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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

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Abstract

Results are presented for void swelling, 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 ~350 °C 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 resultant 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.

1. 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 pressurized (PWRS) and boiling (BWRS) water power reactors the steel AISI 304 (with chemical composition similar to 18Cr–9Ni) is used for such purposes. In addition Soviet-design fast reactors such as BR-10, BOR-60, BN-350 and BN-600 also use 18Cr–10Ni–Ti as a pressure vessel material, whereas Western-design reactors usually 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 in Russian VVERs since much of the austenitic internals are not easily removable [1,2]. VVER is the Russian acronym for water-cooled, water-moderated energetic reactor. Plant life extension is also an issue for Russian fast reactors such as BN-600 which are approaching their design lifetime. In addition many of the material issues confronting austenitic steels in 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

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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 VVERS, 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, 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.

2. Experimental details

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 $\leqslant$ 0.12; Si $\leqslant$ 0.8; Mn $\leqslant$ 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 types of fuel cycle runs, the first and third with PuO_2 fuel and the second 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 inlet temperature of the vessel was 350 °C, but during the second cycle it was 430 °C.

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.

One specimen was cut from the bottom level of the fuel basket, in which the lower ends of 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 ~80 °C.

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 TEM specimens and flat specimens for measurements of short-term mechanical properties were prepared from these strips.

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

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	–

at temperatures of 25 and 350 °C. The test temperature of 350 °C 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 $1.4 \times 10^{-3}\ s^{-1}$. 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.

3. 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 μm. Austenitic grains, in turn, were divided into sub-grains by dislocation walls with sizes ranging from ~1 to 5 μm (Fig. 1). The average dislocation density is $(4–5) \times 10^{13}\ m^{-2}$. In addition, twins, large TiC precipitates with mean diameter of 0.5–1 μm, 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 $\sim 3 \times 10^{19}\ m^{-3}$. 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 distributions of both dislocation loops (Fig. 3) and voids (Fig. 4). Frank dislocation loops were found with a mean diameter of 33 nm and mean concentration of $3 \times 10^{21}\ m^{-3}$ but which are arrayed in extended line arrays at higher concentrations 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 concentrations, i.e. in the former dislocation walls (Fig. 4). Smaller voids, however, are distributed nearly uniformly throughout the

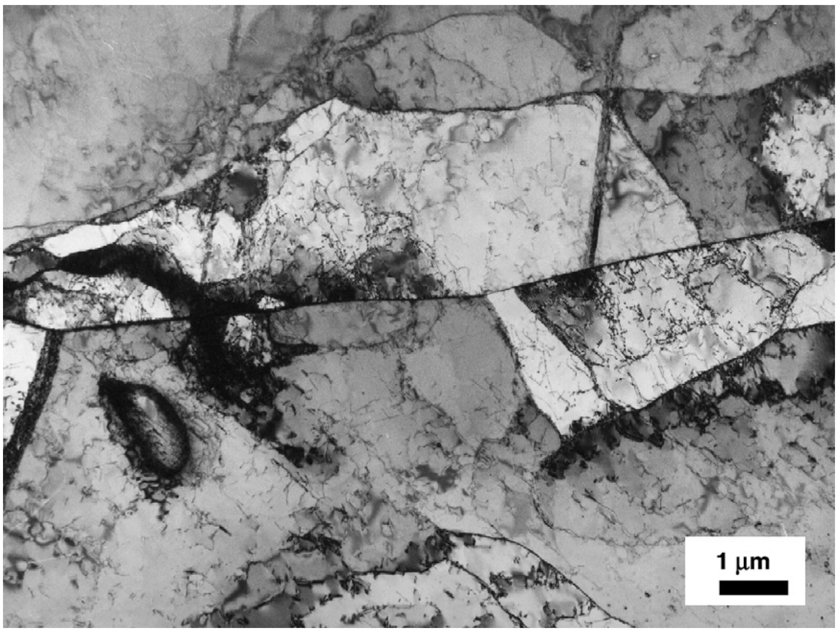

Fig. 1. Microstructure of the unirradiated steel 12X18H9T from the template cut out from the upper flange of the BR-10 reactor first vessel.

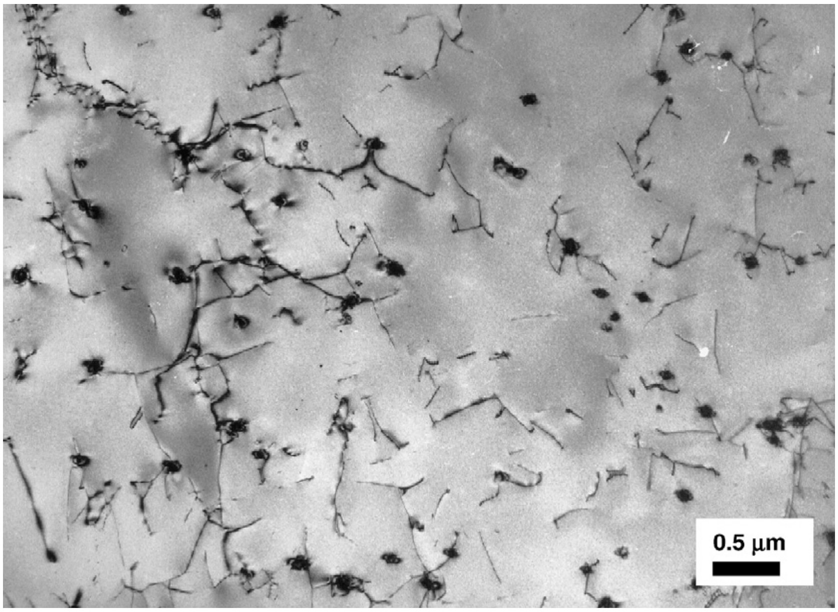

Fig. 2. Dislocations and TiC precipitates in unirradiated steel 12X18H9T (cross section of the BR-10 reactor vessel at the level of the upper flange).

grain. The swelling of the steel equals ∼0.1%, associated with a mean void diameter of 11 nm and concentration of $6 \times 10^{20}\ m^{-3}$. Precipitates observed in the irradiated steel are essentially identical to those in the unirradiated steel.

The average values of ultimate strength, yield strength, 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 350 °C to 0.64 dpa has resulted in substantial strengthening and some ductility loss. The yield strength increased by 286 MPa at $T_{test} = 25$ °C, and by 223 MPa at $T_{test} = 350$ °C. The total elongation of the steel has decreased from 53.3% to 34.5% at $T_{test} = 25$ °C, and from 28.6% to 18.7% at $T_{test} = 350$ °C.

4. Discussion

The cross section taken from the BR-10 vessel at the basket bottom level was quite remote from the reactor core. The dose of 0.64 dpa in this cross section was 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 $0.64\ dpa/2.2 \times 10^{8}\ s = 2.9 \times 10^{-9}$ dpa/s with an average dose rate of 1.9×10^{-9} dpa/s. In comparison, the dose rate at the center of the BR-10 core was 3.5×10^{-7} dpa/s.

In the BN-600 fast reactor core this rate is higher at 1.8×10^{-6} dpa/s [3]. The internals of Russian power reactors (VVER-440, VVER-1000) operate at considerably lower dose rates [2,4]. 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 in the 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 BR-10 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 430 °C. For this comparison only swelling data derived from bottom of the wrappers and claddings were selected in order to

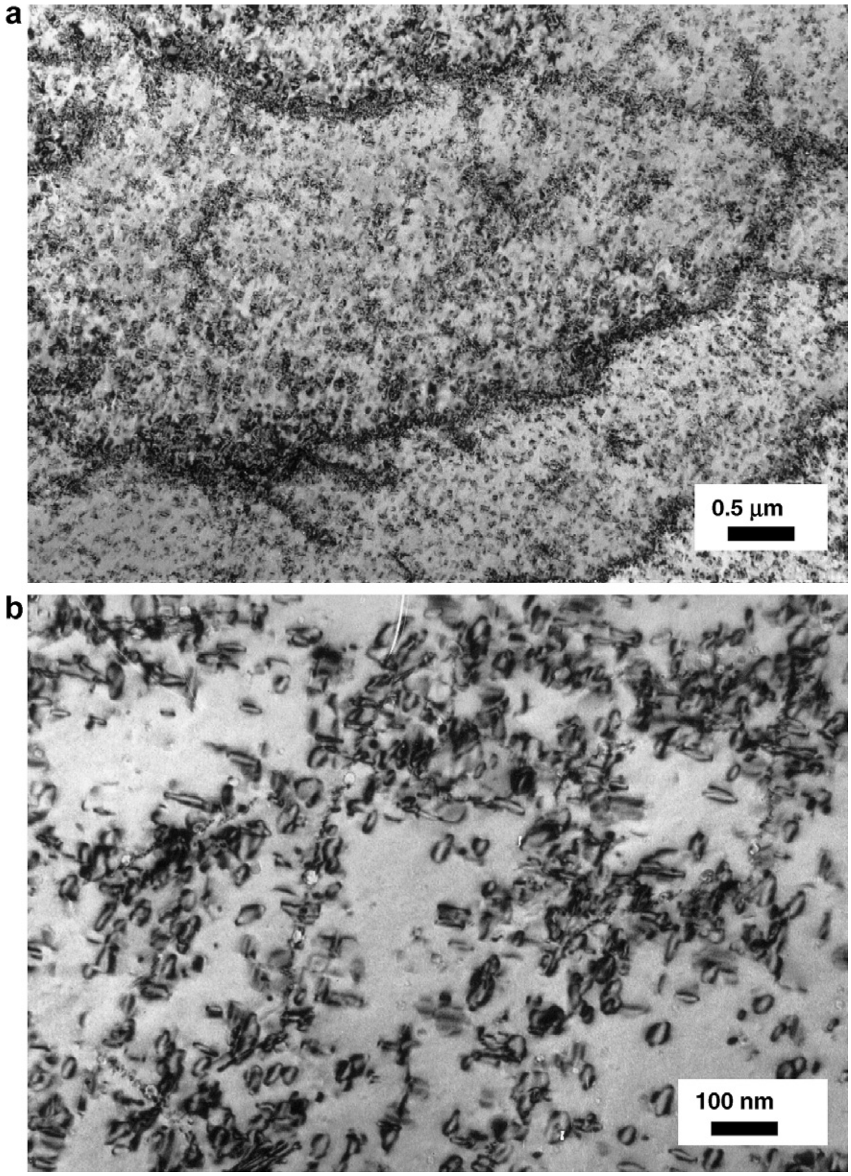

Fig. 3. Dislocation loops in neutron irradiated 12X18H9T steel (cross section of the BR-10 reactor vessel at a level of the basket bottom): (a) general view, (b) dislocation loop arrays along previously existing sub-grain boundaries.

keep the temperature very close to 430 °C so there is very little uncertainty in the temperature. These in-core data are a subset of a larger data base shown in Fig. 6 and were published in an earlier report [5].

The data for cladding and wrappers at 430 °C are shown in Fig. 5 together with the data for the vessel as a function of dose. It is seen from Fig. 5 that in the range of 7–12 dpa the swelling of the wrapper and cladding at ~430 °C is an approximately linear function of dose with a swelling rate of ~0.1%/dpa, an order of magnitude below the well-known 1%/dpa terminal swelling rate of stainless steels [6,7]. Presumably the swelling is still in the transient regime.

If we ignore the effect of displacement rate on swelling, one would expect that the vessel specimen, which spent two-thirds of its life at 350 °C, would swell much less at 0.64 dpa compared to isothermal swelling of wrappers and cladding at 430 °C, but the swelling of the vessel steel (as measured by microscopy) is higher than expected, reaching ~0.1%. It appears that there has been a significant

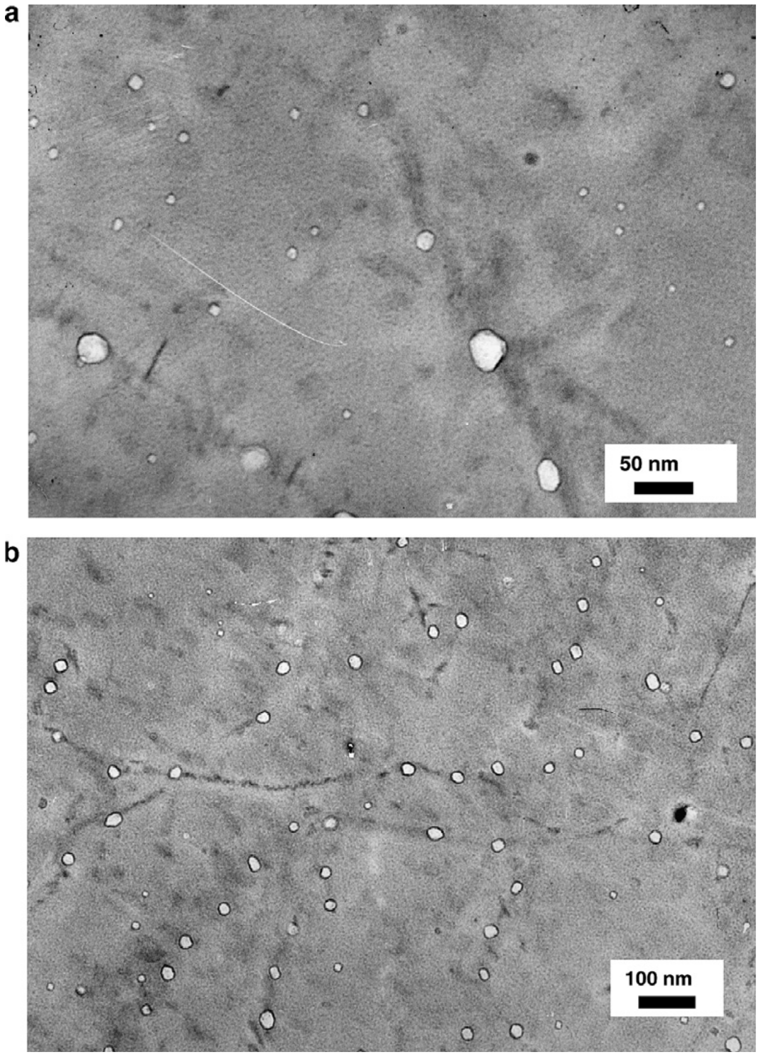

Fig. 4. Voids in neutron irradiated 12X18H9T steel: (a) large voids on pre-existing sub-grain boundaries, (b) spatial distribution of smaller voids.

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

Cross section	Test temperature, °C	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

reduction in the duration of the transient regime as a consequence of the lower dpa rate.

This conclusion is consistent with the results of a study on AISI 304 stainless steel irradiated in the EBR-II fast reactor [8]. 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

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

Structural component	Dose, dpa	Dose rate dpa/s	References
BN-600 fast reactor			
Subassembly sheath	35	4.5×10^{-8}	[4]
Guide tubes of control rods	15–18	$(1.9–2.3) \times 10^{-8}$	[4]
Collector bottom grid plate	2	0.6×10^{-8}	[4]
Reactor vessel	<1	$<0.13 \times 10^{-8}$	[4]
VVER-1000			
Baffle assembly	2–50	$(0.3–7.4) \times 10^{-8}$	[2]

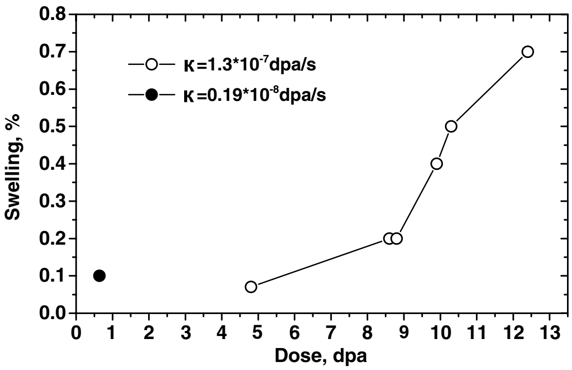

Fig. 5. Dependence of steel 12X18H9T swelling on dose. Light circles – wrappers of fuel assemblies and fuel pin claddings of BR-10 reactor at 430 °C, black circle – vessel specimen with 350–430–350 °C history. The displacement rates are shown in the figure for both sets of specimens. All microscopy measurements are considered to be accurate to ±20%.

of the incubation period was also observed to decrease with decreasing displacement rate [9].

Since irradiation conditions for the vessel and reference fuel assemblies of the 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 in the transient period of swelling. The most significant observation is that voids can form at such a low dpa level, regardless of the irradiation 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 much lower-than-expected temperatures. Based on some earlier studies Garner and coworkers predicted that austenitic steels serving as internal components in PWRs would exhibit unanticipated levels of void swelling [10,11]. 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 the 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 various austenitic stainless steels strongly increases at lower dpa rates [12–20], often allowing the observation of the lower swelling temperature limit (~280 °C) 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 $\sim 10^{-8}$ dpa/s the transient regime of swelling approaches zero dpa.

As noted earlier, the irradiated microstructure displays an especially 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.

It is well known that in stainless steels, especially, but also in other metals and alloys, the initial stages of void nucleation often display heterogeneity of void nucleation that is dependent on pre-existing dislocation, loop or precipitate populations [21,22] so the association of Frank loops and voids with pre-existing dislocation walls is not unexpected.

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 25 °C 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%. There are also significant reductions in uniform elongation as shown in Table 2, falling from 47.7% to 28% at room temperature.

Irradiation hardening and ductility loss at low temperatures of austenitic stainless steels in the solution treated condition has been observed many times [23,24]. The change of yield strength with dose initially occurs very quickly. At doses in the

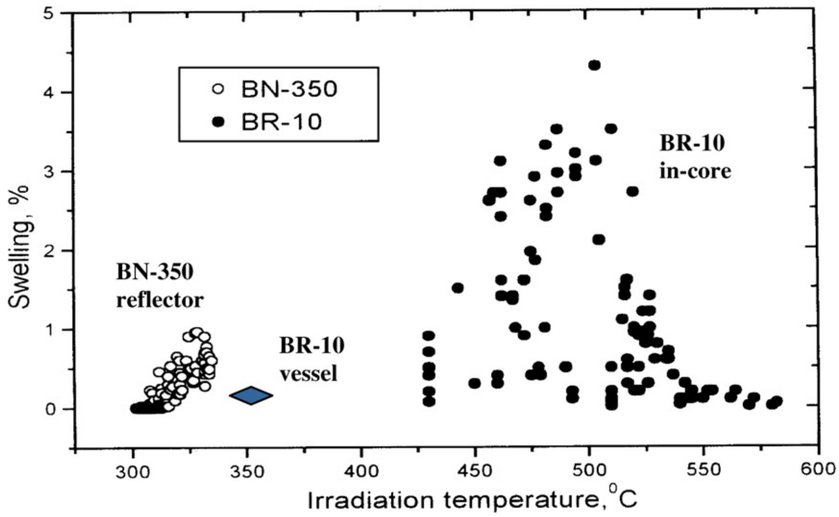

Fig. 6. Comparison of swelling data on annealed austenitic steel 18Cr–10Ni–Ti derived from three separate sources derived from two fast reactors [17]. The BR-10 data spans doses from 1 to ~25 dpa while the BN-350 data span doses of 4–55 dpa. The BR-10 data shown at 430 °C was presented earlier in Fig. 5. The added datum of the BR-10 vessel at 0.64 dpa is shown only for convenience.

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.

It is of interest to compare the change of the yield strength of 12X18H9T steel with microstructural changes that occurred under irradiation. Pre-existing straight dislocations have disappeared completely under irradiation with dislocation loops and voids appearing without any changes in the precipitate structure.

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:

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

where the Taylor factor $M = 3.06$, $G = 78.7$ GPa (at 25 °C) 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 pre-existing density of straight dislocations [25].

Using the microstructural data for d_v, d_l, 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 ($\alpha_v = 1.0$, $\alpha_l = 0.33$ and $\alpha_d = 0.20$) 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 350 °C in BR-10 and 365–370 °C 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 and 430 °C, 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 280 °C.

5. 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 transient duration as compared with that of cladding and wrapper materials at a dose rate of $\sim 1.3 \times 10^{-7}$ dpa/s.
2. The spatial distribution of both dislocation loops and voids in the irradiated steel is non-uniform and appears to be associated with the initial non-uniformity of dislocation structure.
3. Irradiation-induced loops and voids resulted in significant hardening accompanied with losses in both uniform and total elongation. Changes in precipitation were negligible and did not contribute to radiation-induced hardening.

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