IRRADIATION EFFECTS ON TENSILE PROPERTIES OF HIGH-CHROMIUM FERRITIC/MARTENSITIC STEELS—R. L. Klueh (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this work is to develop an understanding of the effect of irradiation on fracture behavior of reduced-activation ferritic/martensitic steels of interest for fusion applications and to use that knowledge to develop steels with improved properties.

SUMMARY

Tensile specimens of four ferritic/martensitic steels were irradiated at 390-395ºC in the Experimental Breeder Reactor (EBR-II) to 32-33 dpa. The steels were the ORNL reduced-activation 9Cr-2WVTa and that steel containing 2% Ni (9Cr-2WVTa-2Ni), modified 9Cr-1Mo, and Sandvik HT9 (12Cr-1MoVW). The 9Cr-2WVTa and 9Cr-2WVTa-Ni were irradiated after normalizing and tempering some specimens 1 hr at 700ºC and some specimens 1 h at 750ºC; the 9Cr-1MoVNb and 12Cr-1MoVW were tempered 1 h at 760ºC. Based on the change in tensile properties, the results demonstrated the superiority of the 9Cr-2WVTa steel over the two commercial steels. Charpy properties of the 9Cr-2WVTa-2Ni steel were similar to those of the 9Cr-2WVTa steel, indicating no adverse effect of the nickel on the properties after irradiation in a fast reactor around 400ºC.

PROGRESS AND STATUS

Introduction

The 9Cr reduced-activation ferritic/martensitic steels are candidates for applications as first wall and blanket structural materials for future fusion reactors. Displacement damage by neutron irradiation of these types of steel below 425-450ºC hardens the steel lattice, causing an increase in strength and a decrease in toughness. The effect on impact toughness is measured in a Charpy test as an increase in the ductile-brittle transition temperature (DBTT) and a decrease in the upper-shelf energy (USE).

The possible effect of helium on hardening and embrittlement is important because large amounts of transmutation helium will form in the ferritic/martensitic steel first wall of a fusion reactor. Nickel-doped 9 and 12 Cr steels have been irradiated in a mixed-spectrum reactor such as the High Flux Isotope Reactor (HFIR) to study the effect of helium on fracture [1]. Helium (an alpha-particle) is formed in a mixed-spectrum reactor by a two-step transmutation reaction between ^{58}Ni and thermal neutrons in the mixed-neutron spectrum. This technique allows for the simultaneous production of displacement damage and helium in the steel matrix, thus simulating what will happen in a first wall. Results from such irradiation experiments at 400ºC have been interpreted to indicate an effect of helium on embrittlement. This conclusion was based on the comparison of the steels with and without nickel and on the differences between the behavior of the nickel-doped steels in a mixed-spectrum reactor, where considerable helium forms, and in a fast reactor, where little helium forms [1].

More-recent irradiation experiments of nicked-doped 9Cr reduced-activation steels have indicated that the nickel-doped steels hardened more than steels without nickel [2,3]. A 9Cr-2W steel with and without 1% Ni was irradiated in the Japanese Materials Test Reactor (JMTR) to 0.15 dpa at 170ºC, and an increase in the room temperature yield stress of up to 350 MPa was observed for the nickel-containing steel, compared to a 120 MPa increase for the steel without nickel. However, no difference in the strength increases was observed for steels irradiated at 220ºC [2]. Irradiation of the steels to 2.2 and 3.8 dpa at 270 and 348ºC, respectively, in the Advanced Test Reactor (ATR) indicated that the nickel-containing steel hardened about 20% more than the steel without nickel at 270ºC, but strengths were similar after irradiation at 348ºC [3]. Also, a larger shift in DBTT occurred for the nickel-containing steel than the one without nickel when irradiated at 270ºC, but not after irradiation at 348ºC. TEM analysis indicated that nickel refined the size of the irradiation-produced defect clusters, which were more numerous in the nickel-containing steel [3].

These results indicate that the nickel-doping simulation technique should be used below about 300ºC with caution, if at all. The results using nickel doping that most strongly indicated that helium caused an increase in the DBTT above that caused by displacement damage alone were on steels irradiated in HFIR to high fluence at 400ºC [1]. Tensile tests of specimens irradiated under similar conditions gave no indication of hardening due to helium (or nickel) [1]. Also, when the same steels were irradiated at similar conditions in the Fast Flux Test Facility (FFTF), no difference was observed in the embrittlement of the nickel-doped and undoped specimens [4]. Transmission electron microscopy (TEM) studies of nickel-doped steels irradiated in HFIR and FFTF showed that a high density of M_6C formed in the nickel-doped steel but not in the undoped steel [5]. Since the shift in DBTT in HFIR, where helium forms, was larger than in FFTF, where little helium forms, the results were taken to mean that helium caused the shift [1].

In this report, tensile properties are reported for the reduced-activation steel ORNL 9Cr-2WVTa and this steel containing 2% Ni (9Cr-2WVTa-2Ni) after irradiation in the Experimental Breeder Reactor (EBR-II). The commercial non-reduced-activation steels modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) steels were also irradiated and tested. In a previous report, the Charpy properties of these same steels irradiated in EBR-II were reported [6].

Experimental Procedure

Compositions and designations of the steels used in this experiment are given in Table 1. In the original Oak Ridge National Laboratory (ORNL) alloy development program for development of reduced-activation steels [7], an 18-kg heat of the electroslag-remelted heat of the 9Cr-2WVTa steel was prepared by Combustion Engineering Inc, Chattanooga, TN. Material from that heat was used as the master alloy to prepare 450-g vacuum arc-melted button heats of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels. The 9Cr-2WVTa heat was a remelt of the master alloy so that the steels could be compared after similar processing.

Table 1. Chemical composition of the steels tested

Element[a]	9Cr-2WVTa	9Cr-2WVTa-2Ni	9Cr-1MoVNb[b]	12Cr-1MoVW[c]
C	0.098	0.098	0.092	0.20
Si	0.19	0.19	0.15	0.17
Mn	0.39	0.38	0.48	0.57
P	0.014	0.014	0.012	0.016
S	0.003	0.003	0.004	0.003
Cr	8.71	8.55	8.32	12.1
Mo	<0.01	<0.01	0.86	1.04
W	2.17	2.15	<0.01	0.61
Ni	0.02	2.01	0.09	0.51
V	0.23	0.23	0.20	0.29
Nb	<0.01	<0.01	0.06	<0.001
Ta	0.06	0.06		
N	0.016	0.016	0.054	0.027

[a]Balance iron
[b]Modified 9Cr-1Mo steel
[c]Sandvik HT9

The small heats were cast as 25.4 mm x12.7mm x 152 mm ingots, after which they were rolled to 6.4-mm plate and 0.76 mm sheet. The steels were normalized by austenitizing for 0.5 h at 1050ºC in a helium atmosphere and quickly cooled in flowing helium. Specimens were irradiated in two tempered conditions: 1 h at 700ºC and 1 h at 750ºC.

The modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) steels were from large commercial-size heats that have been irradiated previously and were included in this experiment as benchmarks for the reduced-activation steels.

Tensile specimens 44.5-mm long with a reduced gage section of 20.3 x 1.52 x 0.76 mm were machined from the 0.76-mm sheet with gage lengths parallel to the rolling direction. Specimens were heat treated after machining. Tests were conducted on irradiated and unirradiated specimens at 400ºC (near the irradiation temperature) in vacuum on a 44-kN Instron universal testing machine at a nominal strain rate of $4 \times 10^{-4}\ s^{-1}$.

Two tensile specimens of each heat and each heat-treated condition were irradiated in the COBRA experiment in EBR-II at temperatures of 390 to 395ºC. Fluence was determined from flux monitors in the irradiation canisters. There was some variation for different specimens, depending on their position in the canisters, but the individual sets of specimens for a given steel and heat treatment were kept together in the canisters and experienced the same irradiation conditions. Specimens were irradiated to 6.7×10^{26} to $6.9 \times 10^{26}\ n/m^2$ (E>0.1 MeV), which produced between 32 and 33 dpa. Helium concentrations were calculated to be between about 3 to 6 appm, depending on the dose and composition (the 6 dpa was for the steel containing 2% Ni).

Results

Irradiated specimens were tested at 400ºC, which is near the irradiation temperature (reported as 390-395ºC). Tensile data for the unirradiated and irradiated steels are summarized in Table 2.

First, the results for the reduced-activation 9Cr-2WVTa steel with and without nickel will be discussed to examine the effect of tempering and the effect of nickel. After that, results for the 9Cr-2WVTa steels will be compared with the commercial modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW) steels.

Table 2. Tensile data for unirradiated and irradiated steels tested at 400ºC

Steel	Temper	Irradiation	Strength (MPa)		Elongation (%)	
			Yield	Ultimate	Uniform	Total
9Cr-2WVTa	700ºC	Unirradiated	701	758	2.7	9.7
9Cr-2WVTa	700ºC	390ºC/32.6dpa	698	732	1.2	6.5
9Cr-2WVTa	750ºC	Unirradiated	517	595	3.3	11.0
9Cr-2WVTa	750ºC	390ºC/32.6dpa	613	645	1.3	7.1
9Cr-2WVTa -2Ni	700ºC	Unirradiated	707	788	2.7	11.3
9Cr-2WVTa -2Ni	700ºC	390ºC/32.6dpa	649	690	3.4	10.0
9Cr-2WVTa -2Ni	750ºC	Unirradiated	674	787	2.7	8.3
9Cr-2WVTa -2Ni	750ºC	390ºC/32.6dpa	637	699	3.4	10.8
9Cr-1MoVNb	760ºC	Unirradiated	562	636	1.4	4.6
9Cr-1MoVNb	760ºC	395ºC/32.1dpa	612	626	0.7	5.3
12Cr-1MoVW	760ºC	Unirradiated	558	697	1.9	4.3
12Cr-1MoVW	760ºC	395ºC/32.1dpa	720	776	1.7	4.0

9Cr2WVTa and 9Cr-2WVTa-2Ni Steels

Figure 1 shows the yield stress and ultimate tensile stress for the 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels given the different tempers before and after irradiation. There appears to be little effect of irradiation on strength for the 9Cr-2WVTa for the 700ºC temper and on the 9Cr-2WVTa-2Ni for both tempering conditions. There is a small amount of irradiation hardening for the 9Cr-2WVTa after the 750ºC temper.

The effect of irradiation on ductility (Fig. 2) appears to be greater for the 9Cr-2WVTa than the 9Cr-2WVTa-2Ni. For both tempering conditions, uniform elongation of the steel containing nickel increased after irradiation, while that for the steel without nickel decreased. Total elongation of the 9Cr-2WVTa also

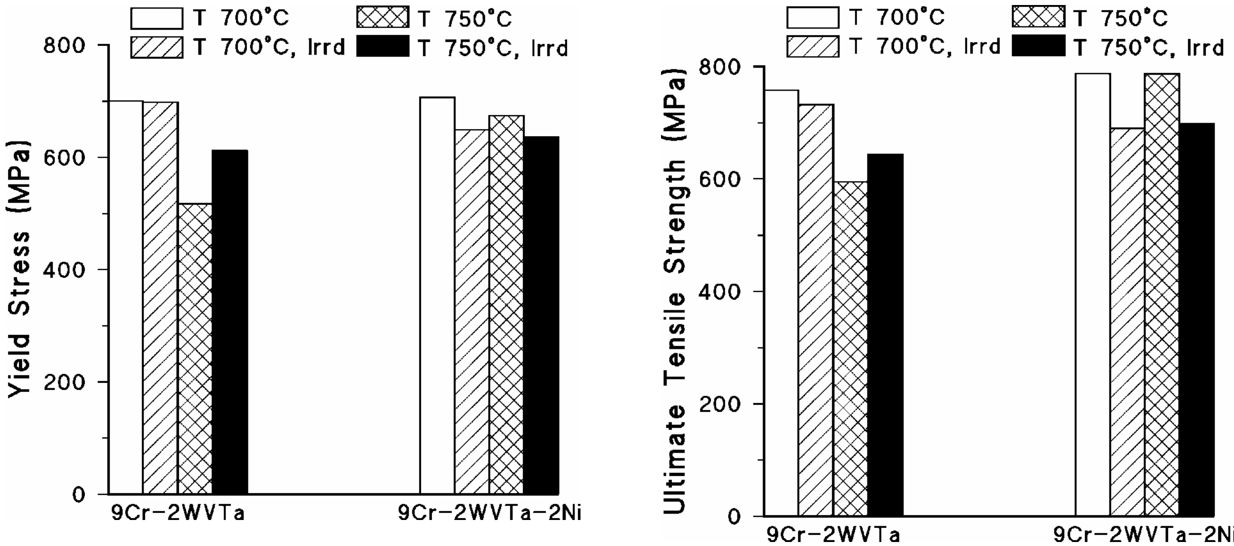

Fig. 1. The yield stress (left) and ultimate tensile strength (right) of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

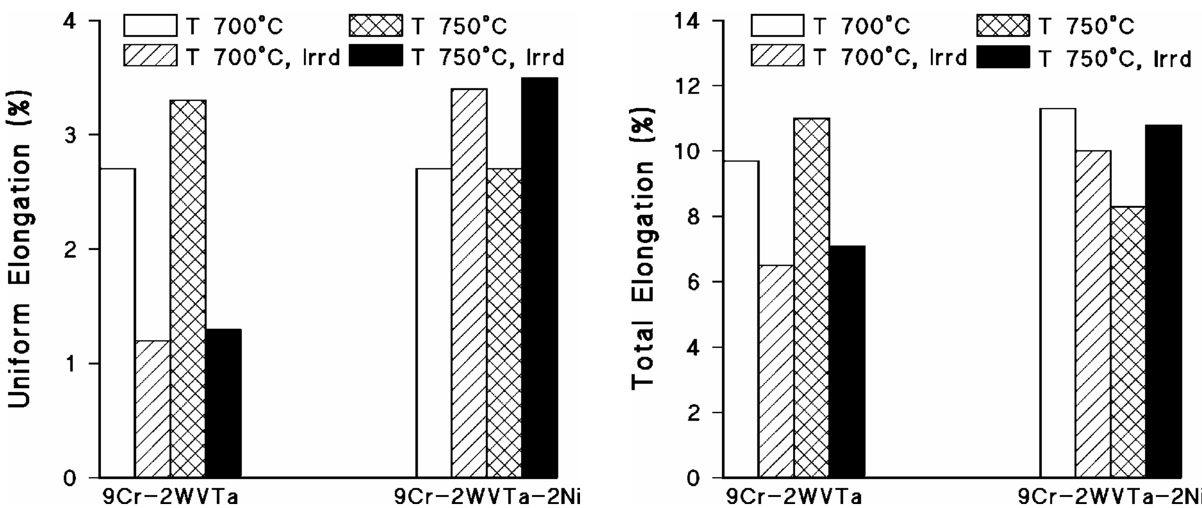

Fig. 2. The uniform elongation (left) and total elongation (right) of 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels for two different tempering conditions before and after irradiation in EBR-II.

Decreased after irradiation for both tempering conditions, but the total elongation for the 9Cr-2WVTa-2Ni decreased slightly for the steel tempered at 700ºC and increased slightly for the steel tempered at 750ºC.

Comparison of Reduced-Activation and Commercial Steels

Tensile properties of the reduced-activation 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels were compared with those of 9Cr-1MoVNb and 12Cr-1MoVW steels. Comparison was for 9Cr-2WVTa and 9Cr-2WVTa-2Ni steels tempered 1 h at 750ºC and the commercial steels both tempered 1 h at 760ºC, the conventional tempering temperature for 9Cr-1MoVNb. In both the unirradiated and irradiated conditions, there is relatively little difference in strength among the steels (Fig. 3) with somewhat more variation in the ductility (Fig. 4).

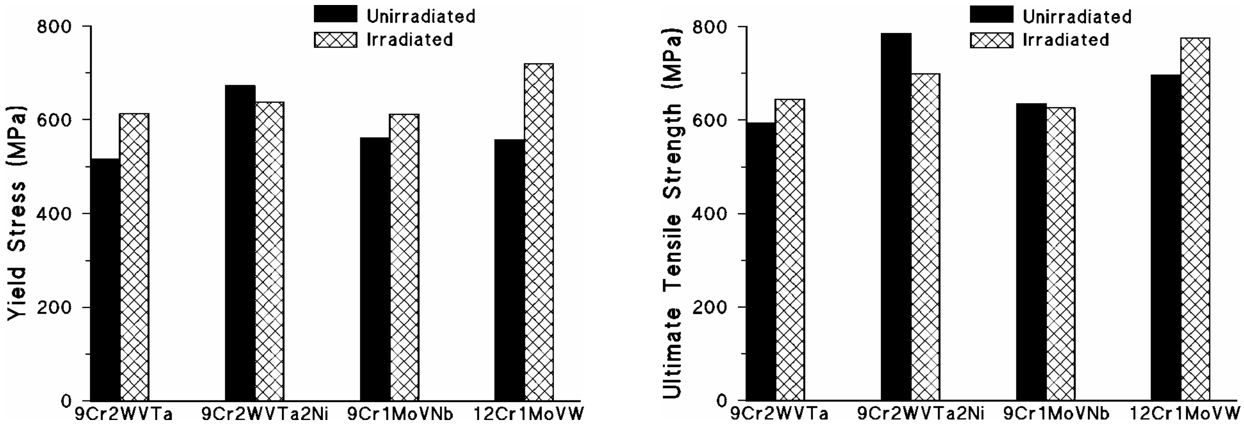

Fig. 3. Yield stress (left) and ultimate tensile strength (right) of unirradiated and irradiated 9Cr-2WVTa, 9Cr-2WVTa-Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels.

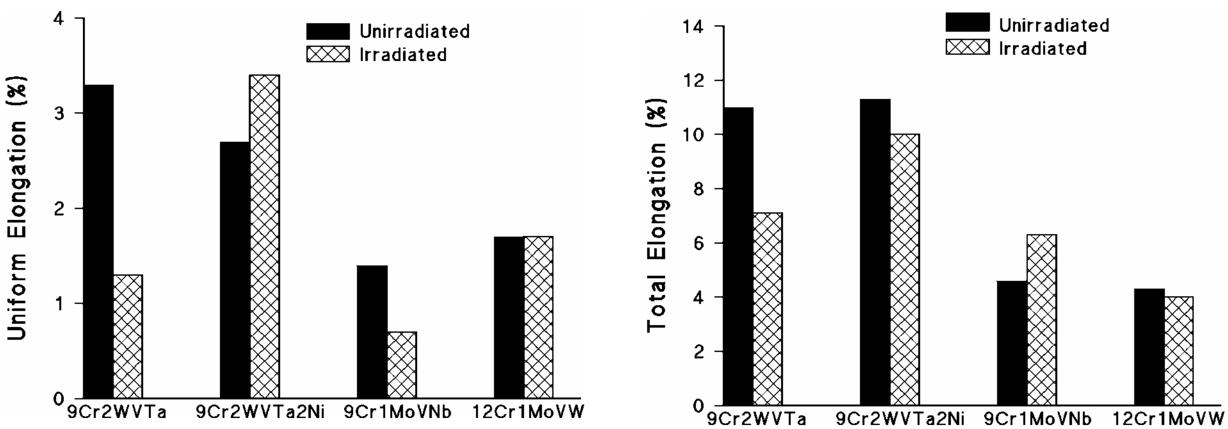

Fig. 4. Uniform (left) and total (right) elongation of unirradiated and irradiated 9Cr-2WVTa, 9Cr-2WVTa-Ni, modified 9Cr-1Mo (9Cr-1MoVNb), and Sandvik HT9 (12Cr-1MoVW) steels.

Both the yield stress and ultimate tensile strength indicate that relatively minor irradiation hardening occurred and, in the case of 9Cr-2WVTa-2Ni, softening occurred (Fig. 3). This is reflected in the ductility (Fig. 4), where the uniform elongation of the 9Cr-2WVTa-2Ni steel and the total elongation of the 9Cr-1MoVNb increased after irradiation. This steel had the highest uniform and total elongation of the four steels after irradiation. The greatest change in ductility was observed for the 9Cr-2WVTa steel, although after irradiation it had ductility as good as or better than that for 9Cr-1MoVNb and 12Cr-1MoVW. The latter steel showed little change in uniform and total elongation after irradiation.

Discussion

It is well known that one effect of adding nickel, an austenite stabilizing element, to a ferritic steel is a reduction in the Ac_1 temperature, the temperature where ferrite begins to transform to austenite when a steel is heated. If the tempering temperature is above the Ac_1, then any austenite formed during tempering will transform to martensite, and such a "normalized-and-tempered" steel will contain untempered martensite. For the commercial steels in the present investigation, it is known that nickel lowers the Ac_1 below the 750°C tempering temperature [8], and because of the similarity of the commercial and reduced-activation steels it is assumed the Ac_1 for those steels is also below 750°C.

This is verified because the effect of the presence of untempered martensite can be seen in the unirradiated strength of the 9Cr-2WVTa-Ni by comparing the strengths of this steel after the 700 and 750ºC tempers with the strengths of the steel without nickel (Fig. 1). The unirradiated strengths of the 9Cr-2WVTa-2Ni steel after the 700 and 750ºC tempers are similar, compared a large reduction for the 9Cr-2WVTa tempered a 750ºC relative to that tempered at 700ºC.

As discussed above, the primary reason for irradiating 9Cr-2WVTa-2Ni is that reactions between ^{58}Ni and the thermal neutrons in a mixed-spectrum produce helium, thus providing a method to study helium effects. Irradiation of the steel in a fast reactor, where very little helium forms, provides a control for helium-effects studies. Generally, those studies are made on the steels with and without nickel tempered below the Ac_1 to similar strengths [1]. In the present experiment, the 750ºC temper was used because it was decided to investigate the effect of small amounts of untempered martensite.

The results for the 9Cr-2WVTa-2Ni steel provided the most interesting observations. First, before irradiation, the increased strength of the 9Cr-2WVTa-2Ni after the 750ºC temper is not accompanied by a greatly reduced ductility relative to the 9Cr-2WVTa steel (Fig. 2). Second, a most unusual observation after irradiation was that there was essentially no hardening of the 9Cr-2WVTa tempered at 700ºC and no hardening—actually softening—of the 9Cr-2WVTa-2Ni steel. Despite the relatively small irradiation hardening, the uniform and total elongations of the 9Cr-2WVTa steel decreased, whereas the uniform elongation of the 9Cr-2WVTa-2Ni, the steel containing untempered martensite, actually increased for both tempering conditions, as did the total elongation for the steel tempered at 750ºC. A slight decrease in total elongation was observed for the 9Cr-2WVTa-2Ni steel tempered at 700ºC. The ductility of the nickel-containing steel after irradiation was superior to that of the steel without nickel for all conditions.

This unexpected behavior of the 9Cr-2WVTa-2Ni is also indicated when the 750ºC-tempered steels are compared with 9Cr-1MoVNb and 12Cr-1MoVW steels tempered at 760ºC, where the 9Cr-2WVTa-2Ni was the only steel not hardened, although none of the other three steels hardened significantly (Fig. 3). Likewise the 9Cr-2WVTa-2Ni steel had the best ductility after irradiation. The results for the comparison of the different steels demonstrated the excellent behavior of the reduced-activation 9Cr-2WVTa steel relative to the two commercial steels.

The relatively minor hardening—and softening—was unexpected from previous experiments where these steels were irradiated at ≈400ºC [9-11]. A possible explanation may be found in results from previous studies that found a peak in irradiation hardening with increasing fluence. This effect was observed for reduced-activation steels [12] and for commercial-type Cr-Mo steels [13,14]. The effect for the reduced-activation steels is shown in Fig. 5 [12].

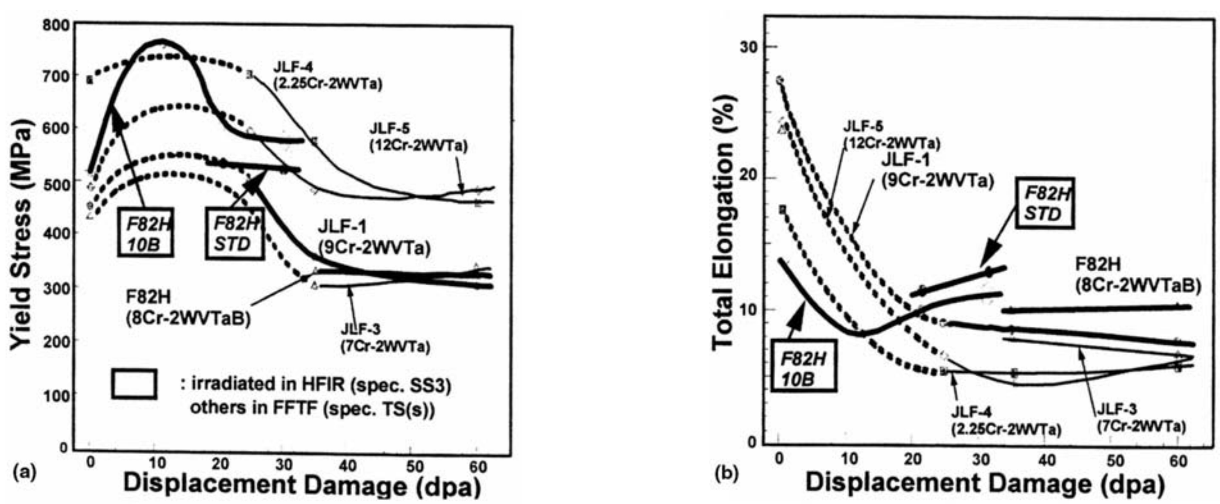

Fig. 5. The (a) yield strength and (b) total elongation as a function of irradiation doses for several reduced-activation steels [11].

The results in Fig. 5 indicate that that the reduction in irradiation hardening with increasing dose begins to approach the unirradiated value near 30 dpa for some of the steels shown, which is similar to the dose of the specimens irradiated in the present experiment that showed this behavior. Total elongation appears to decrease to a plateau around 30 dpa. There is an indication of a minimum in total elongation for one of the steels in Fig 5, but the minimum is indicated somewhere beyond 30 dpa. A possible explanation for the peak in strength is that irradiation-enhanced recovery of the microstructure offsets the irradiation hardening [14].

The positive effect of nickel and the untempered martensite in the irradiated 9Cr-2WVTa-2Ni steel tempered at 750ºC could also be an effect of irradiation-enhanced recovery and, in particular, irradiation-enhanced recovery of the untempered martensite. In this case, the larger rate of recovery of the untempered martensite would partially or wholly offset the irradiation hardening. This assumes that the effect is not due to the nickel, which appears to be a reasonable assumption, since there is relatively little difference in the tensile properties between the 9Cr-2WVTa and 9Cr-2WVTa-2Ni tempered at 700ºC, either before or after irradiation. If this explanation is what is occurring, then one way to improve the irradiation resistance of the reduced-activation 9Cr-2WVTa steel would be to temper above the Ac_1. If the results of the present experiment are then reproduced, a higher-strength steel with less irradiation hardening would result. Since irradiation hardening causes embrittlement, as measured in an Charpy impact test, less embrittlement should occur. The Charpy results previously reported for these steels appear to support this conclusion.

References

[1] R. L. Klueh and D. J. Alexander, J. Nucl. Mater. 187 (1992) 60.
[2] R. Kasada, A. Kimura, H. Matsui, and M. Narui, J. Nucl. Mater. 258-263 (1998) 1199.
[3] A. Kimura, Report of IEA Workshop on Reduced-Activation Ferritic/Martensitic Steels, JAERI-Conf 2001-007, 348.
[4] W. R. Corwin, J. M. Vitek, and R. L. Klueh, J. Nucl Mater. 149 (1987) 312.
[5] P. J. Maziasz, R. L. Klueh, and J. M. Vitek, J. Nucl. Mater.141-143 (1986) 929.
[6] R. L. Klueh and M. A. Sokolov, Fusion Materials Semi-Annual Progress Report for Period Ending June 30, 2003, DOE/ER-0313/34, 63.
[7] R. L. Klueh and P. J. Maziasz, Met. Trans. 20A (1989) 373.
[8] R. L. Klueh, J. M. Vitek, and M. L. Grossbeck, Effects of Radiation on Materials, ASTM STP 782, (American Society for Testing and Materials, Philadelphia, 1982) 648.
[9] R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 132 (1986) 27.
[10] R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 137 (1986) 44.
[11] R. L. Klueh and D. J. Alexander, Effects of Radiation on Materials: 18th International Symposium, ASTM STP 1325 (American Society for Testing and Materials, Philadelphia, 1999) 911.
[12] A. Kohyama, A. Hishinuma, D. S. Gelles, R. L. Klueh, W. Dietz, and K. Ehrlich, J. Nucl. Mater. (1996) 138.
[13] R. L. Klueh and D. J. Alexander, J. Nucl. Mater. 258-253 (1998) 1269.
[14] V. S. Khabarov, A. M. Dvoriashin, and S. I. Porollo, J. Nucl. Mater. 233-237 (1996) 236.