appear in well localized regions of each cable, indicating that some inhomogeneity is present. Results up to the maximum working temperature of MI cable will be presented together with thermal annealing of the MI cable.

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17-51

Thermal stability of neutron irradiation effects on KS4V and KU1 fused silica

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Fused silica is a key component in windows of heating systems for Magnetic Fusion reactors and in final focusing of lasers in Inertial Fusion reactors. A significant irradiation from neutrons, charge particles and X-rays can appear in those components.

In the case of ITER, KU1 and KS4V grades, provided by the Russian Federation, are being considered. That means a detailed knowledge of the behavior of these grades under irradiation is required.

In this work, the concentration of defects like NOBHC, E', POR and others in samples with different hydrogen content (KU1, KS4V and I301 grades) is measured after neutron irradiation at high doses (1020 and 1021 n/m²) using visible and infrared optical absorption and electron paramagnetic spectroscopy.

The thermal stability of the neutron irradiation induced defects is also studied. Several isochronal annealing treatments in air are done over all the different samples, which are heated up at progressively higher temperatures until a temperature of 850°C is reached. After every treatment all the samples are optically characterized by the methods mentioned before. To determine if the observed effects are due to ionic or electronic annealing, low dose X-ray irradiations are made at 450°C, 650°C and 850°C.

It will be shown that the observed thermal stability is a function of the irradiation dose and is different for the different grades studied. The effect of the X-ray irradiations upon the defects concentration, observed by optical absorption decreases when increasing the temperature.

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17-52

Anomalous Radioluminescence behaviour for KS-4V

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KS-4V is a promising material to be used in ITER for remote handling and optical diagnostics systems. One of the main limitations foreseen for optical materials is radioluminescence. Radioluminescence will occur due to intrinsic defects present in the material before irradiation and to additional defects produced during irradiation. This implies that one should expect that the radioluminescence will vary with irradiation dose and temperature. Hence in order to characterize the optical behaviour of the material it is important to perform systematic studies of the radioluminescence as a function of dose, dose rate, and temperature. KS-4V samples have been electron irradiated in the beam line of a 2 MeV Van de Graaff accelerator, and radioluminescence from 200 to 800 nm measured during irradiation. The measurements were performed at dose rates from 0.2 to 14 kGy/s, temperatures between 50 and 300 C, and doses up to 3600 MGy.

Radioluminescence for KS-4V (low OH content) and KU1 (high OH content) has been compared. KS-4V exhibits higher radioluminescence than KU1. For KU1 the main emission is due to Cerenkov effect. In contrast for KS-4V both band-like emission around 440 nm and Cerenkov emission are present from the onset of the irradiation. With dose the 440 nm band emission for KS-4V decreases indicating that some kind of radiation induced annealing or quenching is taking place. The radioluminescence behaviour for KS-4V with dose rate and temperature is also very complex with the an emission band at 530 nm appearing at high dose and temperature.

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17-53

Luminescence of Cr doped alumina induced by charged particle irradiation

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Luminescence from oxide materials induced by energetic particles can be used for detecting flux, dose and energy distribution of He ions generated by D-T reaction in a fusion reactor. Cr doped alumina (ruby) emits intense red luminescence by charged particles, and has been applied to monitoring ion beam profiles in accelerators. However, there have been few efforts to clarify quantitatively the ion-induced luminescence in ruby for utilizing an optical measurement system. In this work, we investigated the characteristics of luminescence in ruby, by systematically changing ion energy, irradiation dose and temperature under light ion irradiation.

The samples used in the present experiment were commercially available plates of Al₂O₃: 0.5wt% Cr₂O₃ and plasma-sintered pellets prepared from mixtures of Al₂O₃: 5 x 10⁻³wt%Cr₂O₃ powders. H and He ions were irradiated in a vacuum chamber with pressure of 10⁻⁵ Pa and at room temperature using a tandem accelerator and a low energy ion gun, in the flux range 4 x 10¹⁵ to 2.5 x 10¹⁶ ions/m², in the energy range 3 keV to 3 MeV. Isochronal annealing for 10min was performed in the temperature range 323 to 1273K.

For MeV energy proton irradiation, the observed luminescence yield was proportional to projected range of the incident ion rather than the total deposited energy. The results for the energy dependence of the luminescence yield suggest that the intensity didn't depend on the electronic energy imparted per unit length, but was related with the number of luminescence centers along the pass of the ion. With increasing irradiation dose, the intensity rapidly decreased to about 40% of un-irradiated sample up to a dose of 1 x 10²⁰ ions/m², and gradually decreased to about 20% at a dose of 5 x 10²⁰ ions/m². During the heat treatment, the intensity recovered at two stages of around 673K and 873K, corresponding to stages of decrease described above. On the other hand, no change was observed for the luminescence yield during the heat treatment up to 673K after irradiation by keV energy proton and He ions. We will discuss the difference of effects between nuclear and electron energy deposition on the luminescence.

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17-54

Isotopic effect on Thermal Conductivities of Silicon Single Crystal and Diamond Film

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Isotopes are generally known as labeling elements utilizing mass difference or are utilized as nuclear fuels. Recently isotope materials have been reported to improve thermal properties in semiconductor materials where phonon excitation controls the thermal flows. We have thus far developed purified ²⁸Si single crystals using floating-zone melting method and ¹²C diamond films by plasma CVD. In the present paper, thermal conductivities of these materials at temperatures from 1.5K to 300K were studied. The materials used are ²⁸Si single crystals with isotope concentration of 99.9%. The growth direction of silicon was <111>. The samples, 3mm x 5mm x 15mm were cut from the crystals for measurements of thermal conductivity. The thermal conductivity was

measured by a steady heat flow method. One end of the sample bar contacted the heat source. The thermal gradient was measured using sensors. Diamond films with average grain size of 15 μm were formed using plasma CVD from CH₄ of with ¹²C isotope abundance of 99.95%. The specimens, 3 mm x 15 mm x 100μm, for thermal conductivity measurements were cut from the films. The main crystal orientation was <111>. Thermal conductivities of Si and diamond samples with natural isotopic abundance were also examined.

Isotopically enriched silicon and diamond showed higher thermal conductivities compared to materials with natural isotope abundance. The maximum values for ²⁸Si and ¹²C diamond were 6 and 1.5 times higher than those of Si and diamond with natural isotope abundance at around 30K and 100K, respectively. The temperature dependence of thermal conductivity for isotope materials can be explained by Callaway's model.

The improvement of thermal conductivity by isotope materials will be useful for windows and semiconductors in fusion systems.

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In-situ Measurements of Optical Fibers under Gamma-ray Irradiation

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There is a big concern in relation to the use of optical fibers as light guides in fusion installations for plasma diagnostics, remote control and optical communications. We report in this paper our first results regarding the on-line evaluation of the gamma-ray induced absorption in some large diameter (400 microns) optical fibers having UV-enhanced response. The dose rate was 0.33 kGy/h, while the total dose was 34.70 kGy.

For the studied optical fibers (OH content of 600-800 ppm) we noticed:

The peak value of the optical attenuation increases with the total dose, up to the value of 21.5 dB, for the total dose of 18.2 kGy .

The peak of the optical attenuation is shifted during the irradiation from $\lambda = 237$ nm (non-irradiated) to 280 nm at the total dose of 18.2 kGy.

The optical absorption peak located at 725 nm increases slightly its value during the irradiation as compared to the non irradiated fiber (0.1 dB, for the total dose of 18.2 kGy).

In the spectral band 550 – 650 nm a very small absorption peak built up at high radiation doses (starting from 10 kGy).

A decrease of the optical attenuation of 3 dB was noticed for a recovery time of 18 h, for the total irradiation doses of 0.33 kGy ($\lambda = 245$ nm) and 2.48 kGy ($\lambda = 260$ nm).

No radioluminescence was observed because of the low dose rate of the gamma source.

We used the deconvolution of the acquired spectra to study the dynamics of the radiation induced color centers in various conditions: under gamma irradiation, during the recovery of the optical transmission at room temperature and as the optical fiber sample was subjected to both gamma irradiation and to the photobleaching effect as it is exposed to a broad band optical light source, at the following wavelengths: 215 nm (E_V colour centre); 230 nm (E_β colour centre); 240 nm ($B_{2\beta}$ colour centre); 250 nm ($B_{2\alpha}$ colour centre); 260 nm (D_0 colour centre); 280 nm (oxygen vacancy colour centre); 318 nm (for bound chlorine); 330 nm (for molecular chlorine).

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17-56

Research for High Temperature Measurement Using Fused Silica Core Optical Fiber

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In fusion reactor, diagnostic system is exposed to strong electromagnetic field and high intensity radiation. Therefore, we consider that using optical fiber as the signal transmission device and dosimeter itself will be the best candidate for the diagnostic system, because optical fiber has several advantages over usual electrical measurements, such as no electrical noises, wide optical signal band and self powered property. The effect of optical fiber in radiation environment has been researched. However, the phenomenon when wide part of optical fiber is exposed to high temperature up to 1000°C has not been researched in radiation and non-radiation fields. The purpose of this study is to measure high temperature by applying thermal radiation of optical fiber itself and heating element (optical fiber was used only for optical signal transmission in this case). The effect of optical fiber which is exposed to high temperature in both fields was observed.

Two kinds of experiment were performed. Thermal radiation spectrum of optical fiber itself and Al₂O₃ as the heat element were measured. The experiment was performed at ⁶⁰Co irradiation facility in Japan Atomic Energy Research Institute. The dose rate was 1.0 Gy/s. The optical fiber whose core diameter is 200 μm is doped fluorine and contained some ppm OH. The temperature was changed from 400°C to 1000°C by electric furnace.

It was observed that optical fiber itself emits the thermal radiation at 1240 and 1390 nm in radiation and non-radiation fields. From the results of two experimental fields, it was found that these peaks were caused by thermal radiation of OH contents. The luminescence of 1240 and 1390 nm had linear property to temperature and had gone invisible by the optical noise at 1000°C. And the optical fiber itself didn't have gamma-ray induced effect at this dose rate. The thermal radiation of Al₂O₃ was observed and reasonable to the thermal radiation theory in both fields. And the gamma-ray induced effect wasn't also occurred in this experiment. As the thermal radiation spectrum had linear property to temperature, we consider that this type of thermometer is favorable.

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17-57

The role of the fused silica stoichiometry on the intrinsic defects concentration

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Fused silica is a model material of great interest, not only because it can be considered as the reference model for amorphous materials, but also due to its increasing use in many technology areas. For thermonuclear fusion applications, it may be used in optical fibers and windows. It is also a critical element of the optical systems both in magnetic (ITER) and in inertial (NIF) confinement concepts.

Materials properties of interest (optical absorption, radioluminescence, mechanical properties, ...) are closely related to the presence of defects. Therefore a detailed knowledge of the defects properties are encouraging required. These defects may be present in the material, in many cases associated to impurities, or may be generated by irradiation.

On the other hand, simulation of radiation damage for metallic materials using classical molecular dynamics techniques have reached in recent years a situation in which it seems possible the comparison with different types of experimental data. In the case of insulating materials the availability of data (mainly spectroscopic techniques) able to provide information of point defects types and concentrations is even higher but the empirical interionic potentials required for the radiation damage simulations have not yet been fully developed. Fused silica has been one of the most studied insulating materials, being one of the interionic potentials used that proposed by Feuston and Garofallini.

In this work the defect types and their concentration as a function of stoichiometric deviations are studied using classical molecular dynamic. The Feuston-Garofallini interionic potential, currently used for radiation damage simulation in fused silica, is tested in this study by analysing the obtained defects. The defects structural properties will be characterized and compared with those obtained using other simulation techniques and with experimental data from a number of different fused silica grades. In this case, the defect types and concentration will be obtained using visible and infrared optical spectroscopy.

Preliminary results indicate that the final number and type of intrinsic defects obtained depends strongly on the stoichiometry. In particular the number of defects is minimized if a small oxygen deficiency is included before constructing the amorphous sample.

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Search for Luminescent Materials under 14 MeV Neutron Irradiation

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It is necessary to develop a novel measurement system to install a fusion reactor such as the International Thermonuclear Experimental Reactor (ITER); this is because the system is usually placed in a harsh environment such as under heavy irradiation and in a large electromagnetic field. The optical measurement system using an optical fiber and luminescent material is a strong candidate for the measurement system because of its insensitivity to electromagnetic force and radiation-induced electrical phenomena such as radiation-induced electromotive force (RIEMF). In this study, luminescent materials—important components of the optical system—that have luminescence under fast neutron irradiation, were researched.

Neutron irradiation was performed at the Fusion Neutronics Source in the Japan Atomic Energy Research Institute. The energy spectrum of the neutrons exhibits a sharp peak at 14.1 MeV and the flux was 2.6×10^9 n/cm²/s. The luminescence generated by fast neutron irradiation was detected by a photonic multichannel analyzer via an optical fiber, which covered a distance of 40 m from the irradiation area to the detector. The commercially available scintillators—ZnS:Ag and ZnS:Cu—and the long lasting phosphors (LLPs)—SrAl₂O₄:Eu²⁺, Dy³⁺ and Sr₄Al₁₄O₂₅:Eu²⁺, Dy³⁺—were irradiated for the purpose of measuring their luminescence.

All samples exhibited fast neutron-induced luminescence, and the ZnS group did not exhibit luminescence immediately after irradiation. It is considered that the neutron-induced luminescence does not arise from the product nuclide, but from the recoil nucleus and/or the prompt gamma-ray generated by the nuclear reaction between the host material and a fast neutron. Although the others possessed a good radiation resistance, the luminescence of ZnS:Ag decreased as the irradiation fluence increased. ZnS:Cu and SrAl₂O₄:Eu²⁺, Dy³⁺ maintained their respective luminescence intensities up to the fluence of 10^{14} n/cm². The luminescence characteristics of the LLPs were also examined under proton irradiation, by changing the incident energy. These compounds exhibited some luminescence peaks that can be attributed to their dopants—Dy³⁺ and Eu²⁺—and the luminescence intensity ratio of Eu²⁺ to Dy³⁺ displayed incident energy dependence. LLP is a good candidate for use as a scintillator in the optical measurement system.

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Deformation-induced Dislocation Channeling and Martensite Formation in Neutron-irradiated 316 Stainless Steel

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The plastically deformed microstructure in 316 stainless steels (316SS) after fission neutron irradiation at the range of 65-100°C to 1 dpa was investigated by transmission electron microscopy (TEM). Particular emphasis is placed on the deformation-induced dislocation channeling and martensite formation. The deformation microstructure in irradiated 316SS consisted of a mixture of dislocation bands, tangles, twins, dislocation channels, and martensite phase. Deformation-induced martensite transformation was observed at relatively higher irradiation dose (>0.1dpa) and various strain levels. In addition, martensite transformation tends to be observed on {111} planes with dislocation channeling. The resolved shear stress associated with each dislocation channel was estimated with the assumption that geometric constraint stresses due to neighboring grains were negligible. The dependence of angle between tensile axis and slip plane normal and slip direction on channels indicated a tendency for resolved shear stress and channel width to be greatest when the angle is around 45°. Furthermore, channel width increased with increasing resolved shear stress, indicating that the most extensive localized channel deformation tends to occur at a high resolved shear stress level. On the other hand, martensite formation in dislocation channels occurred even at low resolved shear stress levels. It is suggested that a very high stress could be locally generated in dislocation channels and this led to the localized martensite formation.

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17-60

The Penetration Of Tritium Through Aging Austenitic Radiation-Resistant Reactor Steel

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Radiation resistance of austenitic reactor steel is enhanced by increasing the titanium content from 0.3-0.4 (SS316 type) to ~1 wt.% (16Cr15Ni3Mo1Ti type). As a result, the steel properties change qualitatively and the steel is classified among precipitation hardening materials. The steel is hardened thank to precipitation of the Ni₃Ti γ' - phase during neutron irradiation, which is followed by several-fold decrease in the radiation swelling $\Delta V/V$ of the modified steel, as compared to ordinary austenitic steel. The 16Cr15Ni3Mo1 steel was shown to be highly resistant to swelling under irradiation with neutrons up to 60 dpa (~773 K) and krypton ions up to 200 dpa (773-973 K). Penetration tritium and deuterium was analyzed by method hydrogen permeability. Localization of tritium was examined using the method of electron microscopic autoradiography. In the 16Cr15Ni3Mo1Ti austenitic steel tritium is located at crystal lattice defects (such as dislocations, dislocation subgrains, incoherent boundaries of secondary phases, and grain boundaries with particles). Temperature dependences of the coefficients of penetration and diffusion of tritium and deuterium at temperatures from 293K to 1000K and pressure from 50Pa to 1000 Pa. It was shown that the increase in the tritium concentration disturbs the linear dependence of the diffusion coefficients and solubility on the inverse temperature. This fact has been related to appearance of heavy hydrogen isotopes traps on boundaries of titanium-containing particles, namely carbides TiC and γ' - phase Ni₃Ti.

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17-61

On the Tearing Resistance of AISI-316L – Austenitic Stainless Steel

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The AISI 316L type austenitic stainless steel was selected as a structural material for the first wall and blanket structures of the International Thermonuclear Experimental Reactor (ITER). This steel was extensively characterized using a variety of testing techniques. However, while an abundant literature can be found on a variety of mechanical properties, in comparison, the tearing resistance was less investigated, in particular in the high temperature range, namely above 300°C. This can be explained by the difficulty of using the adequate instrumentation to monitor crack growth during testing. Nowadays, such limitations were overcome and no sophisticated instrumentation is anymore needed to perform such tests.

In this work, we characterized the tearing resistance of the AISI 316L steel in a temperature range between -150°C and +550 °C. Tensile tests were also performed in the same range of test temperature. Although not directly relevant from the operational point of view, the low temperature test results are important in understanding the relationship between the ductile tearing resistance and the flow properties. Effects of the yield strength and work hardening on the initiation toughness and tearing resistance were extensively studied. Finally, dynamic loading effects were also investigated to provide the necessary elements in understanding the fracture behavior of AISI 316L stainless steel and its relation to the tensile properties. Such data will be very useful to assess the structural integrity of fusion components.

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17-62

Ageing Effect on the Properties of CuCrZr Alloy Used for the ITER HHF Components

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CuCrZr alloy is used as a heat-sink material for the high heat flux (HHF) components of ITER (first wall, limiter and divertor). The solution anneal at 980-1000°C for 1 hr, water quench, and ageing at 450-480°C for 2-4 hrs is specified as a reference heat treatment for the ITER. However, the real manufacturing cycle might be different from such "ideal" heat treatment. For example, temperatures 500-800°C and time 5-60 min are used for the amour tiles brazing. So, it is important to know the ageing kinetic of reference CuCrZr alloy, and to know the strength and ductility of this alloy after different ageing heat treatments. The paper deals with the investigation of tensile properties, hardness and structure changes due to various heat treatments.

CuCrZr alloy with the composition specified for the ITER application has been used for investigation. Material was subjected to solution anneal at the temperature 960-1000°C for 0.5-1 hr and water quenched. The variation of an additional anneal temperatures (ageing) were 350-650°C. The exposure time was varied from 3 to 120 minutes. Effect of secondary heat treatment has been studied. After fist ageing, an additional heat treatment (anneal at 650-800°C) has been applied at that. The tensile properties, hardness and microstructure have been studied after applied heat treatments.

The results have shown that during first 3-20 minutes occur the most valuable changes in strength due to age hardening. However, the CuCrZr alloy is not very sensitive to the overageing. Strength and ductility remains at a relatively high level even after anneal at 650°C, that allows exploiting the advantages of high temperature manufacturing process.

Temperature-time diagrams of properties changes have been plotted. This gives the guideline for the selection of manufacturing heat treatment and prediction of the strength and ductility of CuCrZr alloy with applied manufacturing cycle.

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17-63

Dislocation structure in deformed irradiated single crystal Ni

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It is well known that plastic deformation of irradiated materials can be localized in so-called defect free channels, which are at the origin of premature failure. These channels maintain a relatively constant width of 100 nm and a separation of about 1 µm, which are independent on the materials. There is however still a lack of understanding on channels formation mechanism. Experimental transmission electron microscopy observations were performed in single crystal Ni irradiated with 590 MeV protons to low dose at room temperature and deformed in uniaxial traction to failure. Samples were observed with the {111} channels both in planar view and in cross sectional view. In planar view, the nature and morphology of dislocations remaining in the channels could be identified. The amount of rotation conveyed by each channel was measured by convergent beam electron diffraction pattern. From these results the origin and evolution of defect free channels are discussed.

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17-64

Effects of Water Chemistry on Stress Corrosion Cracking Behavior of Unirradiated and Irradiated Type 316LN-IG

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Type 316LN-IG stainless steel is one of the structural materials of a vacuum vessel and in-vessel components for ITER. The vacuum vessel and in-vessel components are exposed to high heat flux loading due to high energy neutron and cooled by high temperature waters. In such service conditions, austenitic stainless steels become susceptible to irradiation assisted stress corrosion cracking (IASCC). Because the components will be used at wide temperature range, authors have studied the IASCC susceptibility of type 316(LN) stainless steels in oxygenated water at temperature range from 333 to 673 K. It was clarified by authors that if the irradiation temperature of the stainless steels was below 603K, the IASCC susceptibility was disappeared below 513K in oxygenated water. The purpose of this study is to evaluate the effects of water chemistry and stress concentration on the IASCC susceptibility.

Tensile specimens were prepared from type 316LN-IG stainless steel joints those were prepared by hot-isostatic pressing method. The specimens were irradiated at nominal temperature of 473K to about 1 dpa in High Flux Isotope Reactor (HFIR). Some specimens had a single-edged notch to make a stress

concentration. The slow strain rate test was performed in an oxygenated water (dissolved oxygen=10ppm) and a hydrogenated water (dissolved hydrogen=1.3ppm) in order to evaluate the effects on the IASCC susceptibility.

For irradiated specimens, both intergranular (IG) and transgranular (TG) stress corrosion cracking (SCC) occurred in oxygenated water at 573K, but did not in hydrogenated water. The hydrogen addition to water was beneficial to suppress the IASCC susceptibility at 573 K. At 513K, IASCC did not occur in oxygenated water even in a single-edged notch specimen. However, TGSCC was observed at a notch root in the hydrogenated water. Therefore, the water chemistry had a different effect on the IASCC susceptibility at lower temperature.

The effects of water chemistry on SCC behavior of unirradiated specimens were also studied at 603 K. TGSCC mainly occurred in both oxygenated and hydrogenated water. In the oxygenated water, cracks initiated at plastic strain of about 10%, but the specimen was ruptured by TGSCC at the engineering strain of about 50% due to slow propagation rate of the TGSCC. Large necking was observed on the specimen. In hydrogenated water, on the other hand, crack initiation seemed to occur during elastic deformation, but specimen elongated to about 40% and then ruptured due to TGSCC. Necking was not observed on the specimen tested in the hydrogenated water. Many cracking were observed on the fracture surface of the specimen. Water chemistry affected the crack initiation behavior of unirradiated materials at 603K.

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Low-temperature Embrittlement of Austenitic Steel Examined using Ring-pull Tensile Tests and Microhardness Measurements

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The prediction of mechanical property changes during fusion neutron irradiation requires that suitable surrogate data be obtained at relevant environmental conditions of temperature, dpa rate and neutron spectra. Often compromises must be made to obtain such data, involving the use of surrogate spectra, small specimen technology and property-property correlations. Although the neutron spectra of fusion devices are not available, important parameters such as appropriate He/dpa ratio can be attained in light water reactor spectra for austenitic steels as a consequence of the two-step $^{58}\text{Ni}(n, \gamma)^{59}\text{Ni}(n, \alpha)$ sequence. Light water reactors also involve temperatures in the vicinity of 300°C that are typical of water-cooled ITER conditions. Therefore we have explored the mechanical property changes of an austenitic thin-walled instrument tube removed from a VVER-1000 pressurized water reactor. This tube was formed from annealed 06X18H10T steel (analog of AISI 321) and spanned temperatures of 285-315°C and doses of 1-9 dpa, which produced significant embrittlement.

Mechanical properties were measured at room temperature and elevated temperatures using rings cut from the tubes and a ring-pull tensile test frequently used in the countries of the former Soviet Union. Also used were Vickers microhardness measurements performed on the ring sections at room temperature. Transmission electron microscopy was also performed on rings prior to deformation, showing that the primary damage structure was that of Frank loops and unresolvable black dots. The changes in mechanical properties were easily correlated to the black spot distribution using a standard model.

Comparing the results using a universal correlation between changes in yield stress and microhardness recently compiled by Busby and coworkers, it was found that the results of this current study agreed well with the correlation in the hardness range covered by Busby, but beyond this range the correlation broke down completely, indicating that new factors begin to dominate the behavior at higher hardness levels characteristic of such low temperature irradiation. This divergence in behavior is discussed in terms of deformation-induced martensite formation associated with such low-temperature irradiation and its dependence on the deformation modes peculiar to each test, as well as to some peculiarities associated with the ring-pull tensile test.

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Atomic-scale modeling of primary damage in copper

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Atomistic modeling was conducted for an investigation of primary damage creation, self-interstitial and vacancy clusters formation, their stability and interaction in high energy displacement cascades in copper. The simulations were carried out for a wide range of temperatures ($100\text{K} \leq T \leq 900\text{K}$) and primary knock-on atom (PKA) energies $5 \text{ keV} \leq E_{pka} \leq 25 \text{ keV}$. This study of over 400 cascades is the largest yet reported for this metal. From 20 to 50 cascades for each (E_{pka} , T) pair were simulated in order to ensure statistical reliability of the results. The number of surviving point defects for each cascade and the mean value for cascades at the same temperature and PKA energy were found. The corresponding fraction of self-interstitial atoms (SIA) in dislocation loops and vacancies in stacking fault tetrahedron (SFT)-like clusters was calculated. The mean sizes of SIA and vacancy clusters and cluster per cascade yield were evaluated. Strong spatial and size correlation of SFTs and SIA clusters at low temperatures was established.

In the context of high dose irradiation and the spatial overlap of displacement cascades, the stability of SFTs and SIA dislocation loops inside an overlapping cascade region was investigated. It was observed that an SFT destroyed in the collision phase by a cascade is always recreated. On being completely enveloped by the region of displaced atoms, both SFT and SIA dislocation loop are destroyed with corresponding decrease of the number of residual point defects, whereas partial overlapping leads to increase in size of both.

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17-67

In-situ SCC Observation on Neutron Irradiated Thermally-sensitized Austenitic Stainless Steel

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In the design activity of international thermonuclear experimental reactor (ITER), irradiation assisted stress corrosion cracking (IASCC) was concerned as one of the specific problems for water-cooled first wall/blanket components. To examine behavior of the crack initiation and propagation of IASCC, in-situ observations on the surface of specimen were conducted during slow strain rate tests (SSRT) in high temperature water. Type 304 stainless steel (SS) of commercial grade was subjected to a solution heat treatment and a thermal sensitization treatment. Specimens were irradiated at neutron fluence from 1.0×10^{25} to 10^{26} n/m² in the

Japan Materials Testing Reactor (JMTR). After neutron irradiation, SSRT for the specimens was conducted in oxygenated high purity water at 561 K. The surface of the specimen was observed through a window equipped on an autoclave during the SSRT. Fractured surface was examined using a scanning electron microscope (SEM) after the SSRT. For the specimens irradiated to 1.0×10^{26} n/m², a total elongation of irradiated solution annealed SS was smaller than that of irradiated thermally sensitized SS. Using in-situ observations, it was shown that one or two cracks were introduced at the gage length when the stress reached to the maximum strength on stress-strain curve and the cracks propagated until fracture. Total elongation of irradiated solution annealed SS was larger than that of irradiated thermally sensitized SS at neutron fluence of 1.0×10^{25} n/m². Crack propagation rate after the crack initiation is analyzed at present.

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Demonstration of the Separate Temperature and dpa Rate Dependencies of Void Swelling in 300 Series Stainless Steels

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The prediction of swelling-induced strains produced in austenitic steels in fission and fusion environments requires not only a sufficient amount of data but also a clear understanding of the parametric dependencies of various variables that are often strongly synergistic. Recently, a variety of experiments have indicated that the dependence of swelling on dpa rate has been strongly underestimated, with the dpa rate dependence frequently but inadvertently incorporated into the temperature dependence. This inability to separate the separate dependencies of dpa rate and temperature is closely associated with the coupling of neutron flux-spectra and temperature along the axial length of components, especially for relatively small cores such as EBR-II. Complicating the separation process are the often disparate rate dependencies of void/dislocation evolution and that of radiation-assisted precipitation of elements which influence void nucleation. Such complications make it difficult to develop swelling correlations which can be confidently extrapolated to new combinations of dpa rate and temperature.

In order to demonstrate the separate action of dpa rate and temperature, swelling data are presented from hexagonal ducts irradiated in EBR-II, with both axial and radial gradients in dpa rate. Annealed AISI 304 ducts were chosen to avoid complications of precipitation found in other alloys, and its interaction with cold-work. Since this steel never develops multiple-peak swelling behavior and very

little precipitation at high dpa rates, it is ideal for separation of environmental variables. Use of ducts without fuel inside also allows a reduction in through-wall temperature gradients and minimizes temperature histories associated with fuel burn-up, both of which are known to impact the swelling behavior. Comparisons of swelling behavior on various faces of a given duct also rule out the action of other variables that are time-dependent.

It is shown that the combined effect of dpa rate and temperature distribution along one face any given structural component produces a well-defined, scatter-free "swelling loop" vs. dpa that uniquely allows estimation and separation of the separate dependencies of swelling on temperature and dpa rate. Comparison of the swelling loops on various faces of the same duct also allows very clear identification of the effect of dpa rate and its synergisms with temperature. One consequence of the derived flux dependence is that components subject to a dpa rate gradient in general suffer much less distortion than predicted by equations that do not explicitly incorporate a dependence on dpa rate. It is also shown that over a wide range of irradiation conditions the terminal steady-state swelling rate of AISI 304 is consistent with the ~1%/dpa characteristic of austenitic stainless steels. The insights developed in this effort are now being applied to more complex alloys envisioned as candidates for fusion first-wall application.

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17-69

Preliminary Study on Thick Alumina Coating Fabricated by SHS Method for Fusion Application

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Coatings have been proposed as the solution to critical materials constraints for most of the blanket concepts under development for fusion power applications. Al₂O₃ coating has been developed in several laboratories as (1) electrically insulating coatings to mitigate magnetohydrodynamic (MHD) effects in self-cooled/dual-cooled liquid metal systems, (2) tritium barriers within the blanket system to reduce tritium permeation, (3) tritium containment to reduce tritium release to the environments, (4) corrosion barriers to permit higher temperature operation, (5) helium containment to reduce helium leakage into the plasma chamber.

In this contribution a Thick Alumina Coating (TAC) is designed to improve the reliability, corrosion resistance and permeation resistance. Self-propagation High-temperature Synthesis (SHS) method is used to fabricate TAC on iron and LAM (Low Activation Martensitic steel) base. The properties of TAC have been tested to make sure that the TAC is well bond with the base material and is suitable for application in fusion liquid metal cooling system. Test results such as density, heat expansive coefficient, heat conductive, hardness and SEM, XRD, EDS results etc. are presented. Details on SHS method are presented as well.

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17-70

Preliminary Experiment on Corrosion behavior of CLAM in Liquid LiPb Eutectic

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The design activities of FDS series fusion reactors with liquid tritium breeder blankets have been performed at ASIPP (Institute of Plasma Physics, Chinese Academy of Sciences) for years. In the designs, CLAM (China Low Activation Martensitic steel), which is one of the RAFMs (Reduced Activation Ferritic/Martensitic steels) and under development in ASIPP, is considered as the Primary candidate structural material, liquid LiPb eutectic as both tritium breeder and coolant of the blankets, and Al₂O₃ ceramic as a candidate coating on CLAM to reduce the corrosion of CLAM in liquid LiPb etc.. Therefore, corrosion behaviour of CLAM and its coating in LiPb is one of key issues in these systems. So a thermal convection LiPb loop has been built at ASIPP.

The preliminary experiments on corrosion of samples of CLAM and CLAM with Al₂O₃ ceramic coating in still and flowing liquid LiPb have been done in the thermal convection loop,. The samples were examined after keeping in liquid LiPb at the temperature of 480°C for 1000hr. Changes in sample surface morphology as well as in specimen weight were observed. Concentration profiles of the compositions near the sample surfaces were examined by EDX line-scan and point analyses in order to analyze the corrosion mechanism of liquid LiPb to CLAM and its coating.

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17-71

Residual Stresses in Vapor-Deposited Erbium Coatings

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The electron-beam physical vapor deposition (EBPVD) of erbium coatings is pursued for use in a magneto-hydro dynamic environment as a resistive layer to reduce the pressure drops. In general, the morphology and stability of oxide coatings evaporated from stoichiometric targets are dependent upon the electron-beam power and the substrate temperature. Too high a target power density can lead to an oxygen deficiency and too low a substrate temperature to an under-dense porous coating. Process conditions are now developed and evaluated to determine effects on the residual stress state and microstructure of the erbium coatings as deposited onto sapphire wafers. The curvature of the coated substrate is used to compute the residual stress state. Initial findings indicate that the erbium coatings in a highly-compressive stress state. Often, residual compressive stress is considered beneficial for adhesion. However, too large a magnitude of compressive stress can lead to decohesion of the coating from its substrate. A local minimum in stress is found coincident with a process temperature of 600 °C. Variation in the crystallographic texture of the erbium deposits with substrate temperature and an assessment of forming the equilibrium phase is made using x-ray diffraction. Also, the microstructural variations with deposition temperature seen in fracture cross-sections are examined using scanning electron microscopy. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

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17-72

Material Probe Study on Boronized Wall of LHD

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In the Large Helical Device (LHD), boronization using glow discharge with a mixture gas of helium and diborane was conducted in 6th experimental campaign. In this campaign, material probes made by silicon and 316L stainless steel were placed at the plasma facing wall along toroidal direction. The depth profiles of atomic composition and retained amounts of discharge gases in these probes were examined by Auger electron spectroscopy (AES) and thermal desorption spectroscopy (TDS), respectively. The surface morphologies of the probes were also examined by scanning electron microscope (SEM).

The toroidal distribution of the boron film thickness significantly depended on the positions of the anodes and the diborane-inlet nozzles. The thickness of boron film and the atomic concentration of boron at the wall close to the anode and the nozzle were 400 nm and 80 at%, respectively. At the wall far from the anodes and the nozzles, the boron film was very thin, only several nm. The trapped amount of oxygen increased with the thickness of boron film. The retained amount of helium at the wall close to the anodes was one order of magnitude larger than that far from the anodes. The retained amount of hydrogen at the wall with a thick boron film was observed to be smaller than that at the wall with a thin boron film.

Further material probe analysis will be conducted for the 7th and 8th experimental campaigns.

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17-73

Charging of V-4Cr-4Ti by Oxygen to create *in-situ* Insulator Coating

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The self-cooled Li/V-alloy blanket concept is being thought very attractive. One of the critical issues of it is large magneto-hydrodynamic pressure drop when Li flows in the blanket duct under magnetic field. A promising solution is to apply an electrically insulating coating on the inner wall of the ducts. *In-situ* oxide insulating coatings (Er₂O₃, CaO, Y₂O₃) are under development to the self-cooled Li/V fusion blanket. In the beginning, the main attention was given to research of thermodynamic stability and electrical-insulating properties of mentioned compounds in lithium. Then its feasibility of formation and functioning by exposing V-4Cr-4Ti-O in liquid Li doped with activity metal (Er, Ca or Y) were proved. However not only the thermodynamic properties of oxides, but also kinetics of oxygen delivery from solid metal to the interface determines the durability of insulator coating.

In this stage, kinetics and peculiarities of structure formation in V-4Cr-4Ti under the charging procedure by oxygen in the Ar+O₂ mixture at 973-1273 K were investigated. The value of activation energy was determined to predetermine the mechanism of interaction between V-alloy and oxygen in Ar+O₂. At the critical temperature ~1011 K, the mechanism of interaction is changed. At lower temperature (T < 1011 K) the oxidation process is limited by diffusion through surface oxides. The growth of oxidized zone strongly obeys the parabolic law. At higher temperature (T > 1011 K) the oxygen absorption by vanadium matrix dominates.

The obtained results were compared to data of other researchers. Based on comparative analysis the diffusion-deformation model of formation of inner oxidizing zone in the V-4Ti-4Cr at lower temperature has been developed. The preferred oxygen diffusion along grain boundaries and defects accumulation in the grains are taken into account in this model. Within the frame of this model the morphology of coherent Ti-O precipitates and mechanism of their formation are explained. Inverse problem – discharging of oxygen-containing alloy in Li-Er melt is considered.

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17-74

Compatibility of Multi-Layer, Electrically Insulating Coatings For the Vanadium-Lithium Blanket

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Electrically insulating coatings on the first wall of magnetic confinement reactors are essential to reduce the magnetohydrodynamic (MHD) force that would otherwise inhibit the flow of the lithium coolant. Current work is focusing on multi-layer coatings or a flow channel insert as it is unlikely that a single-layer coating will be sufficiently durable to meet the lifetime goals for this application. A metallic vanadium or vanadium alloy layer would prevent Li from interacting with the underlying ceramic insulating layer. A flow channel insert could be made using vanadium foil on the outside surfaces and a ceramic coating on the inside. This strategy switches the compatibility concern from the ceramic layer to vanadium.

As a first step in confirming the compatibility between vanadium and Li, static capsule tests of V-4Cr-4Ti in Li were conducted at 800°C using Mo capsules. After 1,000h, the specimens showed small mass gains that were partially attributed to mass transfer from the Mo capsule to the V-4Cr-4Ti specimens. After the test, the concentration of V, Cr and Ti in the Li was <3ppmw indicating a low solubility of these elements at this temperature.

Initial fabrication work have produced two layer physical vapor deposited coatings of erbia and vanadium. These coatings are being tested in lithium capsule tests as well as in an in-situ test rig for measuring the coating resistance with liquid Li in contact with the coating surface.

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Correlation of Yield Stress and Microhardness in 08Cr16Ni11Mo3 Irradiated to High Dose in the BN-350 Fast Reactor

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The yield strength $\sigma_{0.2}$ is a basic parameter used in engineering calculations and its proper determination is necessary for design of fission or fusion reactors. For fusion applications, however, data generated in the appropriate spectra are not available, so data from surrogate spectra are required to validate the measurement technique and to allow extrapolation to the target spectra.

Even in surrogate spectra it is not always possible, however, to determine $\sigma_{0.2}$ on highly irradiated material using direct techniques such as uniaxial tensile tests, particularly when there are large levels of induced radioactivity. It can also be difficult when the material volume is either too small to produce a tensile or other mechanical property specimen, when the material of interest is in an inconvenient location or configuration, or when significant gradients in mechanical properties are anticipated over small dimensions. The latter might arise where there are strong local gradients in temperature or neutron flux across the material of interest.

In the present work the quantities $\sigma_{0.2}$ and H_{μ} are measured and compared for Russian austenitic stainless steel designated 08Cr16Ni11Mo3 (analog of AISI 316) which were cut from three reflector ducts in the BN-350 fast reactor, with doses as large as 15.6 dpa at ITER-relevant temperatures of 280-337°C. The irradiation proceeded at rather low displacement rates of 0.8×10^{-10} to 4.8×10^{-7} dpa/sec. Tensile and Vickers microhardness tests of both unirradiated and irradiated specimens were performed at room temperature. While tensile specimens of uniform cross-section were cut from the duct faces, microhardness tests were made on both face and corner specimens of more inconvenient geometry. Values of $\sigma_{0.2}$ ranged from 230 to 1010 MPa and values of H_{μ} ranged from 150 to 435 kg/mm².

The correlation between $\sigma_{0.2}$ and H_{μ} for the current steel (see fig. 2) can be described by the following relation: $H_{\mu} \sim k_1 \cdot \sigma_{0.2} + \sigma_1$, where $k_1 = 2.85$ and $\sigma_1 = -171$, with units of MPa and kg/mm², respectively. This result is in excellent agreement with that of Toloczko et al. for unirradiated 316 cold-worked to various levels, where $H_{\mu} = 2.7 \sigma_{0.2} - 125$. Even more importantly, when compared with the universal correlation $\Delta H_{\mu} \sim 3.03 \cdot \Delta \sigma_{0.2}$, developed by Busby, there was excellent agreement.

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17-76

A Semi-automated Small Specimen Fracture Testing Instrument

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A semi-automated device for fracture tests on small pre-cracked bend bars is described. Nine bend bars with allowable dimensions ranging from $\approx 1.65 \times 1.65 \times 8$ mm to $3.3 \times 3.3 \times 18$ mm are loaded in a testing cartridge. The test device consists of: a) an indexed specimen cartridge positioning stage; b) two types of test fixtures; c) stations for dynamic and static loading; d) a station for easy specimen insertion; e) a gas blanket temperature control system; and f) a load-time-displacement instrumentation and data acquisition system. One station is for dynamic loading from ≈ 30 to 3000 mm/s. It consists of a large, high-speed stepper motor driving an instrumented TUP, either on a lever arm, or on a CAM actuated linear loading pin. Two types of specimen fixtures can be used here. In one, the specimen is vertical and the loading resembles an IZOD impact test. After insertion, the specimen is clamped at the bottom end and the center of the side directly opposite the pre-crack is butted on a single support pin. The top-front end of the specimen is then impacted by a TUP mounted on the sweeping lever arm, golf club fashion, with the load reacting on the center pin. In the second configuration, the specimen is horizontally inserted into a three or four-point bend fixture. In this case, the motor actuates a CAM driven linear loading pin. For quasi-static tests, a hydraulic cylinder actuates the lever arm which loads the specimen in a three or four point bend fixture. Specimen displacement is monitored continuously. Temperature is established and maintained by a gas blanket with thermocouple controller actuating a LN_2 tank valve or a heat gun, augmented by heat transfer channels in each of the specimen locations. Other pertinent details of the system are described along with an extensive calibration-validation finite element modeling and database.

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17-77

New microscopic specimen testing method using a bending of micro cantilever specimen

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Successful development of small specimen test technology (SSTT) is a key to effective utilization of intense fusion neutron source, e.g. IFMIF. One of the key requirements for SSTT is to establish quantitative correlation with standard specimens, and this demands detailed understanding of plastic flow and crack initiation/propagation processes in specimens with different geometries. In the present work, a new microscopic specimen testing method using a bending of micro cantilever specimen is proposed. This method can extract mechanical properties of each single crystal from polycrystalline materials.

Vanadium and molybdenum were selected to model fusion structural and high heat flux material. These metals are also interesting since they are known to show very significant orientation dependence in deformation properties. Both materials were annealed at 950°C for 2h to obtain well-grown crystal grains. For preparation of thin bending test specimens ($< 10 \mu\text{m}$), 3mm diameter TEM discs were punched from a sheet material, followed by electro polishing with twin-jets method using tenupol-3. Focused Ion Beam (FIB) processing was carried out to obtain a cantilever shape suitable for micro bending tests. All specimen size were $10 \times 50 \times X$ ($X: 1-15 \mu\text{m}$). The thickness of the cantilever was measured from the SIM images of the FIB. The bend tests were performed using a nano-indenter (CSIRO UMIS). Initial contact load was 0.01mN, and then load was increased in a stepwise manner up to 15mN while measuring the displacement. The maximum load was changed according to specimen thickness.

Load initially increased proportionally with increasing displacement, and then yielding is observed. Yield load increased with increasing cantilever thickness. Maximum stress calculated at the surface of the cantilever was from 500 to 2000MPa for molybdenum and from 100 to 500MPa for vanadium. Yield stress of the specimens was comparable or rather higher than that the yield stress obtained by conventional tensile tests using bulk specimens. Possible reasons for the difference are orientation difference, FIB processing damage to the specimen, error in measuring specimen thickness.

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17-78

Miniaturized Fracture Stress Tests for Thin-Walled Tubular SiC Specimens

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To evaluate the fracture stress of coatings in nuclear fuel particles, two miniaturized testing methods have been developed for thin tubular specimens. In the first method the hoop strength of thin-walled tubular specimen is obtained by internal pressurization using an elastomeric insert (polyurethane), which is compressed by two metal pistons. The second method is a crush testing technique, or a diametrical compressive loading technique. An elastic stress analysis of the first test method showed that internal pressurization produced a relatively homogeneous stress distribution with a small radial gradient through the wall thickness, while diametrical loading produced large radial and circumferential stress gradients. Fracture stress was defined by the maximum hoop stress at fracture, which might occur at the weakest point on the inner surface in the internal pressurization test and on the inner surface area, just below the loading contact, during diametrical loading. Test specimens were tubular CVD SiC specimens with about 1 mm inner diameter, 90 µm thickness, and 5.8 mm length, which were obtained from surrogate nuclear fuels. Test results showed that the two loading methods produced similar mean values, although the variability was larger for diametrical loading. In addition, the testing methods were used to evaluate radiation effects in the CVD SiC specimens and size effects in the alumina tubes, and the results from these applications are discussed.

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17-79

An Application of the Gurson's Model to Small Scale Fracture-Mechanical Specimens

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The aim of the present work is to find a convenient material model for a fracture mechanical (FM) experiment, in particular for the Three-Point-Bending (3PB) or C(T) experiment performed for variously sized samples. Such a model should yield correct results (e.g. crack-tip fields, J-integrals or crack resistance curves) obtained by finite element method (FEM) simulations independent of the sample size and geometry i.e. should be based on the micromechanics. Among a variety of different damage models, the celebrated Gurson's model enhanced by Tvergaard and Needleman (GTN) has been chosen for this role.

The FEM code ABAQUS used for the simulations contains two versions of the Gurson's model: a simplified version within the ABAQUS/STANDARD and an enhanced version within the ABAQUS/EXPLICIT including an accelerated loss of the stress carrying capacity beginning at a given critical voids volume fraction (VVF) and a final VVF when the material fails completely. This resent version allows contrary to the 1st model a simulation of a crack growth by definition of the final VVF. However, a use of the ABAQUS/EXPLICIT is not straightforward. For this reason, a user-defined material routine (UMAT) implemented by Dr. F. Reusch has been applied within the ABAQUS/STANDARD package.

Unknown damage parameters have been adjusted by simulation of verification tensile tests as well as FM experiments. The obtained material parameters have been then applied to a modelling of FM tests using variously sized probes of the same type and simulation results have been compared with the curves obtained experimentally. Furthermore, a possibility to predict the behaviour of a standard-sized FM probe on the basis of results obtained by a testing of a small scale specimen is considered in the present work.

Additionally, advantages and drawbacks of an application of a gradient-enhanced Gurson's damage model already implemented as a UMAT are also discussed here.

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17-80

Master Curve Evaluation of Transition Fracture Toughness of a JLF-1 Reduced-Activation Ferritic Steel

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Small specimen test techniques especially for evaluating fracture toughness of fusion blanket structural materials are effective to reduce the specimen volume for the IFMIF irradiation experiments. The specimens used for measuring a valid fracture toughness have been relatively large for materials showing high fracture toughness and medium tensile strength such as reduced-activation ferritic (RAF) steels.

In the present study, fracture toughness in the transition temperature regime of JLF-1 RAF steels was measured for different small sizes of compact tension (CT) specimens with applying the master curve methodology.

The fracture toughness tests were carried out in accordance with the ASTM E1921-02. Three sizes of the CT specimens, 1CT, 1/2CT and 1/4CT, were prepared to characterize the transition fracture toughness in the T-L orientation. Over-tempered (tempered at a temperate above A_{c1}) and quenched JLF-1 steels were also prepared. The over-tempered JLF-1 showed a higher tensile strength and a lower elongation which was similar to the irradiated materials. The shift of the reference temperature T_0 (ΔT_0) due to the over-tempering was compared with that of ductile brittle transition temperature ($\Delta DBTT$) obtained by Charpy impact tests.

The K_{Jc} values evaluated for the 1/2CT specimens were qualified as valid values in the transition temperature regime. The T_0 of the as-received and over-tempered JLF-1 steels were -52°C and 26°C , respectively. The $\Delta DBTT$ of the JLF-1 steel due to the over-tempering heat treatment was measured to be 84°C that is almost same as the ΔT_0 obtained by the fracture toughness tests. The effect of specimen size on the master curve methodology is also discussed.

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17-81

Fracture Behaviors of F82H Steel for Small Size Specimens

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Small specimen test technology (SSTT) has been developed to investigate mechanical properties of nuclear materials. SSTT has been driven by limited availability of effective irradiation volumes in test reactors and accelerator-based neutron and charged particle sources.

In this study, a new bend test machine has been developed to obtain fracture toughness and DBTT (ductile-brittle transition temperature) of F82H (Fe-8Cr-2W-0.3V-0.04Ta-0.1C-0.002N) steel for small bend specimens of t/2-1/3PCCVN (pre-cracked 1/3 size Charpy V-notch) with 20 mm length and DFMB (deformation and fracture mini bend specimen) with 9 mm length. The ratio (a/W) of the crack length (a) to the specimen width (W) for the DFMB and t/2-1/3PCCVN was controlled within a range from about 0.40 to 0.45. The depths of V-notch or U-notch and the lengths of the pre-crack for t/2-1/3PCCVN and DFMB specimens were precisely measured by an optical microscope before bending tests. The bending tests using a new machine can be performed at temperatures from -196°C to 300°C under unloading compliance method, and the displacement rates of cross head can be changed from 0.01 mm/min to 100 mm/min.

In this study, DBTT of F82H steel for t/2-1/3PCCVN, DFMB and 0.18DCT specimens was evaluated from the changes of fracture toughness as a function of temperature. DBTT of F82H steel for CVN, 1/3CVN and t/2-1/3CVN specimens was also evaluated from the changes of absorbed energy as a function of temperature by using an impact test machine. DBTT of smaller size specimens of t/2-1/3PCCVN and DFMB was lower than that of larger size specimen of 0.18DCT, and DBTT of t/2-1/3CVN specimen was also lower than that of 1/3CVN and t/2-CVN specimens.

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High temperature FIMEC tests on NFR materials

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FIMEC (Flat-top cylinder Indenter for MEchanical Characterization) is an indentation technique employing cylindrical punches with diameters ranging from 0.5 to 1 mm. The test gives load-penetration curves from which yield stress and elasticity modulus are determined. From FIMEC tests carried out at different temperatures indications about DBTT (ductile to brittle transition temperature) can be also drawn. For its features FIMEC test can be usefully employed to measure either the mechanical properties of irradiated materials or those of narrow material's region, like the heat-affected or the molten zone of a welded joint.

The FIMEC machine developed by us and initially presented at ICFRM-8, allowed to test samples in a temperature field ranging from -180 °C to +200 °C. Recently, the heating system of FIMEC apparatus has been modified to operate up to 600°C. So, in addition to get the trends of yield stress and elasticity modulus versus temperature, it is now possible to perform stress-relaxation (or creep) tests in a temperature range of great interest for several NFR (Nuclear Fusion Reactor) candidate alloys.

In this work data regarding fusion-relevant and other structural materials (MANET, EM-10, Eurofer-97, F82H mod., AISI 316 and Ti6Al4V) are presented and compared with those obtained by standard mechanical tests.

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Small Fracture-toughness Specimen for Post-irradiation Experiments: Validation and Results

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A new specimen type for fracture-mechanics tests as well as the associated testing devices have been developed and applied to examinations. Due to its small dimensions and the selected geometry, the three-point bend specimen is suitable for irradiation programs with limited space. New hot cell devices for testing of irradiated small-scale specimens have been installed at Karlsruhe's Fusion Materials Laboratory and serve for all kind of post-irradiation experiments (PIE) and metallographic examinations.

The non-conservative results which are to be feared by the use of small specimens are prevented by a geometrical modification of the fracture zone and the development of a suitable test and evaluation technique. Experimental and computational validation in comparison to standard samples is presented. The analysis of the stress state in front of the crack tip, based on finite element calculations, suggests constraints by lateral notches to effectuate more uniformly distributed stresses and J-integral values under mode-I load.

Isothermal experiments are performed at the three fundamental material conditions ductile, brittle, and ductile-to-brittle transition using different specimen sizes, geometries, and materials. The shift of the fracture toughness ductile-to-brittle transition - in comparison to full-scale specimens - is examined in tempered experiments. It is shown that non-conservative results can be excluded. EUROFER 97 shows for all specimen sizes and geometries clearly better results in fracture toughness than the alloys examined before – as well in absolute values, as in the ductile-to-brittle-transition.

The practical usefulness of the developed specimen type for PIE is demonstrated by examining irradiated ferritic-martensitic steel specimens. The evaluation of samples irradiated up to 0.8 dpa at 250 – 450°C supplies reproducible values for J and K, and complete J-R-curves can be derived. Fracture toughness is strongly reduced by up to 60% by irradiation, and the ductile-to-brittle transition temperature is augmented up to 220°C. The decrease in fracture toughness is dependent on the irradiation temperature, whereas it is worst at temperatures below 350°C.

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17-84

A Universal Relationship Between Hardness and Flow Stress

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A new and powerful indentation hardness (H) approach to evaluating the true stress (σ)-true plastic strain (ϵ), $\sigma(\epsilon)$, constitutive behavior of materials is described. Since measurements of H intrinsically probe a wide-range of ϵ (≤ 0.5), accurate assessment of the yield (σ_y) stress and strain hardening [$\sigma_{sh}(\epsilon)$] pose a significant challenge. Extensive elastic-plastic finite element (FE) simulations were used to assess the relation between H and $\sigma(\epsilon)$. The simulations were based on both a wide variety of analytical $\sigma(\epsilon)$ relations, $\sigma(\epsilon) = \sigma_y + \sigma_{sh}(\epsilon)$, as well as actual $\sigma(\epsilon)$ derived from data on a large number of alloys with a very wide range of constitutive behavior. The analysis led to a remarkable *universal relation* between H and $\sigma(\epsilon)$ given by

$$H \approx 4.05(1 + 34.6\sigma_{flow}/E)\sigma_{flow}, \text{ where } \sigma_{flow} = \sigma_y + \langle\sigma_{sh}\rangle,$$

where $\langle\sigma_{sh}\rangle$ is the average strain hardening between $\epsilon = 0$ and 10%, and E is the elastic modulus. Note we use consistent MKS units of MPa for both H and σ_{flow} . The expression for $H(\sigma_{flow})$ also can be inverted to one describing $\sigma_{flow}(H)$. Experimental σ_{flow} -H data pairs based on this definition of σ_{flow} for the large set of alloys noted above with a very diverse range of $\sigma(\epsilon)$ are in excellent agreement with the model predictions. The σ_{flow} -H relation provides insight into the large variation of the H/σ_y ratios that are observed for different materials, as well as the corresponding variation in the $\Delta H/\Delta\sigma_y$ factor used to estimate $\Delta\sigma_y$ due to irradiation based on measurements of ΔH . We can also use this relation to evaluate $\langle\sigma_{sh}\rangle$ in irradiated alloys that have very low uniform strain capacity using a combination of H and σ_y measurements. Finally we show how this relation can be used with data from hardness transverses across bend beams to evaluate $\sigma(\epsilon)$ high ϵ .

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17-86

The Effects of Specimen Size on the Cleavage Fracture Toughness of Eurofer97: A Model Based Analysis

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Eurofer97 is a precursor to the primary candidate martensitic steel for European ITER test blanket modules (TBMs). Use of ferritic-martensitic alloys requires adequate defect tolerant margins to protect against fast fracture. A Master Curve (MC) method is being developed to provide a highly efficient means of acquiring and applying fracture toughness information. The MC method enables the use of small to very small specimens required in irradiation experiments. The method assumes that a master toughness-temperature curve, $K_{Jc}(T-T_0)$, exists that can be indexed to an absolute temperature scale by a reference temperature T_0 . Neutron irradiation elevates T_0 (embrittlement). It is also known that other factors such as specimen size and geometry have a strong influence on T_0 . Such effects must be accounted for in measuring toughness with small specimens and application of toughness data to assessing failure limits of TBM structures. Size dependence derives from both the statistical and constraint loss effects. It has been previously shown that critical-stress/critical stressed volume micromechanical local fracture in low alloy pressure vessel steels and a martensitic steel F82H, that is similar to Eurofer97, can be used to treat size effects. Here we present an analysis of 96 measurements of the K_{Jc} at -142°C using 3 point bending taken from the middle of a single plate of Eurofer97 with independent variation specimen dimensions designed to systematically probe statistical and constraint loss related size effects. A critical-stress/critical stressed volume model is used to adjust the data to an intrinsic small scale yielding toughness for a reference specimen thickness of 25.4 mm. The resulting $T_0 = -113 \pm 3.8^\circ\text{C}$ is compared to other evaluations and the significance of size scaling to small specimen testing as well as the fracture safe limits of fusion structures is briefly discussed.

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17-89

Manufacturing of W/Cu Mock-ups for Plasma Facing Components

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A manufacturing study has been carried out for W tile bonded structures employed in plasma-facing components. For W tile bonded to the Cu-alloy heat sink with a cooling pipe, a one axis hot press has been used to bond the materials. The FGM inter-layers of Cu-Ni-Mn-Si brazing materials have been used for bonding the W tile to the Cu-alloy heat sink. Using the above bonding techniques, partial mockups for plasma facing components have been successfully manufactured. Because of the heat processes using a conventional hot press, the manufacturing cost can be minimized.

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17-90

Effects of helium and hydrogen implantation on damage during pulse high heat loading in tungsten

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The major part of ITER divertor armor is foreseen to be made of tungsten plates. Armor erosion and damage caused by pulse high heat loading such as disruptions and edge localized modes(ELMs) are critical issues for a good performance of the tokamak. The armor is also subject to high flux particle(hydrogen isotopes, helium) from the plasma. In particular, it is well known that helium implanted in tungsten does not release until high temperatures due to strong interaction with lattice defects. Helium drastically enhances the formation of bubbles due to the strong bonding to vacancies and their clusters. Therefore, it is expected that implanted helium/hydrogen isotope may influence behavior on tungsten during pulse high heat load. In the present work, helium/hydrogen isotope pre-implanted tungsten has been exposed by pulse electron beams to examine synergistic effects of helium/hydrogen isotope implantation and pulse high heat loading.

Tungsten samples used in the present experiment are stress relieved and re-crystallized powder metallurgy tungsten. The samples are repeatedly irradiated by hydrogen or helium beam (~20keV, ~2.5x10²¹/m²s, ~3s) up to a fluence of the orders between 10²² - 10²⁴ /m² using the Particle Beam Engineering Facility (PBEF). After the helium or hydrogen irradiation, the samples are exposed to pulse high heat loading by an electron beam(~1GW/m², 1~2 ms) using the Electron Beam Irradiation Stand(JEBIS). Before and after the irradiations, surface modification is examined with a scanning electron microscope(SEM) and a scanning probe microscope(SPM). In addition, hardness testing is performed to examine degradation of mechanical properties. Weight loss is also measured.

It is shown that surface modification, crack formation and weight loss due to pulse high heat loading on tungsten implanted with helium or hydrogen are different from that of tungsten, which is not implanted. Particularly, in the case of the sample implanted with helium beam, blisters and small holes are formed due to electron beam pulse high heat loading in the area around the melted and re-solidified part. Present results indicate the importance of synergistic effects of helium or hydrogen implantation, and pulse high heat loading on surface modification and erosion of tungsten under the fusion environment.

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17-91

CFC/Cu Joints for ITER Divertor: New Developments*M.Ferraris¹, M.Salvo¹, V.Casalegno¹, M.Merola²*

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The high-heat flux (HHF) components in the divertor of ITER (International Thermonuclear Experimental Reactor) consists of a water-cooled copper heat sink with CFC (carbon fibre reinforced carbon matrix composites) and W tiles. A successful method of joining CFC to copper was developed for the flat-type configuration; this study shows the feasibility of the process also for monoblock geometry and illustrates some possible mechanical shear tests for monoblock configuration.

Materials with significant different properties like CFC and copper alloy are very difficult to join. High residual thermal stresses and the reduction of the joint thermo-mechanical properties are consequences of the large thermal expansion mismatch between CFC and the copper alloy. In order to reduce the stress at the joint interface, a pure copper interlayer between the heat sink and the armour is mandatory. Besides, molten copper shows a very high contact angle on CFC

surfaces ($\theta=140^\circ$). The joining method described in this paper consists of proprietary composite surface modification by solid state reaction with VIB group transition metals and the following casting of pure copper on the substrate.

With regard to CFC/Cu monoblock design, good results were achieved by the use of a slight pressure during the joining processes and by the optimisation of some different sample-holders.

An extensive set of different mechanical tests was performed on CFC/Cu joint flat tile. Moreover, "ad hoc" mechanical characterization techniques were set up for monoblock samples, whose mechanical resistance is not easily measured by usual mechanical tests.

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Development of A Helium-Cooled Divertor: Material Choice, Technological Studies, and Proof Of Principle

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A modular He-cooled divertor concept [1] is being pursued at the Forschungszentrum Karlsruhe. The design goal is to achieve a high heat flux of about 10-15 MW/m². The reference design option is based on the jet impingement cooling technique. The concept is based on the use of small tiles of tungsten as sacrificial layer which is brazed to a finger-like (thimble) structure made of W-alloy. The brazing joint helps stop the crack growth induced from the plasma-facing surface of the tile. The inner surface under each thimble is cooled by 10 MPa He at 600°C that is provided by a jet cartridge which is placed concentrically inside the thimble. Another back-up solution based on the use of a tungsten flow promoter in the form of a slot array to enhance the cooling surface is also under investigation. The cooling finger unit is connected to the supporting structure made of oxide dispersion-strengthened (ODS) steel (e.g. an advanced ODS EUROFER or a ferrite version of it) by means of e.g. brazing and/or mechanical interlock. This transition joint has to survive about 100 – 1000 thermal cycles between operating and room temperatures.

The development and optimisation of the divertor concepts require an iterative design approach with analyses, studies of materials and fabrication technologies, and the execution of experiments. The choice of material and fabrication methods often undergoes a trade-off between material properties and economic aspects, which has an impact on the design. The choice of tungsten as most promising divertor material is based on its excellent sputtering resistance and thermal conductivity. On the other hand, it possesses poor DBTT and RCT values, as a result of which the operation temperature window of the divertor is restricted. Promising methods for line production of divertor components from tungsten are electrochemical milling (ECM) and powder injection moulding (PIM) which are being investigated at the moment. At Efremov, technological studies and experiments are performed with respect to e.g. the joining of the W tile to the W alloy thimble and joining of the W thimble to the steel structure by means of high-temperature brazing. Good results were obtained for the mock-ups brazed with the following filling metal alloys 71KHCP (Co-base, 5.8Fe, 12.4Ni, 6.7Si, 3.8B, 0.1Mn, P?0.015, S?0.015, C?0.08), brazing temperature (T_{br}) = 1100°C and STEMET 1311 (Ni-base, 16.0Co, 5.0Fe, 4.0Si, 4.0B, 0.4Cr), T_{br} = 1050°C. In HHF tests, these mock-ups survived up to 14 MW/m² at least. Studies of alternative structural materials e.g. CVD tungsten, single-crystal tungsten, and Densimet are underway. In this paper, the results shall be discussed and the strategy of out-of-pile HHF tests shall be outlined.

[1] P. Norajitra et al., "The European Development of He-cooled Divertors for Fusion Power Plants", Proceedings of the 20th IAEA Fusion Energy Conference, Portugal, 2004.

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17-93

Features of Plasma Sprayed Beryllium Armor for the ITER First Wall

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Two water-cooled mockups with CuCrZr heat sinks and plasma-sprayed beryllium (PS Be) armor, 5 and 10 mm thick respectively, were fabricated at Los Alamo and tested at Sandia at 1 and 2 MW/m² for multiple cycles. These mockups differ from earlier PS Be mockups in two ways. First, in these mockups, castellations in the surface of the heat sinks provide mechanical locking. Previous armor was sprayed over a wider area and the "wings" that were subject to peeling at the edges were cut away. Second, earlier PS Be mockups had high density coatings obtained by keeping the substrate temperature high during deposition. The somewhat porous quality of the PS Be armor in the mockups recently tested is the direct result of a low (600-650°C) substrate temperature during deposition that was specifically requested in PS Be mockups made for the European Fusion Development Agreement (EFDA). Keeping the temperature of a CuCrZr heat sink low can be desirable to avoid a subsequent heat treatment step to recover fully the properties of precipitation-hardened CuCrZr. The mockups tested at Sandia are similar to mockups fabricated for EFDA, although the pattern of the castellations is somewhat different. Among the features observed in the post-test examinations reported here was beneficial transverse cracking along preferred paths that relieve thermal stresses. The control of cracking that occur during cyclic high heat flux tests is a result of the morphology of Be coatings applied over castellations in the heat sink. Some melting of the armor was observed and expected because these coatings had relatively low thermal conductivity. Based on comparison of the test results with thermal modeling, we estimate the thermal conductivity of these coatings as about ¼ that of fully dense beryllium. For possible first wall applications in ITER, PS Be armor with a density and thermal conductivity below that of fully dense material might still be acceptable since the wall loadings are lower than high heat flux applications such as the divertor. The results of parametric thermal modeling in the paper show this trade-off and provide a guide for further development of the PS Be armor.

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17-94

Methods of Elastic Modulus Determination for Irradiated Porous Tungsten Coatings

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Plasma-sprayed tungsten coating, which is a candidate material for first wall armour, shows a porous microstructure. Due to such morphology, properties of this coating are significantly different from those of the dense material. The aim of this work is to measure the elastic modulus of plasma-sprayed thick tungsten coating deposited on steel (F82H) substrate. To this end, a tungsten coating/steel substrate system was fabricated using the vacuum plasma-spray technique. After machining and polishing of the specimen the coating thickness was reduced to 1.62 mm and the substrate thickness to 1.35 mm.

Both 3-point and 4-point bending tests were applied for the measurement. In the case of the 4-point bending test, displacements were measured by two different strain sensors, that is, contact extensometer and laser speckle interferometer. To confine the maximum stress of the specimens within the elastic regime, the residual stress was estimated using the theoretical method developed by Tsui and Clyne. The computed residual stress at the surface ranged from 31 to 64 MPa, which were comparable to the experimental stress measured by the X-ray diffractometer. The measured surface stress ranged from 12 to 68 MPa. The shape of the specimen curvature, which was concave on the coating side, clearly supported the theoretical result.

The Young's modulus was determined from the measured force-deflection curves using two different approaches. At first, the Young's modulus of the coating was directly extracted by means of rigorous mathematical formulations. Alternatively, the coating modulus was obtained simply by the rule of mixture. Finally, the coating layer was detached from the substrate and tested again. The experiments were performed for three different specimen sizes.

The measured modulus values ranged from 53 to 57 GPa. The modulus of the detached coating strip was 54 GPa. The applied methods yielded consistent modulus values regardless of the testing configuration and specimen size. The testing errors and the dimensional error were less than 1 %. The obtained modulus values correspond to 14 % of the elastic modulus of dense tungsten (400 GPa). This behaviour was attributed to the porous microstructure and micro-cracks formed by poor bonding between splats.

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17-95

Silicon Doped Carbon/Cu Joints Based on Amorphous Alloy Brazing for First Wall Application

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The limitation on using silver-based alloys for brazing in-vessel components of fusion experimental reactor created a development problem for new brazing materials. Amorphous ribbon-type filler metals represent a promising selection for joining heterogeneous materials together the advantage results from the homogeneity of elements and phase compositions and the strictly specified geometrical dimensions of such fillers.

In this paper, rapidly solidified ribbon-type Ti based amorphous filler with a melting temperature of 850°C and a thickness up to 20 µm was used to join silicon doped carbon to copper. According to finite element analysis, very thin Mo foil and Cu foil were selected as middle layer to mitigate the thermal stress between carbon and copper. SEM examinations demonstrated the high quality of brazed joints. The brazed seam has a uniform structure along its entire length. The shear strength test shown that the shear strength of this carbon-copper joint is more than 25Mpa, and the rupture was mainly occurred on the carbon side. The thermal shock resistance is tested and the prospects of these joints for fusion reactor applications are discussed.

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17-96

Effects of Chemical Status of Carbon on Deuterium retention in Carbon Related Materials

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Graphite is one of the most candidate materials for plasma facing materials and has been studied extensively for interaction with hydrogen isotopes. In fusion reactors, however, carbon with various chemical structures could exist especially in re-deposition and co-deposition layers. It is, therefore, an important issue to reveal the effects of chemical states of carbon on deuterium retention in carbon related materials.

Highly oriented pyrolytic graphite (HOPG), polycrystalline diamond, polycrystalline SiC and sintered WC were employed as the samples. They were preheated to remove the impurities and residual gases such as water. The deuterium ions implantation and thereafter thermal desorption spectroscopic (TDS) experiments were performed. The X-ray photoelectron spectroscopic (XPS) analysis was also done after each of the processes. The dependence of implantation temperature on the deuterium desorption behavior in HOPG was also investigated as the underlying interaction between carbon and deuterium.

The dependence of temperature during deuterium implantation in HOPG indicates that one of the two deuterium trapping states could be introduced by a non-equilibrium reaction, so-called hot atom reaction, and the other by thermochemical equilibrium reactions. It is found that the former reaction is dominant in polycrystalline diamond and polycrystalline SiC, in contrast to that the latter is dominant in sintered WC, regarding those trapped by carbon atom. The deuterium trapping states, therefore, would be affected by chemical structure of carbon related materials.

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17-97

Hydrogen isotope behavior in the first wall of JT-60U after DD discharge

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The elucidation of hydrogen isotope behavior in carbon materials for fusion reactor is one of the most critical issues for fusion safety. We have extensively studied hydrogen retention characteristics in JT-60U divertor area and found that the most of the inner divertor area was covered by thick co-deposited layers with maximum thickness of around 100 μ m and the co-deposited layers retained deuterium uniformly throughout the layers. On the other hand, the outer divertor area was erosion dominated and rather high hydrogen (H+D) retention was found only in very near surface region less than 0.5 μ m. Therefore, total hydrogen retention in the divertor area of JT-60U is much less than that observed in other tokamaks like JET and TFTR.

Until now, however, hydrogen retention on the first wall was not examined. This motivates us to examine the first wall tiles for erosion/deposition and hydrogen retention. Four first wall tiles used in JT-60U during DD discharge were taken and analyzed by secondary ion mass spectrometry and thermal desorption spectroscopy to obtain the depth profiles of hydrogen isotopes and characteristics of hydrogen isotope retention, respectively. Erosion and deposition profiles were examined by cross sectional views of a scanning electron microprobe.

The tile surface was very smooth without appreciable erosion and significant amount of boron was found. Deuterium retention was strongly correlated to the boron amount. In TDS analysis, the desorption temperature of deuterium for the first wall samples was appreciably lower compared to that for the divertor samples on which more or less boron was found. All these lead us to conclude that most surface area of the first wall tiles analyzed here remained boron and deuterium originated from boronization occasionally done for the reduction of oxygen impurities in JT-60U plasmas with using decaborane (B₁₀D₁₄) gas. Therefore, deuterium retention owing to plasma exposure would be very small. In addition, temperatures of the first wall tiles was much lower than that for divertor tiles and should not result in the significant loss of deuterium in the boron layer.

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17-98

Manufacturing of a small scale CFC armored monoblock mockup by hot radial pressing

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ENEA is involved in the European International Thermonuclear Experimental Reactor (ITER) R&D activities and in particular for the manufacturing of high heat flux plasma-facing components (HHFC), such as the divertor targets, the baffles and the limiters: During the last years ENEA has manufactured actively cooled mock-ups by using different technologies, namely brazing, diffusion bonding and hot isostatic pressing (HIPping). A new manufacturing process has been set up and tested. It was successfully applied for the manufacturing of W armoured monoblock mockups. This technique is the HRP (Hot Radial Pressing) based on performing a radial diffusion bonding between the cooling tube and the armour

tile by pressurizing only the internal tube and by keeping the joining zone in vacuum and at the required bonding temperature. The heating is obtained by a standard air furnace.

This paper reports the research path followed to manufacture a small scale CFC armoured mockup. An ad hoc rig able to maintain the CFC in a compressive constant condition was also designed and tested. The casting of a soft copper interlayer between the tube and the tile was performed by a new technique: the Pre-Brazed Casting (PBC, ENEA patent).

A mock-up with three NB31 CFC tiles was successfully manufactured and it was tested in the TSEFEY-M electron beam facility at St. Petersburg (Russia). It reached 1000 cycles at 25 MW/m² without suffering any damage.

This activity was performed in the frame of an IETR-EFDA contract.

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17-99

Damage Evaluation Under Thermal Fatigue of a Vertical Target Full Scale Component for ITER Divertor

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An extensive development programme has been carried out in the EU on high heat flux components within the ITER project. In this framework, a Full Scale Vertical Target (VTFS) prototype was manufactured with all the main features of the corresponding ITER divertor design. The prototype consisted of four units having a full monoblock geometry. The armour upper part of the prototype was made of an alloy of tungsten (W-1%La₂O₃) lamellae whereas the lower part was made of Carbon Fibre reinforced Carbon (CFC-NB31). Each monoblock was joined using AMC[®] technology to the solution annealed, quenched and cold worked CuCrZr tube by HIP technique. One of the most critical manufacturing step of such a component is the armour to heat sink joint quality upon which mainly depend the heat removal capabilities and the thermal fatigue performances. A high heat flux testing campaign dedicated to such issues, based on fatigue cycling and lifetime on CFC and W armoured regions, was performed on FE200 electron beam facility.

The CFC monoblock was successfully tested up to 1000 cycles at 23 MW/m² without any indication of failure. The W monoblock endured ~600 cycles at 10 MW/m². Fatigue damage, resulting in water leakage, was observed when pursuing the cycling up to 15 MW/m².

However, these results of fatigue testing proved the capability of a such component to meet the ITER requirements in terms of heat flux performances for the vertical target.

Metallographic examination, performed on a damaged unit of the prototype, showed some initiation of cracks within the CFC distributed along the bond. As a consequence, this local thermal resistivity leads to an increase of surface temperature then to a damage of the CFC by erosion and cracking of the matrix. Nevertheless, the integrity of the component is not affected. On the other hand, the cracks detected in W-1%La₂O₃ are mainly oriented perpendicular to the surface. These cracks can become locally critical because they penetrate in depth the heat sink material

This paper describes the fatigue behaviour observed during HHF tests of the VTFS component. Thermal and mechanical stress finite element analysis performed in order to investigate the fatigue life time and the fracture behaviour of the joint is also discussed.

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17-100

Material Damage due to Edge Localized Modes in ITER

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Plasma facing components in next step fusion devices such as ITER will be subjected to intense quasi-stationary thermal loads $\leq 10 \text{ MWm}^{-2}$. During so-called slow transients the peak heat flux in the divertor strike zone may double for a limited number of discharges and a duration up to approx. 10 s. In addition, significant energy deposition up to several ten MJm^{-2} will appear during vertical displacement events and plasma disruptions; the duration of these events are fractions of a second down to the millisecond range.

The existing database on the material and component performance under extremely short (sub-millisecond) events which are associated with the occurrence of Edge Localized Modes (so-called type I ELMs) during normal plasma operation is still fragmentary. In next step fusion devices ELM events are expected to occur with energy densities of $\approx 1 \text{ MJm}^{-2}$ and a duration of $\approx 0.5 \text{ ms}$ duration. The frequency of these ELMs will be $\approx 1 \text{ Hz}$; i.e. several millions of these events will be accumulated during the lifetime of the divertor targets. To simulate transient thermal loads with pulse durations in the sub-millisecond region, powerful plasma accelerator test stands turned out to provide the most relevant loading scenarios with respect to incident species, particle energies and loaded area. However, high-power electron beam test facilities are an important tool for the evaluation of the component performance under repeated ELM events.

Two test devices (JUDITH 1 and 2) represent qualified test beds capable to generate local power densities up to 10 GWm^{-2} . These electron beam test devices can be operated either in a static mode to simulate ELM-specific thermal loads on localized spots of the test specimens (beam diameter = 1 and 5 mm FWHM, resp.); in a beam scanning mode larger surfaces (several square centimeters) are exposed with ELM-relevant heat pulses; these tests are performed on polycrystalline tungsten and multidirectional carbon fiber composites. The electron beam high heat flux tests are completed by numerical methods.

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17-101

Dependence of oxygen concentration on chemical behavior of deuterium implanted into oxygen contained boron thin film

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In fusion devices, boronization is considered to reduce the impurities, especially oxygen, in plasma. Additionally, energetic tritium, deuterium and helium will be implanted into boron deposited on the plasma facing wall. Therefore, hydrogen isotopes retention and its chemical behavior in oxygen contaminated boron thin film should be studied as a function of oxygen concentration.

The oxygen contaminated boron thin film samples were prepared by PCVD device. The flow rates of oxygen were changed from without oxygen contamination to 4 sccm and these of helium and decaborane were set to be 3.8 sccm and 2.5 sccm, respectively. Thickness of the sample was about 150 nm. After heating treatment, 1 keV deuterium ions were implanted into the samples at room temperature. The ion flux and fluence were set to be $1.0 \times 10^{18} \text{ D}^+ \text{ m}^{-2} \text{ s}^{-1}$ and $7.4 \times 10^{21} \text{ D}^+ \text{ m}^{-2}$, respectively. After deuterium ion implantation, X-ray photoelectron spectroscopy (XPS) and thermal desorption spectroscopy (TDS) were applied to elucidate the chemical state of boron and estimate the deuterium retention.

It was found that the peak top energy of B-1s by XPS was shifted to high-energy side by the deuterium ion irradiation. Large peak shift was observed for the sample with the oxygen flow rate of 4 sccm compared to that without oxygen contamination. For the sample with high oxygen flow rate, the different chemical state was observed in high binding energy side. For TDS spectra, three deuterium desorption stages were observed for all the samples. In this presentation, detailed discussion will be done for the dependence of oxygen concentration on chemical behavior of deuterium implanted into oxygen contained boron thin film.

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17-102

Recent Progress of Plasma Sprayed Tungsten Coated Carbon and Copper

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Plasma sprayed tungsten coating is one of the candidate plasma facing materials of ITER. A good compatibility of tungsten with fusion plasma has been identified in ASDEX-Upgrade. In China, HL-2A tokamak (main chamber came from ASDEX) with closed divertor has been constructed and primary plasma discharges with divertor configuration have been performed. Meanwhile the experiments of plasma sprayed tungsten coating as plasma facing materials have been scheduled and will be performed in the next campaign of HL-2A. In the present work, two kinds of tungsten coating have been prepared by vacuum plasma spray (VPS). One is VPS-W/C (SMF 700 graphite) with multi-layer tungsten and silicon interface pre-deposited by physical vapor deposition (PVD) and the thickness of multi-layered W, Si interface is 10 μm . The other is VPS-W/Cu (CuCrZr alloy) with functionally graded interface. The thickness of tungsten coating is 300-500 μm . Firstly, the principal thermo-mechanical properties of the coating, such as porosity, thermal conductivity, bonding strength, were measured. Then its heat load limit and thermal fatigue properties were carried out in a 60 keV electron beam facility under the modified load conditions of HL-2A (heat flux: 10-30 MW/m^2 , pulse duration: 3-5 seconds).

On the other hand, rhenium as diffusion barrier of carbon is a good established technology for tungsten coated carbon and relatively good high heat flux properties of VPS-W coated CFC (CX-2002U) have been obtained [1]. The comparison of the present W-Si coating with W-Re coating has also been done. Results indicated that although the heat load limit and thermal fatigue assistance probability of the VPS-W/C with Si interlayer and VPS-W/Cu are a little lower than that of VPS-W/CFC with Re interlayer, both of them can meet the requirements of HL-2A.

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17-103

Simulation of Redeposition Patterns in the Gaps between Divertor Tiles

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Chemical erosion of carbon materials is a crucial issue from view points on the lifetime and tritium retention of divertor tiles in magnetic confined fusion devices. Hydrocarbon molecules produced by chemical erosion re-deposit on the tile or the other components after migration in edge plasmas. Recent surface analyses of the used tiles have revealed thick deposited layers with high tritium concentration on the gaps between the tiles. In this study, we attract our attention on the asymmetric patterns of hydrocarbons deposited in the gaps in the toroidal and poloidal directions.

A simple model of toroidal and poloidal gaps with different widths and depths is used for simulation. A homogeneous hydrogen plasma with the temperatures of 1-30 eV and the densities of 10^{18} - 10^{21}m^{-3} , contacts the divertor tile surface. Hydrocarbons, CH_4 , C_2H_2 , C_2H_4 , C_2H_6 and C_3H_6 , are released on the tiles. They exhibit significantly complicated processes. Assuming only the collisions with the plasma, the rate coefficients of four reaction types (electron and proton impact ionizations, electron impact dissociation and dissociative recombination) were taken into account. The effects of plasma flow velocity and the electric fields parallel and perpendicular to magnetic field lines are considered in addition to the gyromotion of ionized particles.

At high plasma temperature (>tens of eV), the redeposition of hydrocarbons is dominated by ionized fragments which are produced through the electron-impact ionisations including dissociation. Therefore, an asymmetry is calculated between toroidal and poloidal gaps in dependent of the magnetic geometry and there is no redeposition at the bottom and the inner part of the gap. At low plasma temperature (a few eV), the patterns are drastically change due to the dominant redeposition of neutral hydrocarbon species, which are produced by dissociative recombination. The redeposition occurs deeply inside the gap and so no asymmetry is obtained. Since the inner part of the gap is not contacted by the plasma, the redeposited hydrocarbons remain for a long time without the re-erosion by the impact of plasma ions. As a result, successive redeposition forms a thick hydrocarbon layer in the gap, as observed in present large tokamaks.

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W-macrobrush melt damage simulation after multiple transient events in ITER

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Tungsten in form of macrobrush structure is foreseen as one of two candidate materials for the ITER divertor. During intense transient events in ITER such as disruptions and bursts of the Edge Localized Mode (ELM) a part of confined plasma is dumped onto the divertor armour, which may result in a surface melting and further evaporation. Melt motion in the thin layer may produce surface roughness and droplet splashing thus causing erosion of the target. An initial assessment of the damage to the macrobrush structure under typical ELM heat loads has been carried out using modified code MEMOS-1.5D [1]. This study has showed that fine details of macrobrush structure may play a major role in determination of damage profile.

During one ITER discharge about 10^3 ELMs are expected, and during ITER operation several hundred disruptions, interspaced by ELMs may occur. The separatrix strike position (SSP) at the divertor plate can vary from one transient event to another one, which may lead to a noticeable decrease of the total damage of macrobrush structure [2].

For W macrobrush divertor armour the results of fluid dynamics simulation of the melt motion erosion after repetitive heat loads caused by multiple disruptions with the energy deposition Q of 10-30 MJ/m² and the duration τ of 1-10 ms interspaced by multiple ELMs with $Q=1-3$ MJ/m² and $\tau=0.1-0.5$ ms are presented. For different single disruptions and ELMs, the heat loads at the divertor surface are calculated using the two-dimensional MHD code FOREV-2D. The target melt motion erosion is calculated by the fluid dynamics code MEMOS-1.5D in the 'shallow water' approximation, with the surface tension and viscosity of molten metal as well as the Lorentz forces due to the currents crossing the melt layer taken into account. Optimization of macrobrush geometry to minimize erosion under ITER-like transient loads is done.

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IASCC Behavior of the Highly Irradiated Stainless Steel at relatively low temperature, 323K at JMTR

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Irradiation assisted stress corrosion cracking (IASCC) is supposed as one of the specific problems for structural materials applied to the international thermonuclear experimental reactor (ITER). However, an amount of experimental data is not sufficient to investigate IASCC behavior of austenitic stainless steels irradiated up to high neutron fluence around relatively low temperatures expected for ITER. We, therefore, examined the IASCC susceptibility and mechanical property on stainless steel specimens irradiated at 323K in coolant water of the Japan Materials Testing Reactor (JMTR).

At the hot laboratory of JMTR, the tensile test and slow strain rate test (SSRT) were carried out using specimens which were obtained by punching from a basket type irradiation capsule made of type 304 stainless steel. The irradiation capsule used in the fuel region of JMTR for approximately 25 years was exposed up to the fast neutron fluence of $1.0-3.9 \times 10^{26}$ n/m² ($E > 1$ MeV) at 323K. Tensile tests were performed at RT, 423K and 561K in the air. Elongations of 28%, 15% and 11% on the specimens supposed to 3.9×10^{26} n/m² were obtained at RT, 423K and 561K, respectively. The SSRT was performed in oxygenated high purity water at 423K and 533K. Applied strain rate was 1.7×10^{-7} s⁻¹. From the results of scanning electron microscope (SEM) examination after the SSRT, the intergranular stress corrosion cracking (IGSCC) was observed on the fracture surface of the specimen tested at 533K. From these data, a possibility of IASCC around ITER temperatures will be discussed based on the temperature dependence of IASCC susceptibility.

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17-106

Dynamics of deuterium implanted in boron coating film for wall conditioning

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In first walls of fusion reactors, boronization is considered to be one of the most useful wall conditioning techniques for reduction of impurities in plasma. The first wall will be exposed to some energetic particles (especially tritium). Therefore it is important for the tritium safety of fusion reactors to elucidate the interaction mechanism of hydrogen isotope implanted into boron coating film. It has already reported that B-D and B-D-B bonds are formed in the boron coating films after D₂⁺ ion implantation. However, studies on dynamics of hydrogen isotopes in boron coating films have been limited so far.

In the present study, the depth profile of deuterium in the boron coating film was investigated by secondary ion mass spectroscopy (SIMS) with various sample temperatures and behavior of hydrogen isotope was discussed. The boron coating film was deposited on a silicon substrate by plasma chemical vapor deposition method and its characterization was performed by X-ray photoelectron spectroscopy (XPS). After pre-heating treatment, 1 keV D₂⁺ ions were implanted into the boron coating film and depth profile was measured by SIMS. The sample exposed to DD discharge in JT-60U was also used for comparison. The sample was heated isochronally and the depth profiles of deuterium were measured at each temperature.

It was found by SIMS that implanted deuterium ions were retained in the depth of 24 nm from the surface and it was migrated toward the surface by heating. From the peak depth of deuterium and its FWHM, the recombination rate was determined and hydrogen diffusion behavior in the boron coating film was also discussed with comparing that in the sample exposed to DD discharge in JT-60U.

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17-107

In-pile Testing of the ITER First Wall Mock-Ups at Relevant Thermal Loading Conditions

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The paper describes experimental technique and some preliminary results of thermal fatigue testing of the water-cooled ITER PFW mock-ups inside channel of the experimental fission reactor RBT-6 (RIAR, Dimitrovgrad, Russia)

This experiment provides simultaneous effect of neutron fluence and thermal gradient based damages on tested mock-ups and it has higher correspondence to real PFC operation conditions in comparison with prevailing tests where neutron and thermal fatigue factors are simulated in series.

The experimental in-pile assembly contains two water-cooled first wall mock-ups with dimensions of 114(L)×56×56 mm³, armored with two beryllium tiles (h=10 mm) each. One of these mock-ups is manufactured by EFDA team with application of the HIP technology and the second one is made in Efremov Institute by method of CuCrZr/SS casting with posterior fast brazing of the armoring tiles. The flat ohmic graphite heater, operated by PC-controlled power supply system is used for cyclic thermal loading of the armored mock-ups surfaces. Transfer of heat from heater to the mock-ups is provided by radiant heat exchange (approx. 70%) and by thermal conductivity of the protective helium atmosphere through a gap of 2.5 mm (approx. 30%). The nominal heat flux onto mock-ups is 0.5 MW/m².

The presented experiment (currently still going on) is planned for 200 days of irradiation and finally it should provide 0.6 dpa damage level in the mock-ups (CuCrZr) with simultaneous accumulation of 20 000 thermal cycles onto beryllium armor.

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Parallel Oral Session 18A – Radiation Effects in FCC Metals

In-Reactor Deformation Behaviour of Copper and CuCrZr Alloy: Experiments, Results and Implications

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Traditionally, effects of neutron irradiation on mechanical properties of metals and alloys have been studied using post-irradiation experiments. These experiments have established that the neutron irradiation at temperatures below the recovery stage V causes an increase in the yield strength but at the same time induces the phenomenon of yield drop leading to plastic instability. Furthermore, the work hardening ability is reduced to practically zero and the uniform elongation (i.e., the ductility) is also reduced drastically. The possibility of the loss of work hardening ability and a drastic reduction in ductility has given rise to a serious concern regarding the mechanical performance and lifetime of materials employed in structural components of a fusion reactor. Since this concern is based entirely on the results of post-irradiation experiments it is very important to ask the question as to whether or not the results and conclusions of

post-irradiation experiments are at all representative of deformation behaviour of materials exposed concurrently to neutron irradiation and stresses in a reactor environment. The reason for raising this issue is that there are some fundamental differences both in the microstructural state and the test conditions between the post-irradiation and in-reactor deformation experiments. It is therefore possible that the deformation behaviour under these two different sets of conditions may be significantly different.

In the present work, first of all, the major differences in experimental conditions and the dynamics of microstructural evolution between the in-reactor and the post-irradiation deformation experiments will be considered. Consequences of these differences on the dislocation-defect interactions and ensuing effects on dislocation velocity and thus on mechanical response of the materials under these two different testing conditions will be outlined. This will be followed by a brief description of different in-reactor deformation experiments that have already been carried out as well as those that are in progress. Some results of in-reactor tensile tests obtained on pure copper and CuCrZr alloy and creep-fatigue tests on CuCrZr alloy will be presented and will be compared and contrasted with the results of post-irradiation tests. Finally, the implications of the fundamentally different microstructural evolution and mechanical response resulting under these two different testing conditions will be briefly discussed.

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18A.2

Disappearance of the Dose Rate Effect on Swelling Upon C Addition to Fe-Cr-Ni-Ti Alloys

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An earlier report in this conference series showed that the atomic displacement rate was the primary determinant of the duration of the transient regime of swelling in ternary model alloy Fe-15Cr-16Ni irradiated at ~400°C. The experiment was conducted in FFTF-MOTA at seven dpa rates covering two orders of magnitude. As the dpa rate decreased the duration of the transient regime decreased in almost direct proportion, but the post-transient swelling rate was unaffected, with the rate remaining at ~1%/dpa. The origin of this flux effect on transient duration was shown to arise primarily from the flux dependence of Frank loop formation and the associated stability of the loop ensemble against unfauling and dislocation network formation.

This earlier reported experiment was part of a much larger program now having reached completion, having examined the influence on swelling of titanium addition, carbon addition and boron-doping, the latter to simulate the effect of increasing He/dpa ratio. In agreement with earlier studies, titanium addition of 0.25% at ~400°C had almost no influence on total void swelling, but it did influence the details of Frank loop and void formation. The effect of dpa rate on swelling in the quaternary alloy was also very strong and was essentially identical to that of the ternary alloy.

However, as shown in the current report, carbon addition at 0.05% to the Ti-modified alloy not only reduced swelling at all seven dpa rates, but caused the strong effect of displacement rate to completely disappear. The terminal swelling rate of 1%/dpa was also not reached in the carbon-added alloy. A similar disappearance of the flux effect was seen in the Fe-15Cr-16Ni-0.25Ti-500 appm B alloy, which suffered decreases in swelling both from boron and carbon. Once again the swelling rate never approached 1%/dpa. The microstructural reasons for this change in behavior with carbon addition are discussed.

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18A.3

Effect of Heat Treatments on Precipitate Microstructure and Mechanical Properties of a CuCrZr Alloy

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Currently, the precipitation hardened CuCrZr alloys are being seriously evaluated for their use in the first wall and divertor components of ITER. Experiments have demonstrated, however, that these alloys when irradiated with neutrons in the prime aged conditions at temperatures below about 473K become harder but lose their ability to work harden and suffer from loss of ductility and plastic instability. In an effort to improve the ductility of these alloys, it was decided to coarsen the precipitates by over aging so that the larger and thereby stronger precipitates may prevent the initiation of plastic flow localization by resisting dislocation motion.

A number of tensile and fracture toughness specimens of the CuCrZr alloy supplied by Outokumpu Oyj (Finland) were first prime aged at 733K for 3hr. After prime aging, some of the specimens were given over aging treatments at 873K for 1, 2 and 4 hr to coarsen the precipitates. The precipitate microstructure of specimens after each heat treatment was characterized using transmission electron microscopy (TEM). A number of tensile and fracture toughness specimens were mechanically tested at 333K and 573K. Some of the prime aged and over aged specimens were irradiated with fission neutrons in the BR-2 reactor at Mol (Belgium) at 333K and 573K to a dose level of 0.3 dpa. Both unirradiated and irradiated specimens in the prime aged and over aged conditions were used for tensile and fracture toughness (three point bend) testing at 333K and 573K. Post-deformation microstructure was investigated using TEM. The results of the mechanical testing and TEM investigations will be described and discussed. Although the over aging heat treatment eliminates the problem of a sharp yield drop and plastic instability, it neither restores the work hardening ability nor ductility of the alloy.

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18A.4

Study on the Interaction Between Dislocations and Helium Bubbles in Copper by In-Situ Straining Experiments in Transmission Electron Microscopy

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In fusion environment, helium atoms induced by nuclear reactions will degrade mechanical properties of materials. Effects of the helium on the macroscopic mechanical properties have been studied rather extensively, while fundamental knowledge about the interactions between dislocations and helium bubbles are not sufficient. The objective of present paper is to investigate the dynamic interactions between dislocations and helium bubbles in ion-irradiated copper by in-situ straining experiments in transmission electron microscopy (TEM).

The specimen used was pure copper (>99.999%). Prior to ion irradiation, TEM discs were electro-polished for the thin-foil irradiation. Helium ion irradiation was performed using an accelerator at room temperature. The energy of incident ions was 10keV and the fluence was 2×10^{17} ions/cm². After the ion irradiations, specimens were annealed at 650°C in high vacuum in order not only to obtain the lower pressure of the helium bubbles but also to observe the dislocation-defect interaction in detail. TEM observations were carried out with an

acceleration voltage of 200kV using the in-situ straining technique. Nano indentation tests were also performed to evaluate the change in mechanical property by irradiation.

Microstructure of homogeneous distributed bubbles was observed after irradiation with helium ions and the successive annealing. The average size of bubbles increased from 1.3nm to 50 nm by the annealing at 650°C, while the number density decreased from 7.2×10^{24} /m³ to 4.3×10^{20} /m³. The pressures of helium bubbles were estimated to decrease from 23.7 GPa to 0.6 GPa. Dislocations glided and interacted with helium bubbles with applying the stress at room temperature. The pinning-depinning behavior of moving dislocation was clearly observed by in-situ straining experiments, which indicated that the helium bubbles contributes the irradiation hardening. The barrier strength of helium bubbles against dislocation motions was quantitatively determined by measuring bow-out angles of dislocation immediately prior to breakaway. The obstacle strength was discussed not only with the dependence of the bubble pressure, but also the size distribution of bubbles. Attractive interaction between dislocations and helium bubbles were also examined in terms of the modulus effect of the obstacles. Macroscopic mechanical properties obtained from nano indentation tests were correlated with the values estimated by dislocation bow-out model.

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18A.5

Atomic-Scale Mechanisms of Cleared Channels Formation in Neutron-Irradiated Low Stacking Fault Energy fcc Metals

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Clusters of self-interstitial atoms (SIAs) and vacancies are formed directly in high-energy displacement cascades in all metallic structural materials. In low stacking fault energy (SFE) metals they can be faulted and perfect interstitial loops (IL) which are sessile and glissile, respectively, and stacking fault tetrahedra (SFTs). These defects are obstacles for dislocation motion and sources for strengthening, hardening and loss of ductility of irradiated metals. These effects are often accompanied with a yield drop (strain softening) and plastic instability when significant plastic deformation occurs within a limited

volume. These shear bands can also be cleared of radiation induced defects, leading to so called cleared channels. This phenomenon leads to unexpected material failure and is extensively studied by experimental and theoretical methods using a multiscale approach. A successful model for mechanical properties prediction must be able to describe creation and evolution of cleared channels which is impossible without knowledge of the atomic scale mechanisms of dislocation generation and motion through the high density of radiation defects (up to $\sim 10^{24} \text{m}^{-3}$, i.e., about 10nm distance between defects). Due to small scale and fast, non-equilibrium conditions the atomic scale modeling is the only technique that permits the necessary information to be obtained. In this paper we present results of large-scale atomistic modeling of edge and screw dislocations interaction with IL and SFTs in model Cu crystals. We report a number of different mechanisms and suggest a possible scenario for how shear bands can be cleared of radiation defects. The results are discussed and compared with experimental observations.

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Parallel Oral Session 18B – Deformation and Fracture

18B.1

Plastic Flow Properties and Fracture Toughness Characterization in the Lower Transition of Unirradiated and Irradiated Tempered Martensitic Steels

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The tempered martensitic steels represent one of the most technologically important class of materials for fusion reactor structural components due to their resistance to void swelling, and good balance of physical and mechanical properties. Understanding the underlying physical processes, mediating their plastic flow and fracture properties as well as their evolution with irradiation, is a key point in the successful development and application of these steels.

In this paper, we present first a model for the strain-hardening of these steels based on dislocation mechanics. We show that two temperature domains have to be considered to describe the strain-hardening, one above and one below 200 K respectively. The difference in the strain-hardening behavior stems from the intrinsic mobility of the screw segments that leads to two distinct relationships

between the flow stress and the dislocation density in each temperature domain. A set of dislocation parameters, consistent between the two domains, is used to describe satisfactorily the post-yield behavior over the temperature range [77 K – 723 K]. Second, a reference fracture toughness–temperature curve for the EUROFER97 has been established with 0.4T C(T) specimens. The results are presented along with those of the F82H steel. It is shown that the scatter in the lower transition of both steels is different from one another. A calibrated local criterion for cleavage is presented for the EUROFER97 based upon 3D finite element simulations of the stress/strain fields at the crack tip. Finally, the effect of neutron irradiation on the K(T) curves have been measured with pre-cracked sub-sized specimens, 0.2T C(T) and Charpy type, on the F82H-mod steel. Two specimen capsules have been neutron irradiated up to 2.5 dpa at two different temperatures at 333 K and 573 K, in the High Flux Reactor in Petten. For each irradiation condition, the fracture toughness-temperature curve K(T) has been characterized in the lower transition. Applying the concept of the master-curve, the irradiation-induced temperature shifts have been determined by calculating the reference temperature T_0 , which indexes the K(T) curve at a reference toughness. The effects of irradiation-induced changes in the constitutive behavior, yield stress increase and strain-hardening changes, on the attainment of the local criterion are outlined on the basis of finite element simulations, where the stress/strain fields at the crack tip are calculated.

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18B.2

Effect of Test Temperature and Strain Rate on Radiation Hardening*Steven J. Zinkle¹ and Bachu N. Singh²*

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Exposure of metals and alloys to neutron irradiation produces an increase in their matrix strength due to the introduction of point defect clusters (cavities, dislocation loops, etc.) and in many cases radiation-induced or -modified precipitates. This radiation hardening may lead to flow localization (localized necking) in structures, and can also lead to a decrease in the fracture toughness. This paper will review radiation hardening in body-centered cubic (BCC) and face-centered cubic (FCC) metals, with an emphasis on the effects of test temperature and strain rate. The dependence on strain rate and test temperature is determined by the dominant hardening centers, which is dependent on dose and irradiation temperature. At low doses where the amount of radiation hardening is small, the test temperature and strain rate dependence is similar to unirradiated material. When the hardening associated with irradiation becomes a significant fraction of the unirradiated strength, the strain rate dependence and test temperature dependence generally change compared to the unirradiated material behavior. For BCC metals, different strain rate behavior is often observed at test temperatures corresponding to the so-called thermally activated (low temperature) and athermal (high temperature) regimes. A further complication at elevated temperatures that is often of particular importance for BCC metals is dynamic strain aging associated with dispersed interstitial solute. Neutron irradiation can promote solute segregation to sinks and thereby modify the strain rate dependence associated with dynamic strain aging. The importance of strain rate and test temperature effects on deformation mechanisms and fracture toughness correlations with radiation hardening will be summarized. The various models for thermally activated flow in irradiated metals will be briefly reviewed and compared with experimental observations.

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18B.3

Effect of Temperature Change on the Irradiation Hardening of the Structural Alloys for ITER Blanket and ITER TBM Irradiated to 1.5 dpa in JMTR*S. Jitsukawa, E. Wakai, N. Okubo and M. Ohmi¹*

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In vessel components of fusion reactors will undergo their temperature change during service. It has been pointed out in several researches that temperature change during irradiation affected microstructural evolution through the change of the nucleation and the growth of the point defect cluster. This may also affect the irradiation hardening, and may result in the enhanced reduction of the ductility and the fracture toughness of martensitic and austenitic steels of the in vessel components.

By using an irradiation capsule with temperature control capability independent from reactor power (developed by JMTR operation at JAERI), reduced activation martensitic and austenitic stainless steels were irradiated at temperatures of 250 and 350C. Irradiation was performed during 10 reactor operation cycles to the accumulated damage level of 1.5 dpa. For some of the specimens, temperature was changed during irradiation; for instance, temperature was elevated from 250 to 350C at the middle period of each reactor operation cycle.

Tensile tests were performed at room temperature after irradiation. The temperature change reduced the irradiation hardening of the austenitic steel. On the other hand, the yield stress level for a reduced activation martensitic steel (F82H) irradiated at both temperatures of 250 and 350C was higher than those irradiated at a constant temperature of either 250 and 350C by 10 and 18%, respectively. The impact of the effect of the temperature change on the residual ductility will be reported, together with the results of the transmission electron microscopy.

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18B.4

Qualification of CuCrZr Alloy to Stainless Steel Joints

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The first wall and divertor structures of ITER are multilayer components consisting of austenitic stainless steel, copper alloys and plasma facing armour materials. There are several types of copper to stainless steel joints which have to withstand the thermal and mechanical loads under neutron irradiation condition. To evaluate structural integrity of these multilayer components a criteria for structural design and for qualification of the dissimilar metal joints is needed. In the present manufacturing rules there is no clear quality or strength criteria for the Cu/SS or SS/SS joint interfaces. Particularly the strength of CuCrZr alloy is very sensitive to heat treatments during manufacturing cycle and consequently also the strength of Cu/SS joints.

The CuCrZr / 316 L(N) joints were produced by HIP method (CEA Grenoble) at 1040 or 960°C for 2 hours at a pressure of 140 MPa followed by heat treatment at 980°C for 30 minutes with subsequent cooling (60-70°Cmin⁻¹) and precipitation heat treatment at 480 or 560°C for 2 hours. The applied thermal cycles represent the foreseen manufacturing cycles of the ITER first wall panels. Metallographic characterisation together with tensile and fracture toughness tests were carried out at ambient temperature and 300°C.

The obtained results indicate that due to HIP thermal cycles the tensile strength of base CuCrZr alloy can be varied by about 70 MPa. The corresponding change in strength difference between the copper alloys and stainless steel joint specimens dominates the mechanical properties of the Cu/SS joint specimens. The main results of these investigations will be presented and their implications for structural design or qualification criteria of the Cu/SS joints will be discussed.

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18B.5

Mechanical Property Degradation of Ferritic/Martensitic Steels after the Fast Reactor Irradiation "ARBOR 1"

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In an energy generating fusion reactor, structural materials will be exposed to very high levels of irradiation damage of about 100 dpa. A simulation facility - like IFMIF - is not available in the nearer future, to study the materials behavior under fusion relevant irradiation conditions, e.g., the specific He/dpa-ratio. Therefore these irradiation damage conditions can be realized in fast reactors only. For this purpose a cooperation between Forschungszentrum Karlsruhe and State Scientific Centre of Russian Federation Research Institute of Atomic Reactors (SSC RF RIAR) had been implemented. The irradiation project "Associated Reactor Irradiation in BOR 60" is named "ARBOR" (Latin for tree).

Tensile, low cycle fatigue and Charpy specimens of Reduced Activation Ferritic/Martensitic (RAFMs) steels, e.g., EUROFER 97, F82H mod., OPTIFER IVc, EUROFER 97 with different boron contents and ODS-EUROFER 97 have been irradiated in a fast neutron flux ($> 0.1 \text{ MeV}$) of $1,8 \cdot 10^{15} \text{ n/cm}^2\text{s}$ and with

direct sodium cooling at a temperature $< 340^\circ\text{C}$ to 15 and $\sim 30 \text{ dpa}$. The unloaded part of irradiation project "ARBOR 1" includes 60 mini-tensile/low cycle fatigue specimens and 90 mini-Charpy (KLST) specimens of nine different RAFM steels.

In the hot cells of SSC RF RIAR a modern instrumented impact testing facility of Zwick-HKE type and an INSTRON-DOLI tensile/low cycle fatigue testing facility with high temperature furnace and direct strain measurement system are operating.

First impact results on EUROFER 97 show a dramatic increase in the Ductile to Brittle Transition Temperature (DBTT) as an effect of irradiation ($T_{\text{irr}} = 330^\circ\text{C}$, 15 dpa, DBTT $\sim 103^\circ\text{C}$, $T_{\text{irr}} = 330^\circ\text{C}$, 33 dpa, DBTT $\sim 132^\circ\text{C}$) compared to unirradiated reference data (DBTT $\sim -91^\circ\text{C}$).

The tensile behavior of EUROFER 97 after irradiation leads to a hardening ($T_{\text{irr}} \sim T_{\text{test}} \sim 330^\circ\text{C}$, 15 dpa, $R_{p0.2} = 870 \text{ MPa}$) in combination with a reduction in uniform strain ($A_g = 0.4 \%$), but with a considerable total strain $A = 9.4 \%$.

Since these post irradiation experiments started in December 2004, we expect to present complete results also for the other materials when the paper will be prepared.

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Parallel Oral Session 19A – Vanadium and Refractory Metals

19A.1

Advances in Development of Vanadium Alloys and MHD Insulator Coatings

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In this paper, recent progress in the development of low activation vanadium alloys and MHD insulator coating for liquid lithium blanket is overviewed. Emphasis is placed on the achievements in the framework of Japan-USA cooperation program, JUPITER-II. Significant progress in the fabrication

technology has been made for vanadium alloys such as manufacturing various shapes of products, weld joints, tubes, and W-coating for application to plasma-facing components. With the resulting high quality pressurized creep tube specimens, systematic thermal creep data have been obtained both in Li and vacuum environments. Fundamental understandings on the effects of interstitial impurities (C, N, O) and their precipitates on mechanical properties, radiation effects on microstructure and mechanical properties, corrosion and compatibility in various environments were enhanced.

New promising candidates were identified for the MHD insulator coating. Especially Er_2O_3 coated by PVD was shown to have high stability in Li to 700C and 1000hr. At present, efforts are being focused on development of two-layer coatings with Er_2O_3 or other insulating ceramics as the intermediate layer and vanadium alloys as the top layer. In-situ coating technology with Er_2O_3 is also making significant progress.

Among the remaining critical issues are the effects of transmutant helium on mechanical properties of vanadium alloys and stability of the coatings in flowing Li environments.

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19A.2

Thermal Creep of Two Heats of V-4Cr-4Ti in a Liquid Lithium Environment

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This paper presents recent thermal creep results in liquid lithium at 700 and 800°C on two heats of V4Cr4Ti, US Heat 832665 and Japanese NIFS-HEAT-2. The thermal creep behavior of US Heat 832665 has been previously investigated in uniaxial creep tests and biaxial creep tests in vacuum environments at temperatures between 650 and 800°C up to 10000 hours. Biaxial creep tests on this heat were also carried out in a liquid lithium environment, but mainly at higher stress levels. It has been recognized that the thermal creep behavior of V4Cr4Ti is controlled by dislocation power-law creep at high stresses at 650 -

800°C. The dominant creep mechanism might be different at lower stresses, but there are only limited data in this stress and temperature regime. This study focuses on the thermal creep at lower stress levels for times up to 2000 hours to generate more comprehensive creep data of the alloy in a lithium environment and to better understand the controlling creep deformation mechanisms in various stress and temperature regimes.

Japanese NIFS-HEAT-2 has a reduced impurity level compared to US Heat 832665. Previous studies indicated that the interstitial impurities, particularly oxygen, have a significant effect on the creep strain rates of this alloy. What remains unclear is if the impurity levels in the starting material and the environmental impurity sources play the same role in the thermal creep performance. This paper investigates the influence of interstitial impurities, O, N, and C on the creep properties by comparing the creep data for the two heats and for two environments, liquid lithium and vacuum.

It was found that there are three different thermal creep deformation mechanisms depending on the stress and temperature. The creep data suggested that V-4Cr-4Ti appears to have sufficient creep strength to be used in Li-cooled fusion structures at temperatures up to 750°C.

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19A.3

Influence of Cr and Ti Content on Compatibility of V-Cr-Ti Type Alloys with Liquid Lithium

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Self-cooled liquid lithium / vanadium alloy blanket system is one of the most promising concepts for fusion reactors. But in this system, a magneto-hydrodynamic (MHD) pressure drop is developed by flowing liquid lithium across the magnetic field. Recent experiments using several ceramics-coating have shown that Y₂O₃ and Er₂O₃ are attractive candidates for MHD insulator coatings because of their compatibility with lithium. On the other hand, because there are likely to be cracks in any coatings, a dual-layer system with a thin outer layer of vanadium alloy appears to be a more attractive MHD coating system. This paper reports the effects of Cr and Ti content on compatibility of vanadium alloys with liquid lithium.

V-(4, 7, 10, 12, 15, 20)Cr-(4, 10, 15)Ti alloys were prepared for exposure tests. Exposure tests were carried out at 800 °C for 1000 h in liquid lithium. After exposure, the weight change of each alloy was measured. Observations of microstructures by using SEM, and characterization of surface oxide or nitride layer by X-ray diffraction for each alloy were carried out. In order to evaluate the influence of exposure in liquid lithium on mechanical properties, tensile tests and hardness tests after exposure were also conducted.

As consequences of exposure tests in Li, weights of V-4Cr-4Ti alloys increased about 0.2g/m². The weight gain of each alloy was reduced as Cr concentration increased, and the weight of V-15Cr-4Ti alloy decreased about 0.75g/m². The V-7Cr-4Ti alloy had about 0.1g/m² weight increasing and the level of weight change was the smallest of all the tested alloys. Nitride was formed on the surface of V-4Cr-15Ti alloy resulting that its weight increased four times larger than that of V-4Cr-4Ti alloy.

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19A.4

The Microstructure of Laser Welded V-4Cr-4Ti Alloy after Neutron Irradiation

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It is well known that interstitial impurities such as oxygen and nitrogen play an important role in V-4Cr-4Ti alloy during irradiation. To investigate the effect of oxygen on titanium oxide formation in the jointed materials, the bead-on-plate laser welding for V-4Cr-4Ti alloy (NIFS-HEAT-2) was performed using YAG laser beams. The microstructure of heat affected zone (HAS) and weld metal, which was observed by transmission electron microscopy, was compared before and after neutron irradiation.

High purity V-4Cr-4Ti alloy (NIFS-HEAT-2), fabricated by National Institute for Fusion Science (NIFS), was used for this study. The samples were annealed at 1273K for 2 hours and bead-on-plate welds were performed using high argon gas. The measured oxygen concentration of weld metal are 139(before welding) and 158 (after welding) wt ppm, respectively. Neutron irradiation was carried out with JMTR in the temperature range of 573K-873K. Irradiation dose was about 0.1 dpa. The microstructure of before irradiation showed that relatively large precipitates which were commonly observed in NIFS-HEAT-2 disappeared in the center of weld metal. After the irradiation, fine titanium oxides with {100} habit plane were detected. But less number density of these oxides were observed in the unwelded samples after the same irradiation conditions. This means that the contribution of oxygen atoms which dissolved from large precipitates during the laser welding is essential to understand the microstructural evolution of welded V-4Cr-4Ti alloys. In this paper, present results were also compared with a previous ion irradiation.

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19A.5

Fracture Toughness Investigations of Tungsten Alloys and Severe Plastic Deformed Tungsten Alloys

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Tungsten and its alloys offer high melting points, low vapour pressures, good thermal conductivity, high erosion resistance and good thermoshock properties. These properties are necessary and essential in applications like fusion reactors. Like other bcc materials tungsten shows a DBTT (ductile-to-brittle transition temperature) which is very dependent on its processing and impurities. This complicates the machining (forging, turning etc.) of complex parts. Thus a low brittleness is essential and desired. In contrast to usual, widely used deforming mechanisms (rolling, drawing etc.) Severe Plastic Deformation (SPD) is not only capable of increasing the tensile strength and hardness of a material, it also can increase its ductility.

High Pressure Torsion was therefore used to deform three different rolled tungsten alloys, namely pure tungsten (W), a lanthanum-oxide dispersion strengthened tungsten alloy (WL10) and a potassium doped tungsten alloy (WVM). Applying high plastic strains reduces grain sizes and influences the particle distributions, leading to a near-nano-structured material. These HPT-specimens also undergo fracture mechanical and recrystallisation tests, to determine the improvement in ductility and their thermal stability. The evolution of the microstructure is also studied.

Additionally the fracture toughness of the three different tungsten alloys in various different rolled conditions is investigated. Because tungsten has a ductile-to-brittle-transition temperature in the regime between 200-600°C, the fracture toughness tests are performed at RT, 200°C, 400°C, 600°C and 800°C. The influence of the initial state as well as the orientation dependence are taken into account.

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Parallel Oral Session 19B – Chemical Compatibility & Coatings

19B.1

Multilayer Systems for Sustaining High Heat Flux

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It is clear that fusion first wall-diverter-blanket structures will require coatings and layered materials with various functions that present tremendous challenges. This is true for ITER first wall-diverter-shield structures and test blanket modules (TBMs), and even more so for power producing demonstration reactors. Key functions include: corrosion-oxidation-erosion protection; tritium permeation barriers; low or high Z armor on plasma facing components; and, electrical insulating coatings for liquid metal coolants. While some, or perhaps many, of the details are markedly different, coatings and layered fusion materials systems present many of the same daunting challenges as they do in high temperature aerospace applications, like thermal barrier coatings (TBCs) used in jet engines and oxidation and erosion protection coatings for high heat flux components in hypersonic aircraft. Common interrelated issues include: manufacturing very complex, high tolerance component geometries; the need for high levels of reliability and safety margins, along with defect tolerance and well defined procedures for in-service inspection and maintenance; high temperatures and heat fluxes; large amplitude temperature cycling; damage accumulation and microchemical and microstructural instabilities; time varying stress driven inelastic deformations in substrate components; chemically reactive environments; and, large economic incentives for long lifetimes. Further, like in aerospace applications, fusion coatings and layers will be comprised of several different types of materials, typically metals and ceramics, in situations where topology and geometry are important to function. Thus new, high performance coatings and layered materials must be ultimately developed in a materials-structure-system context, founded on a solid knowledge base. This lecture will present an overview of a multi-institutional, international science based effort to develop coatings and layered materials systems for practical aerospace engineering applications. The program involves an integrated modeling-experiment balance and combines the talents of experts in many aspects of mechanical, materials and manufacturing sciences.

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19B.2

Coatings and Joining for SiC/SiC Composites for Nuclear Energy Systems

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Coatings and joining materials for SiC/SiC composites for nuclear energy systems are being developed at PNNL using preceramic polymers filled with reactive metal powders and inert powders and using displacement reactions with no polymers. Polymer-filled joints and coatings start with a poly(hydridomethylsiloxane) precursor to produce a poly(methylsilsesquioxane) preceramic polymer. Such preceramic polymers are filled with mixtures of Al/Al₂O₃, Al/SiC, and SiC powders to form polymer-powder slurries that are applied to SiC Hexaloy and SiC/SiC composite substrates using dip coating, painting, or spraying methods and pyrolyzed in a variety of reactive atmospheres at 800°C to 1200°C. Simple joining techniques using these polymer mixtures and using a solid-state displacement reaction involving TiC and Si have been demonstrated, and mechanical shear strength data obtained. Coatings made with starting mixtures of Al/Al₂O₃/polymer form a hard oxide coating on SiC, coatings made with Al/SiC mixtures form a mixed oxide-carbide coating, while coatings made with SiC/polymer form a porous, hard carbide coating. Coatings range from 10 to 50 microns thick, are multiphase, and contain some porosity as processed. Joints made from such mixtures have similar microstructures and joint strengths depend on applied pressure. Joint shear strengths range from 15 to 50 MPa depending on the applied pressure and joint composition. The strongest joints were obtained using tape cast ribbons of Si/TiC powders such that a solid state displacement reaction at 1100°C using 15 MPa applied pressure resulted in shear strengths of 50 MPa, which exceeds the shear strength of SiC/SiC composite materials. However, the polymer slurry joints are much easier to apply and could be considered for in situ or field repair situations.

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19B.3

Comparison of Corrosion Behavior of Bare and Hot-Dip Coated EUROFER Steel in Flowing Pb-17Li

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Low-activation-ferritic-martensitic (RAFM) steels are considered for application in fusion technology as structural materials. The EUROFER steel was developed on the basis of the experience gained with steels of the OPTIFER, MANET and F82H-mod. type. In the blanket concept with Pb-17Li as liquid breeder, this steel will be in contact with the eutectic liquid metal and its corrosion behavior is therefore of significance for a successful application. Corrosion tests of EUROFER in flowing Pb-17Li at 480°C were performed up to about 12,000 h to evaluate the kind and the kinetics of the dissolution attack. The results show that EUROFER 97 is attacked in the flowing liquid Pb-17Li by dissolving of the typical steel elements like iron and chromium. The observed attack is of uniform type with linear kinetics. The corrosion rate is somewhat smaller for EUROFER compared to the other RAFM steels but with equal activation energy, due to differences in the composition of the passive oxide layer.

Tritium permeation barriers on low-activation steels are required in fusion technology in order to reduce the tritium permeation rate through the structural material into the coolant (helium). Fe-Al layers with alumina on top might fulfill the required reduction rate. One of the possible techniques in the EU is the hot-dip aluminizing process (HDA), developed by FZK. Besides the permeation reduction, a sufficient corrosion resistance against dissolution attack by Pb-17Li is required, too, especially when higher temperatures are considered. Therefore, the corrosion behavior of coated EUROFER steel was studied in flowing Pb-17Li under the same conditions as the bare steel to compare the different corrosion behavior and emphasize the effect of a protective coating. It was obvious that a corrosion attack could not be found on coated steel even up to the longest exposure time, whereas the bare EUROFER steel showed a dissolution rate of about 80-100 $\mu\text{m}/\text{year}$.

By concluding the results it is evident, that Fe-Al layers, e.g., produced by HDA process, are able to withstand the corrosive attack by Pb-17Li and therefore can act as tritium permeation and/or corrosion barriers on EUROFER steel, depending on the blanket concept requirements.

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19B.4

Research on Calcium Zirconate as a Insulating Coating for Li Blanket

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A self-cooled liquid lithium (Li) / vanadium alloy blanket concept is one of the most attractive ones for a DEMO fusion reactor. In order to reduce magnetohydrodynamics (MHD) pressure drop induced by the liquid Li flowing across high magnetic fields, fabrication of thin insulating coating (MHD coating) on the inner surface of tubing has been proposed. Candidate materials for the coating are strictly limited because of high chemical reactivity of Li with ceramics. In this study, calcium zirconate and its coatings were investigated to clarify its possibility as the candidate material.

Corrosion behavior of poly-crystal CaZrO_3 were immersed in liquid Li. $\text{CaZrO}_3 + \text{ZrO}_2$ showed a poor compatibility with liquid Li, while CaZrO_3 kept a small

change of mass and thickness. High-density $\text{CaZr}_{0.95}\text{Sc}_{0.05}\text{O}_{3-x}$, supplied by TYK Corp., which contained a small amount of Sc as a binding agent, decreased the change of the thickness and mass at 973 K. Keys to the corrosion-resistance for CaZrO_3 coating are pointed out to increase the crystallinity, the purity, and the density.

Magnetron sputtering device was employed for fabricating coatings. In the device, calcium metal target with RF and zirconium metal target with DC were used, and it was possible to control metallic element composition by adjusting RF or DC power. Oxygen and argon were introduced for generating plasma. Several parameters of gas pressure, RF, DC, temperature of the substrates and bias voltage between the targets and the substrates were employed to control composition rate of the coatings.

Some of the coated specimens had almost the same ratio of calcium to zirconium to stoichiometric composition. No impurities are observed in the coating by XPS analysis. The specimens had no peaks of CaZrO_3 observed by XRD, but had some peaks of $\text{Ca}_{0.15}\text{Zr}_{0.85}\text{O}_3$. To increase the crystallinity is the important to develop the CaZrO_3 coatings.

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19B.5

Investigation of Pb-Li Compatibility for the Dual Coolant Test Blanket Module

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One proposed test blanket module (TBM) concept for ITER uses advanced ferritic alloys with both Pb-17at.%Li and He coolants. In order for the blanket to operate at $\approx 800^\circ\text{C}$ and prevent unacceptably high dissolution of the steel in Pb-Li, a SiC/SiC composite flow channel insert is used. The insert also would reduce the magnetohydrodynamic pressure drop. Two unresolved Pb-Li compatibility issues are being studied in this system. First, there is limited information about the long-term compatibility of SiC/SiC in Pb-Li. Prior static capsule test results showed no detectable dissolution of Si in Pb-Li after 1,000h at 800°C and 1100°C . However, it is possible that dissolution could proceed after an incubation period. In order to further study this issue, specimens of high-purity, chemical vapor deposited (CVD) SiC are being exposed in capsules for up to 5,000h at 800°C and 1,000h at 1200°C . The mass change and Pb-Li chemistry will be measured after the tests are completed.

The second issue involves Pb-Li transport between the first wall and the heat exchanger. The tubing material for this portion will need to be compatible with Pb-Li at $\leq 700^\circ\text{C}$. This temperature is unacceptably high for most conventional Fe- and Ni-base alloys due to dissolution. A SiC coating could be protective, but coating flaws would expose the substrate to rapid attack. Another possibility would be aluminizing the tubing material to produce a surface rich in FeAl and/or NiAl. The coating itself or a pre-oxidized alumina surface layer could act as a corrosion barrier. Thermodynamic calculations like those made by Hubberstey et al. are reexamining the compatibility of Fe-Al, Ni-Al and alumina in Pb-Li. Initial Pb-Li capsule tests also are being conducted to examine the potential benefits of Al-containing coatings. Knowledge gained from the analytical and experimental work will assist in the design of loop tests necessary to verify compatibility issues relevant to the TBM.

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Authors

Abdou, Mohamed.....	109	Baskes, Mike I.....	74
Abdou, M.....	107	Bazaleev, E.N.....	112
Abe, Hiroaki.....	142	Bazylev, B.N.....	241
Abe, K.....	39, 48, 49, 50, 85, 170	Bekris, N.....	200, 202, 204
Abe, S.....	19	Beliakov, V.....	104
Abou-Sena, A.....	107	Beloglazov, Sergey.....	204
Aiello, A.....	44	Benamati, G.....	44, 198
Akasakaa, N.....	88, 139, 191	Bentley, J.....	66
Akiba, M.....	8, 231	Berensky, L.....	129
Akiyoshi, M.....	112, 118	Bernath, J.....	140, 193
Aksyonov, Yu.....	129	Bertsch, J.....	83
Aktaa, J.....	6, 9, 105, 161, 186, 229	Beznosov, A.V.....	108
Al Mazouzi, Abderrahim.....	11	Bhadesia, H.K.D.H.....	4, 127
Alamo, A.....	134	Binyukova, S. Yu.....	77, 121
Alinger, M.....	85, 88, 140, 191, 193	Birtcher, R. C.....	24
Almazouzi, A.....	26	Blokhin, A.I.....	10
Alves, E.....	110, 137	Blöme, M.....	107
Alves, L. C.....	110, 137	Bobin-Vastra, I.....	238
An, Zhiyong.....	109	Boccaccini, L.V.....	202
Anderl, R. A.....	198	Boeriu, S.....	130
Andersen, Michael.....	148	Boev, E. V.....	219
Ando, M.....	15, 82	Bolt, H.....	83, 175, 197, 204
Anma, Y.....	67	Bonadé, R.....	14
Arai, T.....	40, 151, 236	Boonsuwan, Peeravuth.....	30
Arakawa, K.....	24, 84, 101	Borodin, V.....	31
Araki, Hiroshi.....	164, 212	Boskeljon, J.....	106, 188
Arévalo, C.....	27	Bravo, D.....	210
Arnoldov, M.....	129	Brendel, A.....	83, 175
Aruga, T.....	109	Brendel, T.....	175
Asakura, N.....	151	Bringa, E. M.....	16
Asaoka, Y.....	40	Budylnkin, N. I.....	11
Ashikawa, N.....	223, 242	Bulanova T.M.....	86
Aubert, P.....	106	Bus, Tjacka.....	4
Awano, Misa.....	97	Busby, J.T.....	47
Ayrault, D.....	32	Byun, Thak Sang.....	6, 48, 215, 227
Baba, T.....	73	Cabellos, O.....	127
Bacon, D.J.....	2, 24, 92, 219	Calder, Andrew F.....	24
Baluc, N.....	14, 96, 194	Calderoni, Patrick.....	114
Barabash, V.....	105, 174	Cambi, G.....	130
Baranova, O.V.....	108	Campitelli, E.....	83
Barashev, Alexander V.....	24	Cao, Yunzhen.....	240
Bartenev, S. A.....	156	Caro, A.....	16
		Casalegno, V.....	33, 232
		Caturla, M. J.....	27, 28, 91, 94, 214

Causey, R.A.....	200	de Castro, V.....	136
Cayron, C.....	134	de Dinechin, G.....	106
Cepas, P.....	27	Decréton, Marc.....	207
Cepraga, D.G.....	130	Dekeyser, J.....	37
Chaffron, L.....	134	Dell'Orco, G.....	132
Chagnot, Ch.....	32	Demin, N.A.....	10
Chakin, V.P.....	124	den Boef, R.....	188
Chaouadi, R.....	216	Deo, Chaitanya S.....	74
Chehtov, Tz.....	186	Derlet, P. M.....	90
Chen, Fu-Rong.....	56, 103	Di Maio, P. A.....	108
Chen, H.....	157	Díaz, S.....	127
Chen, J.....	144	Dohi, Kenji.....	61
Chen, M.....	87, 157, 222	Dolinskii, Yu. N.....	216
Chen, Y.....	15, 133, 157	Domonkos, Peter.....	176
Chernavskaya, N.A.....	21	Doronina, T.A.....	176
Chernov, I.I.....	77, 121	Drazic, G.....	57
Chernov, V. M.....	10, 21, 25, 66, 86, 129, 173	Duan, Jinglai.....	23
Chikhray, Y.....	65, 203	Dudarev, S L.....	30, 91
Chimi, Y.....	109	Durocher, A.....	32, 33, 238
Chiovaro, P.....	108	Dux, R.....	34
Chiu, Y. L.....	28	Dvoriashin, A. M.....	11
Cho, H.S.....	134, 196	Ebisu, Shinji.....	240
Cho, Hang-Sik.....	62, 196	Egawa, Takayuki.....	202
Cho, S.....	104	Egorov, V.A.....	95
Chubarov, S.V.....	77	Eldrup, M.....	76, 176
Chuto, T.....	50	Emmoth, B.....	107
Ciampichetti, Andrea.....	156, 198	Endo, Shinya.....	220
Clift, M.W.....	200	Enoeda, M.....	8
Coad, J.P.....	200	Esaka, Hisao.....	139, 187
Cockeram, B.....	48	Escourbiac, F.....	33, 238
Coffe, y G.....	53	Esteban G. A.....	84
Constans, S.....	33	Evtikhin, V.A.....	112
Constantinescu, Bogdan.....	5, 213	Ezato, K.....	231
Cook, I.....	160	Ezawa, T.....	102
Coppola, R.....	97	Fabritsiev, A.....	105
Cottrell, G.A.....	4, 127	Fabritsiev, S.A.....	46, 149, 150, 162, 174
Courtoi, s X.....	33	Faulkner, R. G.....	141
Cowgill, D.F.....	200	FDS Team.....	157
d'Hulst, D.S.....	106	Fedotovskiy, V.....	129
da Silva, M.R.....	110	Feng, Y.....	87, 222
Dafferner, B.....	6, 9	Fernández, P.....	135, 136
Dai, Y.....	178	Ferraris, M.....	232
Damm, E.....	200	Ferreira, Jose Antonio.....	201
Davydov, D.A.....	153	Fields, K. A.....	226
de Carlan, Y.....	134		

Filacchioni, G.	86, 229	Gould, J.	140
Fischer, U.	15, 128, 133, 233	Gould, Jerry	193
Flewitt, P.E.J.	141	Gragg, D.	87, 186, 226
Fokkens, J.H.	111	Greuner, H.	34
Forrest, R. A.	131, 151	Grismanovs, V.	109
Fortuna, E.	36	Gröschel, F.	126
Franco, N.	110	Grossbeck, Martin L.	82
Frisoni, M.	130	Grosse, M.	178
Fujita, D.	170	Gruber, O.	34
Fujita, S.	226	Gusev, M. N.	225
Fujita, T.	185		
Fujiwara, M.	7, 48, 49, 50, 134, 135, 136, 138, 196	Ham, N.	20
Fujiwara, T.	20, 231	Hanada, K.	20
Fujiwara, Y.	124	Hara, M.	201
Fukada, S.	198	Hasegawa, A.	39, 48, 54, 85, 120, 170
Fukuda, Kunio	209	Hasegawa, M.	193
Fukumoto, Ken-ichi	7, 63	Hasegawa, M.	65, 70
Fukumoto, Masakatsu	36	Hashida, T.	7
Furuichi, Kazuya	152	Hashimoto, Kazuaki	168
Furuya, K.	119	Hashimoto, N.	15, 48, 215
		Hashimoto, Naoyuki	6
Gaganidze, E.	6	Hashimoto, Takuya	43
Gámez, M. L.	32	Hashitomi, O.	55, 140
Gao, F.	73, 80, 119, 120	Hashizume, K.	69
García-Herranz, N.	127	Hatakeyama, M.	70
Garner, F. A.	11, 173, 176, 177, 179, 219, 222, 225,	Hatanaka, M.	101
Gartvig, K.S.	162	Hatano, Y.	69, 71, 198
Ge, Chang chun	35, 38, 231, 235	Hayakawa, R.	71
Gelles, D. S.	53, 68, 148	Hayashi, T.	185
Gervash, A.	163, 233, 242	Hayashi, Takumi	152
Gerwash, A.	174	Hayashishita, E.	51
Gessi, A.	44	Hayes, J.	224
Ghoniem, N. M.	29, 92, 148, 159	Hayes, Jeffrey P.	223
Gilbert, E. R.	179	He, M. Y.	143, 230
Giniyatulin, R.	163, 233	Hegeman J.B.J.	42, 111, 169
Glade, S.C.	193	Heinisch, H. L.	
Glade, Stephen C.	118	Heinisch, H. L.	25, 73, 80, 120
Glasbrenner, H.	126	Heinzel, V.	128
Glugla, M.	200, 202, 204	Henager Jr, C. H.	55
Golovanov, V.N.	86	Hide, Koichiro	220
Golubov, S.I.	23, 98, 117	Higashi, S.	158
Goraieb, A.A.	37	Hino, T.	50, 51, 68, 89, 223
Gotoh, Y.	151	Hinoki, Tatsuya	52, 102, 142, 165, 166, 169
		Hirai, T.	33, 239
		Hirohata Y.	40, 51, 68, 89, 223, 236

Hirose, T.....	8, 184	Ishii, Y.....	167
Hiwatari, R.....	40	Ishikawa, A.....	15
Hodge, A.....	16	Ishikurab, Takefumi.....	212
Hodgson, E.R.....	, 210, 211	Ishimoto, Y.....	40, 151, 236
Hodgson, Eric.....	207	Ishimoto, Yuki.....	242
Hoelzer, D.....	191	Ishizaka, Tomotaka.....	41
Hoelzer, D.T.....	64, 66, 192	Ivanov, A.D.....	217
Hoffelner, W.....	90, 144	Iwai, T.....	97, 173
Hollis, K. J.....	234	Iwakiri, H.....	73
Holstein, N.....	233	Iwakiri, Hiroto.....	79, 235
Honda, A.....	214	Iwasawa, Misako.....	93
Hong, Jun Hwa.....	100, 137, 185	Iwase, A.....	109
Hong, Soon Hyung.....	137	Iwata, N.Y.....	136
Hono, K.....	67	Jacquet, P.....	37
Hori, Jun-ichi.....	154	James, Michael R.....	74
Horiike, H.....	132	Janeschitz, G.....	233, 241
Horiki, M.....	22	Jang, C.H.....	195
Höschen, T.....	234	Jang, Je-Wook.....	100
Hoshino, Tsuyoshi.....	43	Jang, Jinsung.....	137
Hou, M.....	29	Janin, F.....	106
Hribernik, M.....	85, 88	Jankowski, A.....	224
Huang Q.....	15, 40, 82, 87, 157, 222	Jankowski, Alan F.....	223
Huang, D.....	157	Jenkins, M L.....	30, 91
Huang, Q.Y.....	188	Jia, X.....	178
Huang, Qunying.....	12	Jiao, Z.....	20
Hurchand, H.....	141	Jin, Yunfan.....	23
Hyuga, Hideki.....	168	Jinzenji, Ryushi.....	155
Iacovone, B.....	229	Jitsukawa, S.....	14, 15, 19, 54, 85, 109, 120, 146, 167, 183, 218, 228
Ibarra, A.....	28, 32, 94, 210, 214	Jones, L.....	106
Ice, G. E.....	17	Jong, M.....	169, 188
Ida, M.....	126	Joseph, T.D.....	143
Igawa, N.....	54, 120, 167	Jung, Hun Chae.....	171
Ihli, T.....	163, 233	Jung, P.....	76, 144
Iida, Toshiyuki.....	113	Kai, Ji-Jung.....	56, 103
Iikubo, T.....	50	Kaito, T.....	194
Ikeda, S.....	52	Kakie, M.....	49
Ikeda, Toshiji.....	113	Kalashnikov, A.N.....	77
Ilyin, A.M.....	93	Kalin, B.A.....	77, 121
Imai, Masamitsu.....	3, 168, 209	Kalinin, G.M.....	147, 217
Imamura, J.....	102	Kallenbach, A.....	34
Inai, Kensuke.....	240	Kaminaga, A.....	160, 205
Inouye, A.....	211	Kang, S.M.....	166
Ioltukhovskiy, A.G.....	86	Karduck, P.....	107
Ishiga, M.....	85		
Ishii, T.....	241		

Kasada, R.	76, 140	Köck, T.	175, 197
Kasada, Ryuta.....	196, 228	Kohayama, A.....	135
Katayama, Kazunari.....	152, 154, 206	Kohno, Y.....	7, 82
Katayama, Keiichi	208	Kohyama, A.....	142
Kato, Y.	15, 119	Kohyama, A.....	7, 8, 51, 52
Kato, Y.	4	Kohyama, Akira.....	55, 82, 102, 164, 165, 166, 171, 184, 187
Kato, Yutai.....	56, 169, 227	Kolbasov, B. N.....	153, 156
Kawakami, T.	75	Kolbayenkov A.	65
Kawamura, H.	45, 203	Komarov, V.....	242
Kawano, Hiroko.....	208	Komatsu, T.....	49
Kawasaki, Hiromitsu.....	154	Komazaki, S.	7
Kawashima, N.	136	Kondo, Keitaro.....	205
Kelly, Joseph.....	17	Kondo, Naoki.....	168
Kemp, R.	4, 127	Kondo, Sosuke	102, 165
Kempe, Helmut	9	Konobeev, Yu. V.	11, 177
Keng, Hsu-Tsu	56	Konstantinov, V.L.	108
Kenny, S.D.	141	Korotaev, A.D.	66
Kenzhin Y.....	65	Kovalev, A.M.	124
Kholopova, O.V.....	153	Kozlov, A. V.....	147, 173
Kikuchi, Kenji.....	199	Kraft, O.....	186
Kim, C. S.	104	Krauss, W.....	233
Kim, I.S.....	195	Kreter, A.	107
Kim, Sa-Woong.....	82, 184	Krieger, K.	34
Kim, Sung Ho	185	Kruessmann, R.....	233
Kim, W.-J.....	166	Kubota, A.....	28
Kimura, A.	76, 134, 136, 140, 146, 195, 196	Kubota, Naoyoshi	205
Kimura, Akihiko	62, 196, 228	Kudo, T.....	48
Kimura, Hiromi	235	Kudo, Y.....	160, 185
Kinjyo, Tomohiro	41	Kühnlein, W.....	33
Kirk, M A.....	30, 91	Kulcinski, G.L.....	16
Kirschner, A.....	107	Kulsartov, T.	65, 203
Kishimoto, Hirotatsu	55, 187	Kunz, C.L.....	200
Kishimoto, H.....	4, 76, 123, 140, 190	Kupriyanov, I.B.	112, 124
Kita, Hideki	168	Kuramoto, Eiichi	22
Kizu, Kaname.....	242	Kurbatova, L.A.....	112
Klimiankou, M.....	37, 138, 192	Kurinskiy, P.	37
Klingensmith, D.....	226, 230	Kurishita, H.....	65, 87
Klotz, M.	161	Kurtz, R. J.....	120
Klueh, R. L.	12, 13	Kurtz, R. J.....	42, 49, 64, 68, 73, 80, 119
Knott, J.F.....	141	Kurzydowski, K.J.	36
Kobayashi, Kazuhiro	152, 199	Kusama, Y.....	89
Kobayashi, S.	65	Kusuyama, Hiroyasu	187
Koch, F.....	197	Kuwabara, T.....	65
Koch, F.....	204	Kuznetsov, V.	163, 233
Koch, Freimut.....	207		

Kuznetsov, Yu.G.	147	Luo, G.-N.	80
Kvasnitskij, I. B.	156	Luo, Tianyong.	199
Lancha, A. M.	135, 136	Lyublinski, I.E.	112
Landman, I.S.	241	Maday, Marie-Françoise.	125
Lapeña, J.	135	Magielsen, A.J.	111, 202
Lara-Curzio, Edgar	227	Magnani, M.	97
Lee, Byeong-Joo	100	Maier, H.	34, 197, 204
Lee, Hyon-Jee	30	Maier, Hans	207
Lee, J.H.	85	Majerus, P.	33
Lee, J.S.	195	Makhankov, A.	163
Lee, Sang Pill	171	Makhankov, A.	233
Leenaers, A.	72	Makita, Junichi.	43
Legarda, F.	84	Maksimkin, O. P.	176, 225
Leguey, T.	136	Malerba, L.	26, 29
León, M.	28, 32, 94, 214	Maloy, Stuart A.	74
Leont'eva-Smirnova, M.V.	10	Mancini, A.	237
Leonteva-Smirnova, M.V.	86	Marian, J.	27
Leontyeva-Smirnova, M.V.	77	Marmy, Pierre.	1
Levchuk, D.	197, 204	Martínez, E.	27
Levchuk, S.	83	Masaki, K.	40, 151, 185, 197, 236
Lewinsohn, C.A.	170	Masaki, Kei.	205
LHD Experimental Group	223	Massaut, V.	153
Li, C.	86, 157, 222	Masuda, J.	69
Li, Huailin	189	Masuzaki, S.	223
Li, Meimei.	48, 82, 177	Mathon, M.H.	134
Li, Y.	82, 87, 222	Matsubara, N.	118
Libera, S.	237	Matsuda, Y.	194
Lindau, R.	97, 138, 192	Matsuhira, K.	189
Lindig, S.	83, 234	Matsui, H.	19, 67, 70, 87, 95, 124, 145, 226
Linke, J.	33, 239	Matsui, Hideki.	7, 63
Lipa, M.	32	Matsukawa, M.	160
Litnovsky, A.	107	Matsukawa, S.	14, 228
Litunovsky, N.	242	Matsukawa, Yoshitaka	18
Liu, S.	157	Matsuyama, M.	71, 201
Liu, Xiang	240	Matui, H.	65
Loarte, A.	241	Mazul, I.	163, 233, 242
Loginov, N.	129	McClintock, D.A.	192
López, F. J.	210	McNaney, J.	16
Lorenzetto, P.	242	Melder, R.	242
Loughlin, M. J.	131, 151	Merola, M.	232, 238
Lu, L.	40	Metals and Ceramics Division	
Lu, Z.	141	Oak Ridge National Laboratory	1
Lucon, E.	216	Metelkin, E.V.	95
Lucon, Enrico	11	Meyder, R.	202
Lukyanenko, A.	224	Miao P.	85, 88, 140, 190, 191

Mikheyev, A.....	129	Murayama, T.....	170
Miller, R.....	140	Muroga, T.....	50, 64, 69, 70, 71, 82, 224
Miller, Robert.....	193	Muroga, Takeo.....	63, 113, 189, 207
Mills, B.E.....	200	Murooka, K.....	158
Mironova, E. G.....	11	Mutzke, A.....	78
Miskiewicz, M.....	36		
Missirlianv, M.....	238	Nagai, Y.....	193
Miura, Y.....	185	Nagakawa, Johsei.....	5, 122, 144
Miwa, S.....	120	Nagao, Y.....	241
Miwa, Y.....	241	Nagasaka, T.....	50, 64, 69, 71, 82
Miwa, Yukio.....	218	Nagasaka, Takuya.....	63, 189
Miya, N.....	40, 151, 185, 197, 236	Nagase, Hiroyasu.....	154
Miya, Naoyuki.....	242	Nagata, S.....	20, 72, 110, 158, 211, 214, 215
Miyamoto, M.....	20, 24	Nägele, W.....	200
Miyamoto, Mitsutaka.....	84	Naito, A.....	109
Miyauchi, H.....	35	Nakahata, T.....	35
Miyauchi, Hideo.....	239	Nakahata, Toshihiko.....	235, 242
Miyawaki, K.....	124	Nakai, T.....	138
Miyazaki, Hiroyuki.....	209	Nakai, K.....	65
Miyazaki, Shintaro.....	155	Nakamura, H.....	126, 129
Mizuno, Nobukazu.....	202	Nakamura, Hirofumi.....	199, 205
Mizuta, S.....	81	Nakamura, Hiroo.....	132
Moeslang, A.....	37, 233	Nakamura, Kazuyuki.....	139
Moilanen, P.....	37, 141	Nakamura, Y.....	69, 71
Mollá, J.....	94, 214	Nakano, Hiromi.....	112
Monge, M. A.....	136	Nakano, J.....	241
Montanari, R.....	229	Nakano, Junichi.....	220
Morgan, S.....	106	Nakashima, Kazuo.....	41
Mori, H.....	101	Nakata, T.....	7
Morioka, A.....	160, 185	Nakazawa, T.....	109
Morishita, K.....	118, 123	Namba, Haruyuki.....	152
Moriyama, H.....	118	Nanstad, RandyK.....	1
Moroño, A.....	211	Narita, T.....	194
Morozov, V.....	129	Narui, M.....	72, 87, 110
Moser, J. L.....	224	Neu, R.....	34
Möslang, A.....	31, 97, 133, 138, 192	Neumann, M.....	33
Mota, F.....	28, 32, 94, 214	Neustroev, V. S.....	219
Motojima, O.....	223	Ngayasu, Ryo.....	113
Mukai, Hideki.....	168	Nikolaev, G.N.....	124
Munakata, K.....	45	Nishi, M.F.....	80
Muñoz, A.....	136	Nishijima, S.....	152
Murakawa, H.....	170	Nishikawa, M.....	189
Murase, Y.....	5	Nishikawa, Masabumi.....	41, 152, 154, 202, 206
Murase, Yoshiharu.....	122, 144	Nishikawa, Masahiro.....	36
Murata, M.....	139	Nishikawa, Y.....	35

Nishikawa, Yusuke	235	Ohnuki, S.....	64, 139, 191
Nishimura, A.....	152	Ohnuma, Toshiharu.....	61, 93
Nishimura, Arata	189	Ohsawa, Kazuhito	22
Nishimura, K.....	223	Ohtsuka, H.	228
Nishimura, Kiyohiko.....	242	Ohtsuka, S.....	135, 194, 195
Nishitani, T.	126, 152, 160, 215	Ohya, Kaoru	240
Nishitani, Takeo	154, 205	Oikawa, Akira	205
Nishiuchi, T.	118	Oka, K.	119, 139
Nita Nobuyasu.....	63	Okada, Jun	240
Nita, N.	67, 124, 145, 226	Okano, K.	40
Niwase, K.	26, 61	Okita, T.....	173
Nobuta, Y.	223	Okita, Taira.....	94
Noda, N.....	35, 223	Oku, D.	68
Noda, Nobuaki	239	Okubo, N.	14, 15, 19
Noda, Tetsuji.....	164	Okubo, Nariaki.....	183
Noda, Tomohiro	62	Okuniewski, Maria A.....	74
Nodaa, Tetsuji	212	Okuno, K.	35, 40, 160, 198, 236
Nogiwa, K.	145	Okuno, Kenji.....	235, 239, 242
Norajitra, P.	163, 233	Oliveri, E.	108
Nordlund, K.	78	Onishi, Y.....	40, 236
Noronha, S. J.	29	Ono, K.	24, 101
Novak, S.....	56	Ono, Kotaro	83
Novikov, V.V.....	86	Ooms, Hans	207
Novovic, M.	141	Ooms, L.....	153
Nozawa, Takashi.....	165, 169	Osetsky, Yu .N.	2, 18, 24, 71, 92, 219
Nygren, R.E.....	234	Oshima, R.	102
Obushev, A.N.....	217	Otsuka, T.....	69
Ochiai, K.....	152	Ovcharenko, A.M.....	117
Ochiai, Kentaro	154, 205	Ovchinnikov, I.....	233
Oda, Takuji.....	111, 155, 199	Ovchinnikov, S.V.....	66
Oda, S.	65	Oya, Y.	35, 40, 198, 236
Odette, G. R.	4, 80, 85, 87, 88, 103, 123, 140, 143, 148, 186, 190, 191, 193, 226, 230	Oya, Yasuhisa	111, 155, 199, 235, 239, 242
Odette, R.....	159	Oyaidzu, M.	40, 236
Odette, Robert.....	193	Oyaidzu, Makoto	235, 239, 242
Odrizola, J. A.....	137	Ozawa, Kazumi	55, 165
Ogawa, T.....	39	Pan, Xiao.....	177
Ogawa, Y.	40	Panchenko, V. L.	173
Ogiwara, H.	8, 135	Papp, Daniel.....	114
Ogiwara, Hiroyuki.....	187	Pareja1, R.	136
Ogorodnikova, O.	239	Park, J.Y.....	166
Ohkubo, Hideaki.....	62	Park, Joon-Soo.....	166, 171
Ohkubo, T.	67	Park, K. H.	52, 142, 166
Ohmi, M.....	241	Paúl, A.	110, 137
		Peacock, A.	174
		Pellettieri, A.	43

Peña, A.	84	Ricapito, I.	202
Peng, L.	87	Ricapito, Italo.....	198
Penzhorn, R.-D.	204	Riccardi, B.	84, 229
Perlado, J. M.	27, 28, 32, 94, 214	Rieth, M.	192, 233
Pestchanyi, S.E.	241	Rieth, Michael.....	47, 89, 172
Petersen, C.	161	Roberts, S.G.....	143
Petti, D.	198	Robertson, Ian M.	30
Philipps, V.	36, 107	Rodchenkov, B.S.....	147, 217
Phillipp, F.	26	Rödiger, M.	33, 239
Pichel, Mónica León.....	210	Rodin, M.V.....	217
Pilloni, L.	86	Rolli, R.	229
Pilloni, Luciano	125	Romanoski, Glenn.....	17
Pinaev, S.S.	108	Romanov, P.V.	156
Pinhero, P. J.	44	Romanov, V.A.	21, 25
Pinna, T.	130	Romanovskij, V.N.	156
Pint, B. A.	224	Rong, Zhouwen.....	2
Pint, Bruce A.	207	Rowcliffe, A.F.	64, 66
Pinzhin, Yu.P.	66	Rubel, M.	107
Pisarek, M.	36	Rubel, M.J.	36
Pizzuto, A.	237	Ryazanov, A.I.	95
Plini, P.	229	Ryu, W.S.	166
Podgornova, I.V.	216	Ryu, Woo Seog.....	185
Pokrovsky, A.	105	Sabau, Adrian.....	17
Pokrovsky, A.S.	46, 149, 150, 174	Sagara, A.	198, 223
Porollo, S. I.	11, 177	Sagara, Akio.....	239
Portnykh, I. A.	173	Sagaradze, V.V.	216
Potapenko, M.M.	10, 66	Saikaly, W.	32
Pütterich, T.	34	Saito, T.	241
Puzzolante, J.L.	216	Saitoh, Shigeru.....	199
Qiang, Li.....	231	Sakamoto, M.	20
Rabaglino, E.	43, 72	Sakasegawa, H.	8, 135
Radel, R.F.	16	Sakasegawa, Hideo.....	187
Rai, A.	78	Sakawa, Y.	206
Ramar, A.	194	Sakizono, Daisuke.....	36
Rascón, A.	210	Sakurai, S.	160, 185
Reimann, J.	202	Salonen, E.	78
Remington, B.A.	16	Salvo, M.	232
Rensman, J. W.	148	Samaras, M.	90
Rensman, J.	106, 186, 188, 230	Sannen, L.	43, 72
Rensman, J-W.	190	Sanz, J.	127
Reusch, F.	227	Sasajima, T.	185
Reviznikov, L.I.	86	Sasaki, T.	118
Reyes, S.	127	Sato, Fuminobu.....	113
		Sato, K.	22, 100, 206

Sato, M.....	119	Shimizu, M.....	241
Sato, S.	160	Shimoda, Kazuya	102
Sato, Satoshi.....	154	Shin, Byung Chal.....	171
Sato, T.....	173	Shinohara, K.....	185
Satoh, Y.	19, 226	Shinozuka, Kei	139, 187
Satou, M.....	48, 49, 50, 170	Shishulin, V.	129
Sawada, Akihiko.....	113, 207	Shu, W. M.....	80
Sawada, T.	49	Silnaygina, N. S.....	225
Sawai, T.	14, 15, 139, 185	Simakov, S. P.....	128, 133
Sawai, Tomotsugu.....	82, 183	Simakov, S.	133
Sawamura, Isao	36	Simpson, M. F.	44
Schaeublin, R.....	194	Simpson, M.	198
Schaeublin, Robin	77	Singh, B. N.	23, 25, 37, 76, 92, 98, 117, 141
Schäublin, R.....	28, 91, 96, 217	Singh, Bachu.....	92, 176, 177
Schedler, B.....	238	Singh, M.	170
Schmalz, F.	106	Sirch, M.	204
Schmitz, O.....	107	Sivak, A.B.....	21, 25, 173
Schneider, H.-C.....	6, 229	Skinner, C.H.....	200
Schneider, R.	78	Smith, R.....	16, 141
Scibetta, M.	43, 72, 216	Smolik, G. R.....	44, 198
Scibetta, Marc	11	Snead, L. L.....	4, 47, 54, 120
Seeger, A.	26	Snead, Lance L.	17, 57, 169, 227
Sekimura, N.	173	Sokolo, Mikhail A.....	1, 12
Sekimura, Naoto	94, 142	Sokolov, M. A.	13, 81, 146, 192
Seletskiaia, T.	71	Solonin, M.I.	86
Semenov, A.V.	108	Son, S. J.....	104
Semenov, E.A.	95	Soneda, Naoki.....	61, 93
Sergienko, G.	107	Song, B.J.....	185
Serizawa, H.....	170	Song, Yin.....	23
Serra, Anna	18	Spätig, P.....	14, 83, 96, 148
Serrano, M.	135	Sporea, Adelina.....	213
Shamardin, V.E.....	86	Sporea, Dan	213
Sharafat, S.	159	Srivilliputhur, Srinivasan G.	74
Sharpe, P.	198	Stijker, M.P.	111
Shen, Wei-Ping	38	Stijker, M.S.	42
Shepelyev, Oleksandr	142	Stocks, G. M.....	71
Shestakov, V.	65, 203	Stoller R.E.	17, 18, 71, 98, 117, 192
Shiba, K.....	7, 8	Strebkov, Y.S.	147, 217
Shiba, Kiyoyuki.....	82, 139, 184	Stroller, R.E.....	15
Shibahara, T.....	40, 206, 236	Stubbins, James F.....	74
Shibayama, T.	23	Stubbins, James.....	177
Shikama, T.....	72, 110, 158, 170, 211, 214, 215	Suda, T.....	64, 139
Shikov, A.	104	Sugano, R.	123
Shikov, A.K.....	66	Sugimoto, M.	126, 189
Shim, J.H.....	31	Sugiyama, H.....	201
Shim, Jae-Hyeok.....	90	Sugiyama, K.....	40, 197, 206, 236

Sugiyama, Masanari.....	7	Tazhibayeva, I.....	65, 203
Sumino, Y.....	99	ten Pierick, P.....	42, 106, 169, 188
Sun, Youmei.....	23	Terai, T.....	198
Sunyk, R.....	105, 227	Terai, Takayuki.....	41, 207
Sutton A P.....	30, 91	Terentyev, D.....	29
Suzuki, Hiroshi.....	164	Thaveethavorn, Somsri.....	164
Suzuki, A.....	204	Thomsen, E.....	53
Suzuki, A.....	224	Tivanova, O. V.....	225
Suzuki, Akihiro.....	113, 207	Toda, Naoki.....	196
Suzuki, S.....	231	Toda, Yoshitomo.....	168
Suzuki, T.....	211	Toh, K.....	72, 110, 211, 214, 215
Suzuki, Y.....	185	Tokitani, M.....	20
Suzukia, Hiroshi.....	212	Tokitani, Masayuki.....	235
Swe, Than.....	77, 121	Tokunaga, K.....	20, 75, 231
Sze, D.-K.....	198	Toloczko, M.B.....	49
Tabarés, F.L.....	201	Tomita, T.....	15, 19
Tachi, Yoshiaki.....	112	Torralva, B.....	16
Tafalla, D.....	201	TRIAM group.....	20
Taguch, T.....	54, 120, 167	Trinkau, s H.....	23, 25, 98, 117
Tähtinen, S.....	37, 141	Tsai, K. V.....	176
Takada, F.....	15, 119, 241	Tsushima, Hidetsugu.....	97
Takagi, I.....	118	Tsuchiya, B.....	20, 72, 110, 158, 214, 211, 215
Takahashi, H.....	88	Tsukada, T.....	241
Takahashi, R.....	185	Tsukada, Takashi.....	218, 220
Takata, Hiroki.....	152, 202	Tsuzuki, K.....	151
Takeishi, Toshiharu.....	152, 206	Tsuzuki, K.....	89
Takemoto, K.....	89	Tsvelev, V.V.....	86
Takenaka, T.....	102	Turubarova, L. G.....	176
Tamai, H.....	160	Tyumentsev, A.N.....	66
Tamura, Manabu.....	139, 187	Uchida, M.....	45
Tamura, S.....	70	Ueda, Y.....	189
Tanabe, T.....	40, 69, 102, 151, 197, 206, 236	Ueda, Yoshio.....	36
Tanabe, Tetsuo.....	205	Ueno, Keiko.....	144
Tanaka, K.....	39	Ukaï, S.....	81, 134, 135, 138, 191, 194, 195, 196
Tanaka, M.....	143	Urra, I.....	84
Tanaka, S.....	236	Uwaba, T.....	138
Tanaka, Satoru.....	111, 155, 199	Valli, M.....	97
Tanaka, Teruya.....	113	van den, Berghe S.....	72
Tanigawa, H.....	8, 15, 81, 135, 139, 146	van der Laan, J.G.....	42, 106, 111, 169
Tanigawa, Hiroyasu.....	12, 82, 184, 187	Van Petegem, S.....	96
Taniwaki, T.....	101	Van Swygenhoven, H.....	16, 90
Tao, Shunyan.....	240	Vatulin, A.V.....	86
Tavassoli, A-A. F.....	163		
Taylor, N.....	160		

Velarde, M.....	32	Wirth, B. D.....	31, 80, 103, 119, 193
Vella, G.....	108	Wirth, Brian D.....	4, 30, 90, 118
Ventelon, L.....	119	Wolfer, Wilhelm.....	94
Ventelon, Lisa.....	118	Wong, K.L.....	31
Verpoucke, G.....	72	Wu, Xianglin.....	177
Vertkov, A.V.....	112	Wu, Y.....	15, 82, 87, 157222
Verzilov, Y.....	160		
Victoria, M.....	16, 27, 90	Xu, Q.....	22, 34, 96, 100
Vila, R.....	210	Xu, Zengyu.....	240
Visca, Eliseo.....	237	Yablokov, N.....	163
Vladimirov, P.....	31, 133		
Vlasov, V.V.....	124	Yagyu, J.....	236
von der Weth, Axel.....	9	Yagyu, Jyunichi.....	242
Voskoboinikov, Roman E.....	2, 219	Yamada, H.....	160
Vreeling, J. A.....	188	Yamada, R.....	167
		Yamada, T.....	68
Wakabayashi, Y.....	85	Yamagata, I.....	19
Wakai, E.....	14, 15, 19, 54, 102, 119, 120, 228	Yamamoto, N.....	5
Wakai, Eiichi.....	183	Yamamoto, Norikazu.....	122, 144
Walker, F.J.....	17	Yamamoto, T.....	4, 15, 80, 85, 87, 88, 123, 159, 186, 190, 191, 226, 230
Wampler, W.R.....	200	Yamanishi, Toshihiko.....	199
Wan, Farong.....	12	Yamashita, S.....	88, 139, 191
Wan, L.....	71	Yamauchi, M.....	126, 215
Wang, M.X.....	35	Yamauchi, Michinori.....	154
Wang, W.....	157	Yamauchi, Y.....	51, 68, 89
Wang, Y.M.....	16	Yamaya, Kousuke.....	3
Wang, Zhiqiang.....	92	Yamazaki, M.....	72, 87, 110
Ward, D.....	160	Yamazaki, Saishun.....	3, 208
Warrier, M.....	78	Yamazaki, Takeshi.....	205
Was, G.S.....	20	Yang, W.J.....	85, 87, 88, 230
Wasastjerna, F.....	128	Yang, Wen.....	164, 212
Wasastjerna, F.....	133	Yano, Toyohiko.....	3, 112, 168, 208, 209
Watanabe, H.....	96, 99	Yano, Y.....	88
Watanabe, K.....	71, 201	Yao, Z.....	91, 217, 224
Watanabe, S.....	64, 139	Yashiki K.....	64
Watanabe, Yoshiyuki.....	79	Yasuda, R.....	186, 230
Webster, A.J.....	21	Yasuda, T.....	64
Wei ping, Shen.....	231	Yeh, Juen-Wei.....	103
Weick, M.....	161	Yeliseyeva, O.....	224
Wen, Ming.....	92	Ying, A.....	107
Wienhold, P.....	107	Ying, Alice.....	109, 114
Wiffen, F.W.....	47	Yokoyama, Sumi.....	199
Wilkinson, A.J.....	143	Yoltukhovskiy, A.G.....	77
Williams, S.....	106	Yoon, Han Ki.....	171
Wilson, P.P.H.....	128	Yoshida, Katsumi.....	168
Win, Myo Htet.....	77		

Yoshida, N.....	20, 34, 70, 73, 75, 99, 231	Zeep, B.....	233
Yoshida, Naoaki	79, 235	Zeng, Zi-Huai.....	56
Yoshida, S.....	160	Zhang, Chonghong.....	23
Yoshida1, N.....	123	Zhang, M.	87, 222
Yoshiie, T.	22, 34, 96, 100	Zheng, S.....	40, 157
Yoshikawa, A.	40, 236	Zhong, Zhi-hong	35, 38, 235
Yoshikawa, Akira.....	239, 242	Zhou, Zhang-jian	35, 38, 235
Yoshitake, T.	88	Zhou, Zhongfu	30, 91
You, J. H.	234	Zielinski, W.	36
Youchison, D. L.....	234	Zinkle S. J.....	47, 48, 98, 117, 159
Young, C.M.	64	Zinkle, Steven J.....	18, 172
Youngblood, G. E.	53	Zouev, Yu. N.	216
Yu, G.	96	Zschack, P.....	17
Yu, Gang.....	12	Zucchetti, M.....	156
Yu, Jinnan	12, 77	Zucchetti, Massimo	198
Yutani, K.....	140	Zushi, H.....	20
Yutani, K.....	76		

The logo for ICFRM-12 Santa Barbara is located in the top-left corner. It consists of a dark blue rectangular box containing a white starburst icon on the left, followed by the text "ICFRM-12" in a large, bold, white sans-serif font, and "SANTA BARBARA" in a smaller, white sans-serif font below it.

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