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Characterization of 08Cr16Ni11Mo3 stainless steel irradiated in the BN-350 reactor

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Abstract

In several recently published studies conducted on a Soviet analog of AISI 321 stainless steel irradiated in either fast reactors or light water reactors, it was shown that the void swelling phenomenon extended to temperatures as low as ~300° C, when produced by neutron irradiation at dpa rates in the range 10^{-7} to 10^{-8} dpa/s. Other studies yielded similar results for AISI 316. In the current study a blanket duct assembly from BN-350, constructed from the Soviet analog of AISI 316, also exhibits swelling at dpa rates on the order of 10^{-8} dpa/s, with voids seen as low as 281 °C and only 1.3 dpa. It appears that low-temperature swelling at low dpa rates occurs in 300 series stainless steels in general, and during irradiations conducted in either fast or mixed spectrum reactors.

1. Introduction

In a recently published study it was shown that in annealed 12X18H10T, the Soviet analog of AISI 321 stainless steel, void swelling inhabits a temperature regime with a lower limit at ~300 °C, when irradiated in the BN-350 fast reactor at dpa rates on the order of 10^{-7} to 10^{-8} dpa/s [1]. This study was conducted on an unfueled flow restrictor element removed from the breeder zone of the reactor. Limited comparison of swelling in the same steel following irradiation at comparable dpa rates in several light water reactors confirmed that void nucleation in general is limited to temperatures >300 °C [2,3]. Similar results were recently observed in 316 stainless steel irradiated in Japanese and European PWRs [4,5].

Previous studies conducted in Western countries could not establish in fast reactors the lower temperature limit of swelling because the inlet coolant temperatures of all second-generation fast reactors in the West are in the range 365–380 °C. First-generation Western reactors such as EBR-I and DFR operated with lower inlet temperatures, but these reactors were decommissioned many years ago. In countries of the Former Soviet Union, however, there exist both first and second-generation reactors. One of these, the BN-350 fast reactor in Kazakhstan, was recently decommissioned. It had an inlet coolant temperature of 280 °C.

A number of recent studies by Garner and coworkers [6–9], have shown that void swelling in austenitic stainless steels actually increases at lower dpa rates, allowing the observation of the lower swelling temperature limit at lower dpa levels. This increase in swelling arises from a decrease in the duration of the transient regime of swelling at lower dpa rates. Both the flow restrictor component and the components from VVERs and PWRs experienced dpa rates that were much lower than those found inside the fueled regions of fast reactor cores.

Another opportunity has recently arisen to provide further confirmation of the lower temperature limit of

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void nucleation in austenitic stainless steels by examining another component from the BN-350 reactor that was irradiated at low neutron flux.

2. Experimental details

A hexagonal blanket assembly designated H-214(II) was irradiated in the reflector region of the BN-350 reactor, reaching a maximum of 15.6 dpa at an average dpa rate of 4.9×10^{-8} dpa/s averaged over its lifetime in reactor. During operation time the assembly was moved five times to new positions in the blanket section of the reactor. The hexagonal duct with faces 50 mm wide and 2 mm thick was formed from 08Cr16Ni11Mo3 stainless steel, a Soviet analog of AISI 316 steel, and was produced with the final thermal-mechanical treatment of the duct being 20% cold deformation followed by annealing at 800 °C for 1 h. The actual composition was measured as (0.08–0.12C)–67.9Fe–17.5Cr–11.1Ni–1.5Mn–1.65Mo–0.7Si, in wt%.

The temperature at the bottom of the assembly was 280 °C and the temperature at the top of the assembly was 420 °C. Due to the thinness of the duct wall, the internal temperature was not raised significantly by gamma heating. Thus, the temperature of the steel is expected to be within 1–2 °C of the local coolant temperature.

At the BN-350 site specimens with 10 mm height and 50 mm width were cut from the duct walls at various locations. Subsequent reduction of these specimens was conducted in a hot cell at INP-Almaty for microstructural analysis and microhardness measurements. Plate-shape specimens with sizes of 5×6 mm were prepared for metallography investigations, microhardness measurements and hydrostatic weighing. To display grain boundaries these specimens were subjected to additional mechanical grinding and polishing. Examination techniques employed included optical metallography using a MeF-2 optical microscope and transmission electron microscopy (TEM), using a JEM-100CX electron microscope operating at 100 keV. Microhardness measurements were carried out by the Vickers method on a PMT-3 device provided with a diamond pyramid with an angle of 136°. The density was measured using a hydrostatic weighing technique employing a CEPN-770 electronic balance with methyl alcohol as the working liquid.

Disks of 3 mm diameter for microscopy studies were prepared from $\leqslant 300$ μm sections cut from the mid-section of the duct face. Mechanical grinding and polishing with subsequent electrochemical polishing were used for final preparation of TEM disks. The irradiation conditions for specimens examined to date are shown in Table 1.

Table 1

Fluence and temperature changes over the length of the H-214(II) duct

Distance from midplane (mm)	Dose (dpa)	dpa rate (10^{-8} dpa/s)	Temperature (°C)
−1200	0.25	0.08	280
−900	1.27	0.39	281
−500	7.08	2.2	309
0	15.6	4.85	337
+500	6.03	1.87	365

3. Results and discussion

3.1. Metallography investigations

The etching-produced microstructure of the outer surface of the duct face revealed several peculiarities. First, in all specimens the grain sizes were non-uniform to a great extent. Fine grains often were grouped in locations between large-scale grains. Within grains and at their boundaries were observed chains of $Cr_{23}C_6$ carbide inclusions, the quantity and size of which depend on the distance from the midplane of the core. The analysis of grain size distributions showed an increase in the fraction of large grains (25–50 μm) with increasing core elevation, and a concurrent decrease in fraction of fine grains (<12 μm). Details are provided in [10].

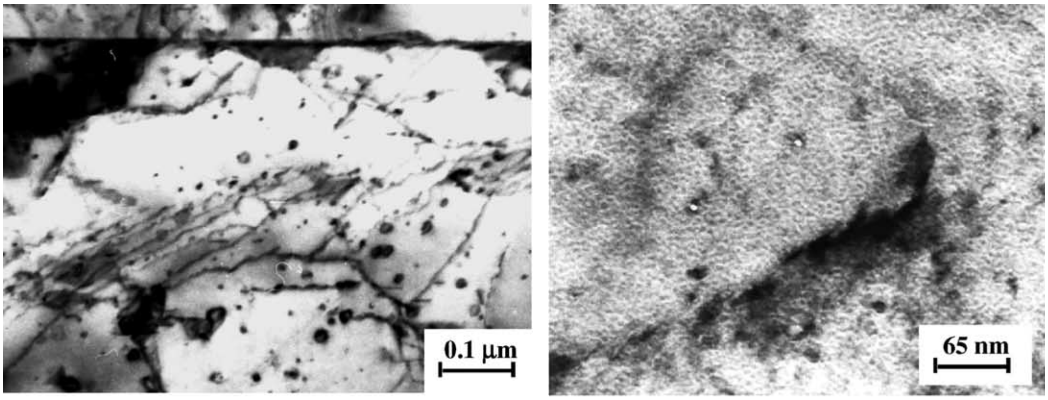
3.2. Electron microscopy

Dislocation loops were observed in the lowest exposure material (0.25 dpa) located a distance of −1200 mm from the midplane, with concentration of 3×10^{15} cm^{-3}, and sizes $\leqslant 25$ nm (mean loop size was 10.7 nm). At the −900 mm level where the dpa and dpa rates were four times higher, $\sim2\times10^{15}$ cm^{-3} was observed, with sizes 5–40 nm and mean of 15.5 nm. Since the temperature of the two positions was essentially identical this represents an effect of increasing dose to grow the loop diameter. Thorough investigation of the loops in dark-field mode identified these as Frank loops of interstitial type.

At other higher levels on the duct with higher temperatures the dislocation loop structure and pattern of strains near defects become more intricate with simultaneous increases in both concentration and size distribution of defects.

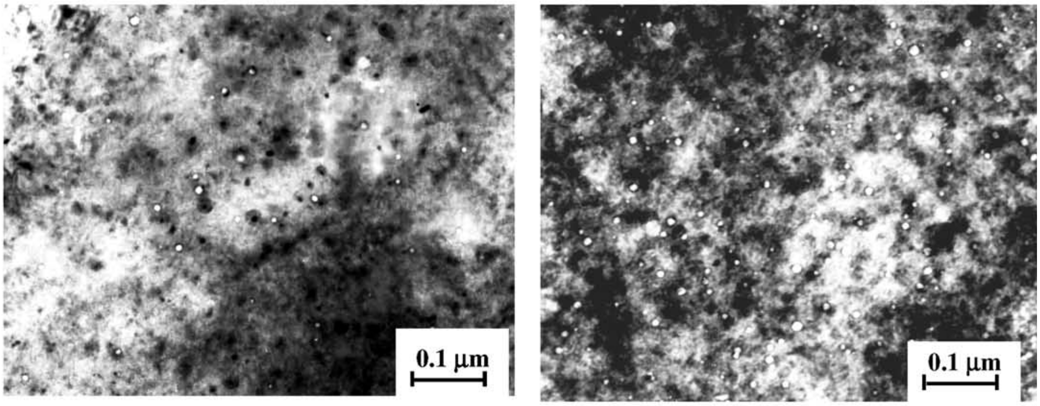
Fig. 1 shows microstructures of specimens when imaged under conditions that allow voids to be visible. Microscopy showed the presence of voids at −900 mm from midplane; where the irradiation temperature was only 280 °C, significantly lower than the ~300 °C limit reported earlier. The concentration of voids observed at this position is very small but allows us to confirm their presence. From TEM-image analysis microstructural data on voids were obtained (see Table 2).

It should be noted that calculated values of swelling are relatively small, in agreement with earlier published flat-to-flat measurements, proportional to ~1/3 of the swelling, for these positions [11], and the relative change in density (Table 3), the latter calculated using the density value at −1200 mm as a reference zero-swelling state.



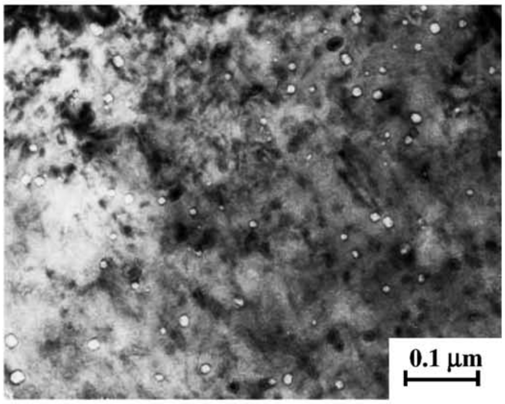
"–1200 mm" - No swelling

"–900 mm"



"–500mm"

"0 mm"



"+500 mm"

Fig. 1. Microstructure of 08Cr16Ni11Mo3 irradiated in the BN-350 reactor at various distances from midplane.

Table 2
Microstructural data on voids

Distance from midplane (mm)	Range for void sizes (nm)	Mean void diameter (nm)	Size of defect max. fraction (nm)	Void density ($\times 10^{15}$ cm $^{-3}$)	Swelling (%)
−1200	–	–	–	No voids	–
−900	<7	–	–	Some voids	–
−500	10–15	10.0	8.0	0.61	0.04
0	4–15	8.6	10.0	2.57	0.13
+500	10–35	14.0	10.0	0.78	0.16

Table 3
Data on hydrostatic weighing and microhardness measurements

Distance from midplane (mm)	Flat-to-flat size change (%) [10]	Density (g/cm^3)	$\Delta\rho/\rho$ (%)	H_v (kg/mm^2)
−1200	0.01	7.972	–	244
−900	0.02	7.961	0.13	286
−500	0.03	7.946	0.3	412
0	0.03	7.947	0.3	380
+500	0.04	7.934	0.4	313

3.3. Microhardness measurements

Microhardness results are presented in Table 3. Microhardness was lowest in the bottom-most specimen of the duct (−1200 mm), where temperature and fluence were also lowest, and reaches a maximum at −500 mm. The hardness appears to increase with dpa level and decrease with temperature, as would be expected.

4. Conclusions

It appears that the irradiation of 08Cr16Ni11Mo3 austenitic steel at low neutron fluxes leads to a recovery process that alters the grain morphology, where the mean grain size increases with temperature and possibly with the total dpa level. This recovery process does not appear to produce deleterious results, however. Concurrently, microstructural evolution proceeds as first Frank loops are produced and then voids at temperatures above 280 °C, with measurable increases in resultant hardening.

In agreement with earlier studies on various austenitic steels voids are observed at very low doses and temperatures in such low flux irradiations, with the first voids observed at only 281 °C and 1.3 dpa.

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References

[1] S.I. Porollo, Yu.V. Konobeev, A.M. Dvoriashin, V.M. Krigan, F.A. Garner, in: 10th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, issued in CD format.

[2] V.S. Neustroev, V.K. Shamardin, Z.E. Ostrovsky, A.M. Pecherin, F.A. Garner, in: International Symposium on Contribution of Materials Investigation to the Resolution of Problems Encountered in Pressurized Water Reactors, 14–18 September 1998, Fontevraud, France, p. 261.

[3] V.S. Neustroev, V.N. Golovanov, V.K. Shamardin, Z.E. Ostrovskiy, A.M. Pecherin, in: Proceedings of the 7th Russian Conference on Reactor Material Science, 8–12 September 2003, in press.

[4] K. Fujii, K. Fukuya, G. Furutani, T. Torimaru, A. Kohyama, Y. Katoh, in: 10th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, issued in CD format.

[5] N.I. Budylkin, T.M. Bulanova, E.G. Mironova, N.M. Mitrofanova, S.I. Porollo, V.M. Chernov, V.K. Shamardin, F.A. Garner, in: The Strong Influence of Displacement

Rate on Void Swelling in Variants of Fe-16Cr-15Ni-3Mo Austenitic Stainless Steel Irradiated in BN-350 and BOR-60, this conference. doi:10.1016/j.jnucmat.2004.04.344.

[6] D.J. Edwards, E.P. Simonen, F.A. Garner, B.A. Oliver, S.M. Bruemmer, J. Nucl. Mater. 317 (2003) 32.

[7] T. Okita, T. Sato, N. Sekimura, F.A. Garner, L.R. Greenwood, J. Nucl. Mater. 207–211 (2002) 322.

[8] T. Okita, T. Sato, N. Sekimura, F.A. Garner, W.G. Wolfer, 11th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 2003, issued on CD format.

[9] V.S. Neustroev, V.K. Shamardin, Z.E. Ostrovsky, A.M. Pecherin, F.A. Garner, in: 19th International Symposium, ASTM STP 1366, M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L. Grossbeck (Eds.), American Society for Testing and Materials, p. 792, 2000.

[10] O.P. Maksimkin, K.V. Tsai, L.G. Turubarova, T. Doronina, F.A. Garner, Fusion Reactor Materials Semiannual Report for period ending December 31, 2003.

[11] V.N. Karaulov et al., Proceedings of the 2nd International Conference on Nuclear and Radiation Physics, 8–11 October 1997, Almaty, p. 44.