MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AUSTENITIC STAINLESS STEEL 12X18H9T AFTER NEUTRON IRRADIATION IN THE PRESSURE VESSEL OF BR-10 FAST REACTOR AT VERY LOW DOSE RATES—S. I. Porollo, A. M. Dvoriashin, Yu. V. Konobeev, A. A. Ivanov, and S. V. Shulepin (Institute of Physics and Power Engineering) and F. A. Garner (Pacific Northwest National Laboratory)

OBJECTIVE

The objective of this effort is to explore the response of Russian austenitic steels to irradiation in various fast reactors of the former Soviet Union, looking for insights on how Western steels of similar composition might behave. Of particular interest is the magnitude of void swelling that might occur for irradiation proceeding at relatively low irradiation temperatures and dpa rates.

SUMMARY

The internal components of various Russian reactors such as VVER-440 and VVER-1000 pressurized water reactors and the BN-600 fast reactor are usually made of Russian designation 18Cr-9Ni or 18Cr-10Ni-Ti austenitic stainless steel. In Western PWRs and BWRs, the AISI Type 304 steel with composition similar to 18Cr-9Ni steel is used for this purpose. Currently, the issue of reactor life extension is very important for both Russian and Western reactors of the PWR type. It has also been recognized that some problems encountered in PWRs, especially those associated with changes in dimension and mechanical properties, can also be expected to occur in water-cooled fusion devices.

Results are presented for void swelling and microstructure and mechanical properties of Russian 12X18H9T (0.12C-18Cr-9Ni-Ti) austenitic stainless steel irradiated as a pressure vessel structural material of the BR-10 fast reactor at ~ 350ºC to only 0.64 dpa, produced by many years of exposure at the very low displacement rate of only 1.9×10^{-9} dpa/s. In agreement with a number of other recent studies, it appears that lower dpa rates have a pronounced effect on the microstructure and mechanical properties. In general, lower dpa rates lead to the onset of swelling at much lower doses compared to comparable irradiations conducted at higher dpa rates.

PROGRESS AND STATUS

Introduction

Near-core internals of Russian power reactors (VVER-440, VVER-1000, BN-600) are made of type X18H9 or X18H9T (18Cr-9Ni or 18Cr-10Ni-Ti) austenitic stainless steels. In Western PWRs and BWRs, the steel AISI 304 (with chemical composition similar to 18Cr-9Ni) is used for such purposes. In addition, Soviet-design fast reactors also use 18Cr-10Ni-Ti as a pressure vessel material, whereas Western-design reactors use low-alloy ferritic steels.

Currently, the issue of plant life extension is very important to many Russian and Western reactors of PWR and BWR type, especially since much of the internals are not easily removable. It has also been recognized that many of the material issues confronting the PWR and BWR reactors will also be faced by water-cooled fusion reactors such as ITER.

Confident validation of reactor life extension requires reliable information on how the properties of structural materials of internal components will change with increasing neutron dose, especially at damage levels not yet reached by the component. In practice, this question is usually solved for pressure vessels by using surveillance samples, which are located at the reactor core periphery and therefore irradiated at higher neutron fluxes than is the vessel. For internal components of PWRs and WWERS, however, higher fluence data are usually needed at lower dpa rates than are available in the reactor type of interest. When available, such data are usually generated in higher flux reactors at dpa rates that are much larger than that of water-moderated reactors. If the properties under consideration are flux-sensitive, then there is some problem in extrapolation to the component of interest.

Recently it became clear that data on surveillance samples are insufficient for Russian life extension efforts concerning austenitic pressure vessels. A similar insufficiency exists for in-core components. This problem is related to the impossibility to obtain data over a sufficiently wide range of dose rates to approximately the same dose that will allow quantification of a flux dependency. One approach to partially fill this need is to examine assemblies irradiated at the core periphery or other components located even farther from the core.

In the present paper are presented results of swelling and microstructure and mechanical properties investigations of Russian austenitic stainless steel 12X18H9T (0.12C-18Cr-9Ni-Ti) irradiated as the structural material of the BR-10 fast reactor vessel to a dose of only 0.64 dpa at the very low displacement rate of 1.9×10^{-9} dpa/s.

Experimental Procedure

Samples for investigation of microstructure and mechanical properties were cut from the first vessel of the BR-10 fast reactor, after the vessel was replaced by a new vessel in 1979. The first vessel was variable in width with a maximum outside diameter of 535 mm and a total length just over 4 m. At the location of fuel assemblies, the vessel has the outside diameter of 366 mm and wall thickness of 7 mm. The vessel material is 12X18H9T austenitic stainless steel in the solution treated condition. The nominal chemical composition of the steel is (wt. %): $C \leq 0.12$; $Si \leq 0.8$; $Mn \leq 2.0$; Cr at 17-20; Ni at 8-11; $Ti < 0.8$.

The first vessel was in operation for 20 years (July 1959 till October 1979) with three fuel cycle runs, the first two with PuO_2 fuel and the third with UC fuel. The total reactor operation during this period was 3930 days or 2562.6 effective full power days. The total neutron fluence accumulated by the vessel at the core midplane was 8.44×10^{26} n/m^2 corresponding to an exposure dose of 33.1 dpa (NRT). On the inside, the vessel was in contact with sodium coolant flowing from bottom to top, but on the outside, it was in contact with air contained in the gap between the vessel and a safety vessel. In the first and last cycles, the temperature of the vessel was 350ºC, but during the second cycle it was 430ºC.

To study the mechanical properties and microstructure, specimens were cut from the vessel at two elevations. Irradiation conditions for these elevations are shown in Table 1.

Table 1. Irradiation conditions for sections cut from the BR-10 reactor vessel

Place of specimen cutting	Distance from core midplane, mm	Total neutron fluence, 10^{26} n/m^2	Dose, dpa	Average irradiation temperature, °C	Dose rate, dpa/s
Level of basket bottom	-425	0.35	0.64	350	1.9×10^{-9}
Level of upper flange	+1890	…	…	80	...

One specimen was cut from the bottom level of the fuel basket, in which the lower ends of fuel assemblies were located. Another specimen was cut at the level of the upper flange of the first coolant circuit. This second specimen was effectively unirradiated but had been aged for 20 years at 80ºC.

Using a remote milling machine, strips with cross section 10 mm × 2 mm or 7 mm × 2 mm were cut from the original sections in an axial direction. Then from these strips, TEM specimens and flat specimens for measurements of short-term mechanical properties were prepared.

Mechanical properties were measured for flat samples having a gauge length of 12 mm and a cross section of 2 mm × 2 mm. The tests were carried out at temperatures of 25 and 350ºC. The test temperature of 350ºC equals the inlet coolant temperature in the core for the majority of reactor operation time and was approximately equal to the temperature of the reactor vessel at the basket bottom level. The initial strain rate employed was 1.4×10^{-3} s^{-1}. At each temperature, three or four tensile specimens were tested and the results averaged.

TEM specimens in the form of disks of 3 mm in diameter with a perforated central hole were prepared using a standard technique employing the two-jet-polishing "STRUERS" device. Microstructural investigations were performed at an accelerating voltage of 100 kV using a JEM-100CX electron microscope equipped with a lateral goniometer.

Results

The microstructure of the unirradiated steel at the level of upper flange is shown in Figs. 1 and 2. It is observed that the steel had the anticipated austenitic structure with a grain size of ~ 10–20 microns. Austenitic grains, in turn, are divided into subgrains by dislocation walls with sizes ranging from ~ 1 to 5 microns (Fig. 1). The average dislocation density is (4–5) × 10^{13} m^{-2}. In addition, twins, large TiC precipitates with mean diameter of 0.5 to 1 microns, and much smaller precipitates distributed uniformly and at much higher density within the grains (Fig. 2), were observed. The diameter of the small precipitates ranges from 50 to 60 nm, with their concentration at ~ 3×10^{19} m^{-3}. An analysis of micro-diffraction patterns obtained from these precipitates showed that these precipitates have the fcc-structure with the lattice parameter of 0.43 nm, identifying them as TiC carbides.

The microstructure of the irradiated steel from the cross section at the level of the basket bottom is shown in Figs. 3 and 4. Even at the low dose of 0.64 dpa, the microstructure has changed significantly, producing non-uniform spatial distribution of dislocation loops (Fig. 3) and voids (Fig. 4). Frank dislocation loops were found with a mean diameter of 33 nm and mean concentration of 3 × 10^{21} m^{-3} but which are arrayed in extended linear clusters, at concentrations higher compared with other regions (Fig. 3). The size of such arrays coincides with the size of sub-grains observed in the unirradiated steel, and thus, it can be assumed that the dislocation loops formed presumably on the dislocation walls separating the sub-grains.

The spatial distribution of voids is also rather non-uniform. Large voids are located mainly in zones having high loop concentration, i.e., in the former dislocation walls (Fig. 4). Smaller voids, however, are distributed nearly uniformly throughout the grain. The swelling of the steel equals 0.1%, with a mean void diameter of 11 nm and concentration of 6 × 10^{20} m^{-3}. Precipitates observed in the irradiated steel were essentially identical to those in the unirradiated steel.

The average values of ultimate strength, yield strength, and total and uniform elongation of specimens from the two cross sections of the BR-10 vessel are shown in Table 2.

From Table 2, it appears that irradiation of steel 12X18H9T at a temperature of 350ºC to 0.64 dpa has resulted in substantial strengthening and some ductility loss. The yield strength increased by 286 MPa at T_{test} = 25ºC, and by 223 MPa at T_{test} = 350ºC. The total elongation of the steel has decreased from 53.3% to 34.5% at T_{test} = 25ºC, and from 28.6% to 18.7% at T_{test} = 350ºC.

Table 2. Results of mechanical tests of flat samples from steel 12X18H9T

Cross section	Test temperature, ºC	Mechanical properties			
		Ultimate strength, MPa	Yield strength MPa	Total elongation %	Uniform elongation, %
Level of basket bottom	25	784	563	34.5	28.0
	350	585	445	18.7	12.8
Level of upper flange	25	553	277	53.3	47.7
	350	396	222	28.6	21.8

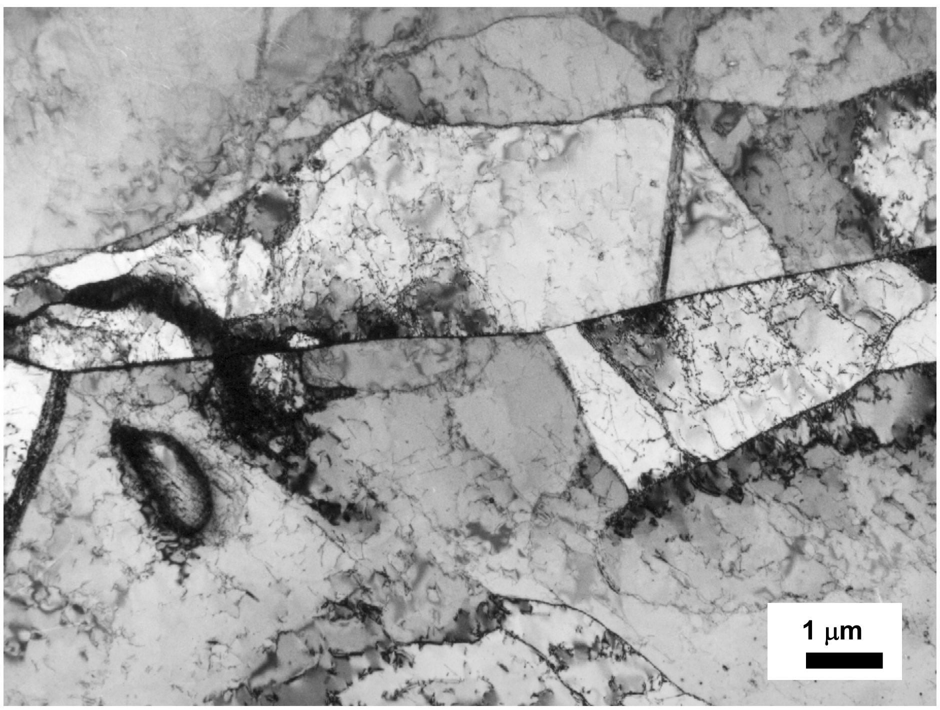


Fig. 1. Microstructure of the unirradiated steel 12X18H9T from the template cut out from the upper flange of the BR-10 reactor first vessel.

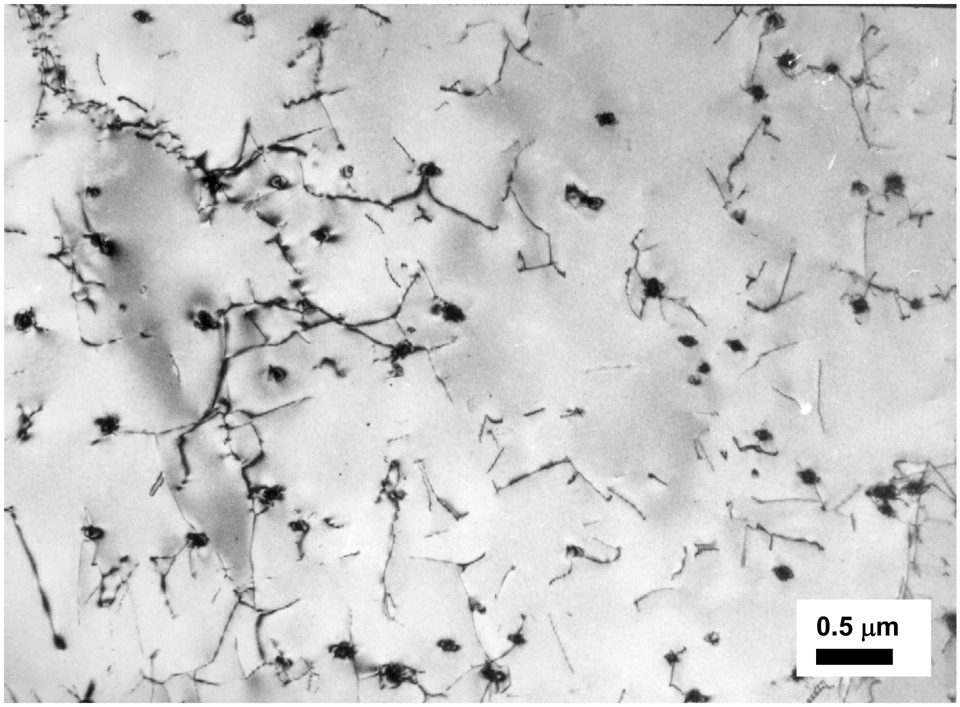


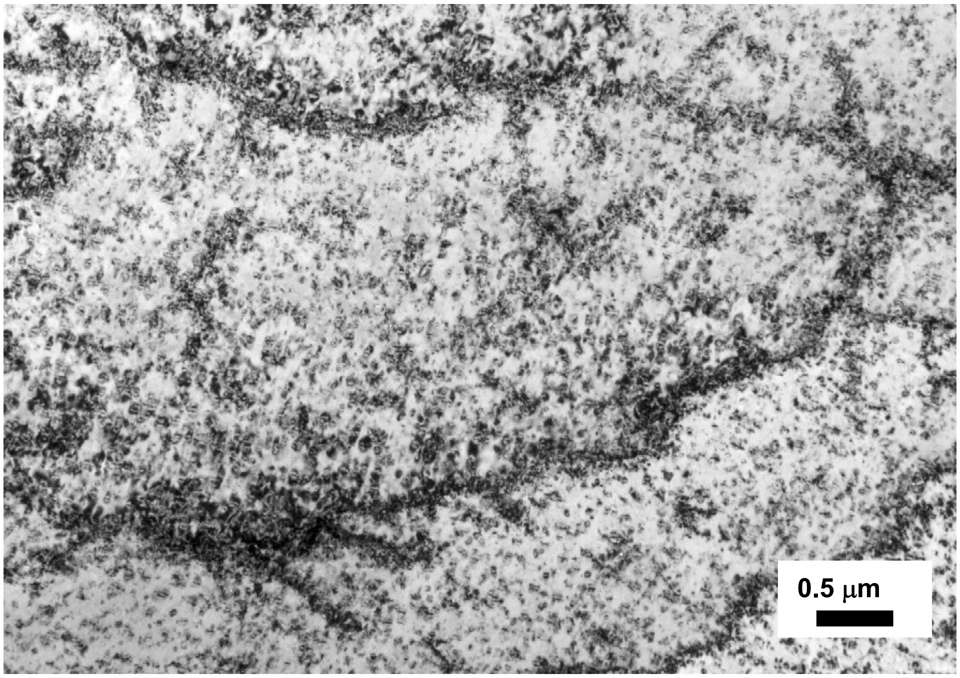
Fig. 2. Dislocations and TiC-precipitates unirradiated steel 12X18H9T (cross section of the BR-10 reactor vessel at the level of the upper flange).

Discussion

The cross section of the BR-10 vessel at the basket bottom level is quite remote from the reactor core. The dose of 0.64 dpa in this cross section has been accumulated in the vessel steel for 2563 effective full power days of reactor operation. Hence, the maximum dose rate in this cross section was equal to 0.64 dpa/2.2 $\times$ 10^8s = 2.9 $\times$ 10^{-9} dpa/s with an average dose rate of 1.9 $\times$ 10^{-9} dpa/s. In comparison, the dose rate at the center of the BR-10 core was 3.5 $\times$ 10^{-7} dpa/s. In the BN-600 fast reactor core this rate is even higher at 1.8 $\times$ 10^{-6} dpa/s [1]. The internals of Russian power reactors (VVER-440, VVER-1000) operate at considerably lower dose rates. Dose rates and doses accumulated in some internals during 30 years of operation are shown in Table 3.

As seen from Table 3, the dose rates in various internals of VVER-1000 and BN-600 are much higher than that of this steel in this current study but vary at least by one order of magnitude. Nevertheless, even at low dose rates some structural components of these reactors can accumulate rather high doses. If there exists a sensitivity to dose rate for either swelling or hardening, then there is considerable uncertainty associated with extrapolation of data from one dose rate situation to a different dose rate situation.

One can compare the swelling observed for the pressure vessel with that of wrappers and pin cladding of BR-10 fuel assemblies made from the same steel and irradiated at much higher dpa rates. The data base on swelling of the steel was obtained from examination of wrappers and fuel pins of the BR-10 reactor where the inlet sodium temperature was equal to 430ºC. For this comparison, only swelling data derived from bottom of the wrappers and claddings were selected in order to keep the temperature very close to 430ºC so there is very little uncertainty in the temperature. These in-core data are a subset of a larger data base later shown in Fig. 6 and were published in an earlier report [13].



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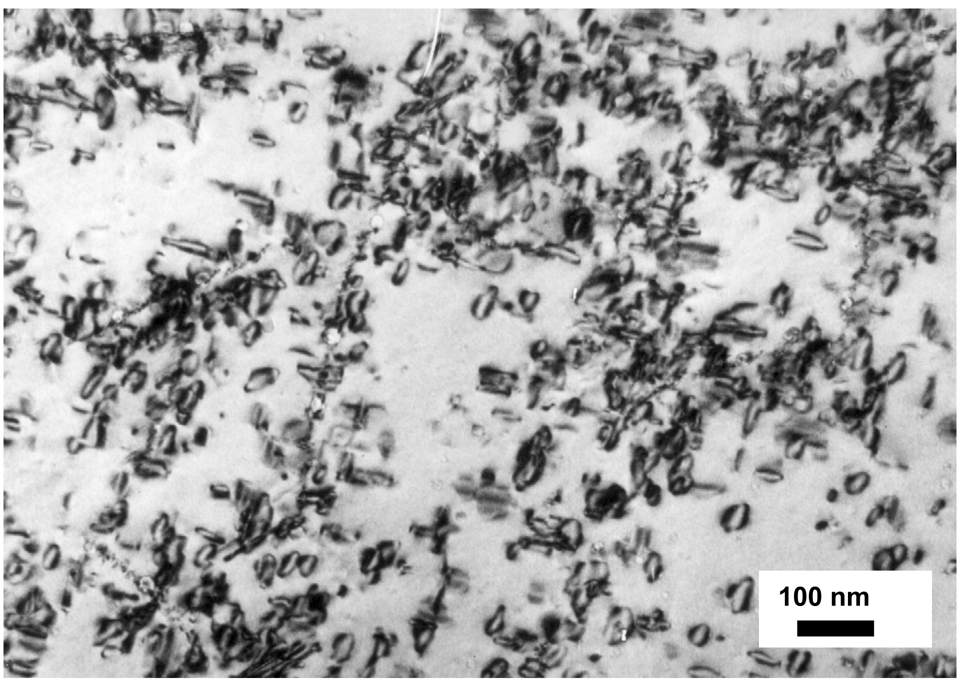

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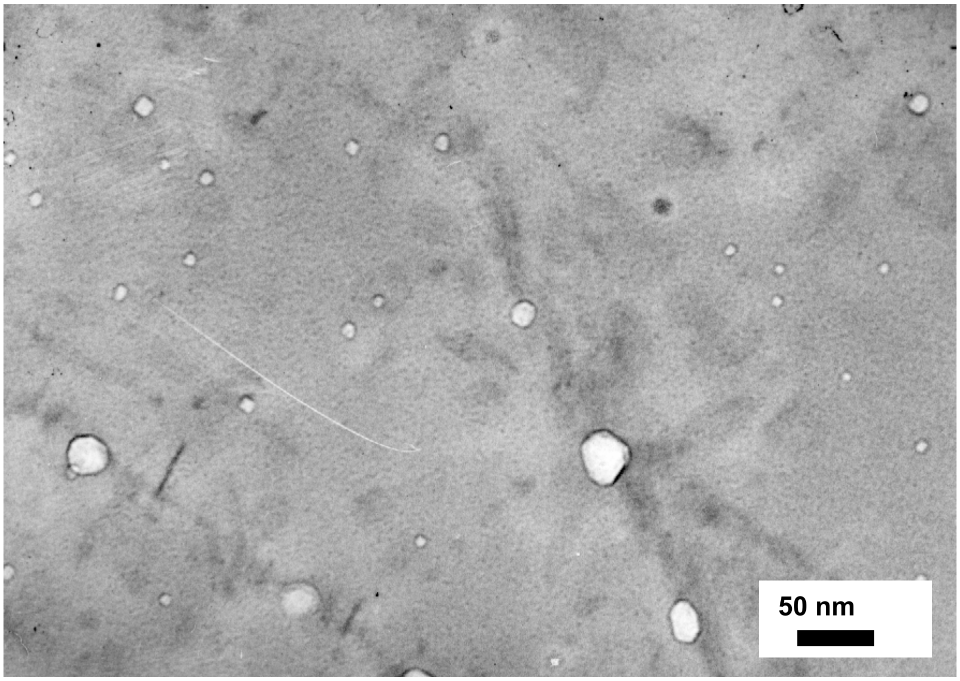
Fig. 3. Dislocation loops in neutron irradiated 12X18H9T steel (cross section of theBR-10 reactor vessel at a level of the basket bottom): a) general view and b) dislocation loop arrays along previously existing sub-grain boundaries.



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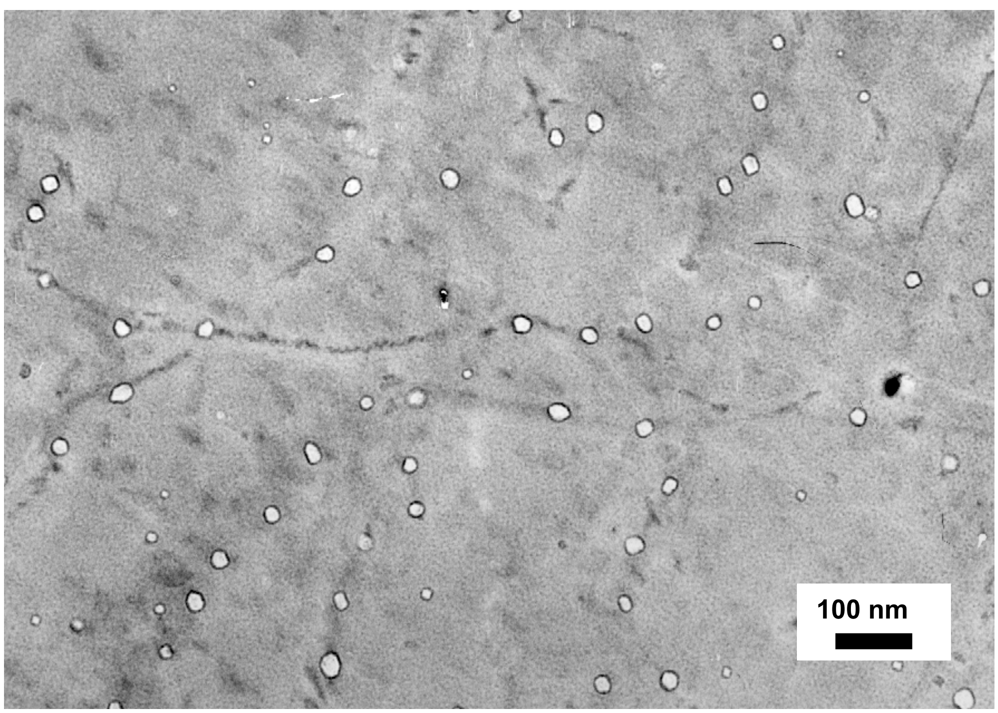

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Fig. 4. Voids in neutron irradiated 12X18H9T steel: a) large voids on pre-existing sub-grain boundaries and b) spatial distribution of smaller voids.

Table 3. Doses accumulated in various internals during 30 years of operation and dose rates

Structural component	Dose, dpa	Dose rate dpa/s	Reference
BN-600 fast reactor			
Subassembly sheath	35	4.5×10^{-8}	[2]
Guide tubes of control rods	15–18	$(1.9–2.3) \times 10^{-8}$	[2]
Collector bottom grid plate	2	0.6×10^{-8}	[2]
Reactor vessel	< 1	$< 0.13 \times 10^{-8}$	[2]
VVER-1000			
Baffle assembly	2–50	$(0.3–7.4) \times 10^{-8}$	[3]

The data for cladding and wrappers are shown in Fig. 5, together with the data for the vessel as a function of dose. It is seen from Fig. 6 that after the incubation dose of 4–7 dpa, the swelling of the wrapper and cladding at ~ 430ºC is an approximately linear function of dose with the swelling rate of 0.08 to 0.13 %/dpa. In general, one would expect that the vessel specimen, which spent two-thirds of its life at 350ºC, would swell less at lower temperatures, but the swelling of the vessel steel measured by microscopy is higher (~ 0.1% at only 0.64 dpa).

Since irradiation conditions for the vessel and reference fuel assemblies of BR-10 reactor differ primarily in dose rate but secondarily in temperature history, one can conclude with caution that a decrease of dose rate results in a reduction of transient period of swelling. A similar conclusion was reached following examination of AISI Type 316 fuel pin cladding after irradiation in the RAPSODIE and PHENIX fast reactors where the duration of incubation period decreases with decreasing the displacement rate [5]. The most significant observation is that voids can form at such a low dpa level, regardless of the temperature.

This rather surprising result is very consistent with a growing body of evidence that shows that a decrease in dose rate leads not only to an earlier onset of swelling with dose but also swelling that extends to lower-than-expected temperatures. Based on some earlier studies [4–5], Garner and coworkers predicted that austenitic steels serving as internal components in PWRs would exhibit unanticipated levels of void swelling [6–7]. Even more importantly, it was concluded that high dose data derived from in-core regions of high flux fast reactors would strongly under-predict the swelling that would arise at lower dpa rates characteristic of PWRs, BWRs, out-of-core regions of fast reactors, and many components of proposed fusion devices such as ITER.

A number of recent studies by Garner and coworkers have shown that void swelling in austenitic stainless steels strongly increases at lower dpa rates [8–16], often allowing the observation of the lower swelling temperature limit (~ 280ºC) at very low dpa levels. This increase in swelling arises primarily from a decrease in the duration the transient regime of swelling at lower dpa rates. As the dpa rate goes below ~ 10^{-8} dpa/sec the transient regime of swelling approaches zero dpa.

As noted earlier, a peculiarity of the irradiated microstructure is an inhomogeneous spatial distribution of dislocation loops and voids. The largest voids formed in regions of higher density of initial dislocations (i.e., in dislocation walls). The dislocation loop concentration is also higher in these regions. Surprisingly, within the limits of measurement accuracy, the mean loop diameter does not depend on the location of loops.

Addressing the change of mechanical properties, as a result of irradiation to a rather low dose of 0.64 dpa, the strength of the vessel steel increased significantly. The yield strength measured at 25ºC has increased from 277 MPa (unirradiated steel) to 563 MPa, i.e., increased by 103%. The steel ductility has also changed; the total elongation has decreased from 53.3% to 34.5%, i.e., by 18.8%.

Irradiation hardening and ductility loss at low temperatures of austenitic stainless steels in the solution treated condition has been observed many times [17–19]. The change of yield strength with dose initially occurs very quickly. At doses in the 0.5–1.0 dpa range, the yield strength of a material increases strongly, and usually reaches a saturation level at < 10 dpa. Therefore the observed strength change of the BR-10 vessel steel is similar to the behavior of other austenitic stainless steels.

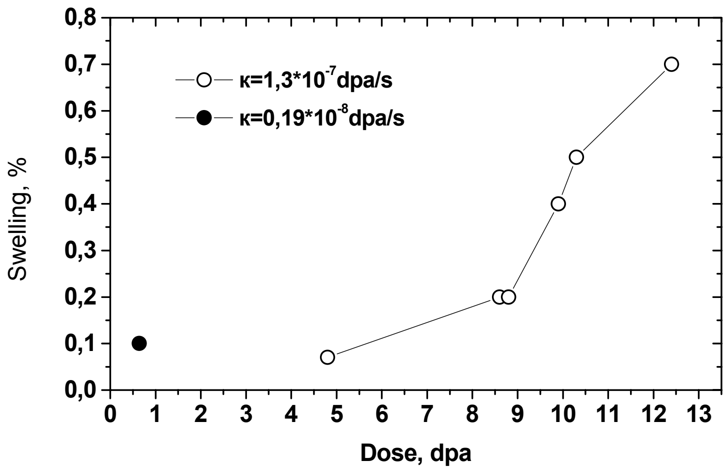

Fig. 5. Dependence of steel 12X18H9T swelling on dose. Light circles - wrappers of fuel assemblies and fuel pin claddings of BR-10 reactor at 430ºC, black circle - vessel specimen with 350-430-350ºC history. The displacement rates are shown in the figure for both sets of specimens.

It is of interest to compare the change of the yield strength of 12X18H9T steel with microstructural changes occurred under irradiation. Pre-existing straight dislocations have disappeared completely under irradiation with dislocation loops and voids taking their place without any changes in the precipitate structure, which is the same in both cases.

In this case, by assuming an additive superposition of different dislocation barriers, the change of yield strength of the steel can be written as follows:

$$\Delta\sigma = MGb[\alpha_v(d_vN_v)^{1/2} + \alpha_l(d_lN_l)^{1/2} - \alpha_d\rho_d^{1/2}] \qquad (1)$$

where the Taylor factor M = 3.06; G=78.7; GPa (at 25ºC) is the shear modulus; b = 0.25; nm is the Burgers vector; α_v, α_l, and α_d are the barrier constants for voids, dislocation loops, and dislocations, respectively; d_v and d_l are the mean diameters of voids and dislocation loops; N_v and N_l are the concentrations of voids and dislocation loops, respectively; ρ_d is the density of straight dislocations.

Using the microstructural data for d_v, dl, N_v, N_l, and ρ_d to obtain the value of $\Delta\sigma$ from Eq. (1), one concludes that when the values of barrier constants known from the literature (α_v = 1.0, α_l = 0.33, and α_d = 0.20) are assumed, the calculated increase of yield strength is 267 MPa compared to the measured value of 286 MPa. If there is any significance to this relatively small difference, the discrepancy may arise from the non-uniform spatial distribution of voids and loops in the irradiated steel, but such conjecture is entirely speculative.

Finally, it should be noted that most previous perceptions concerning the lower boundary of void swelling and the flux-dependence of the lower temperature limit of swelling were established using reactors with relatively high inlet temperatures, such as 350ºC in BR-10 and 365–370ºC in FFTF and EBR-II. As shown in Fig. 6, when swelling data on the same steel are compiled from reactors with different inlet temperatures and from data derived from both fueled and unfueled zones, then the apparent lower limit of swelling moves toward the lowest inlet temperature. Thus the previously published BR-10 in-core data imply that swelling ceases between 400 to 430ºC, but swelling actually develops down to significantly lower temperatures, as seen in both the vessel specimen and in specimens taken from the reflector region of BN-350 with its lower inlet temperature of 280ºC.

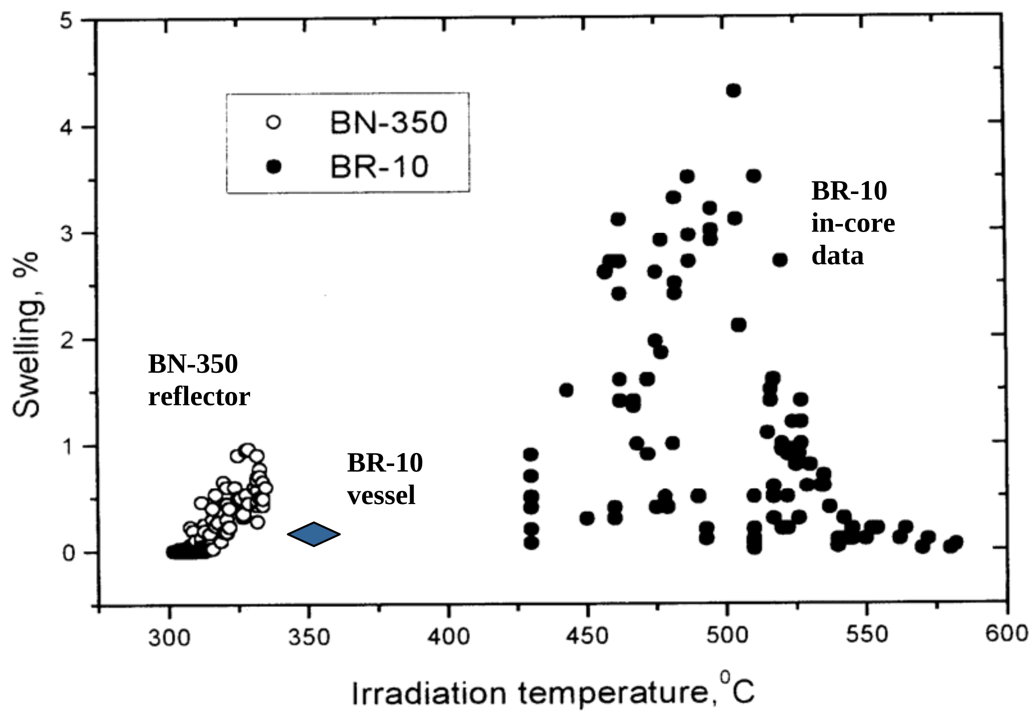

Fig. 6. Comparison of swelling data on annealed austenitic steel 18Cr-10Ni-Ti derived from three separate sources in two fast reactors [13]. The BR-10 data shown at 430ºC was presented earlier in Fig. 5. The added datum of the BR-10 vessel is shown at 350ºC only for convenience.

Conclusions

Examination of the microstructure, swelling, and short-term mechanical properties of the BR-10 reactor first vessel steel (12X18H9T) after irradiation to 0.64 dpa at a very low displacement rate of 1.9 × 10-9 dpa/s leads to the following conclusions:

1. Neutron irradiation under such conditions results in a significant reduction of the swelling incubation dose to < 1 dpa as compared with incubation dose of 4–7 dpa in cladding and wrapper materials of BR-10 reactor at a dose rate of ~ 1.3×10^{-7} dpa/s.

2. The spatial distribution of both dislocation loops and voids in the irradiated steel is non-uniform and appears to be caused by the initial non-uniformity of dislocation structure.

Irradiation resulted in hardening accompanied with a ductility loss.

Acknowledgements

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