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Void swelling of AISI 321 analog stainless steel irradiated at low dpa rates in the BN-350 reactor

O.P. Maksimkin [a], K.V. Tsai [a], L.G. Turubarova [a], T. Doronina [a], F.A. Garner [b,*]

[a] *Institute of Nuclear Physics, Almaty, Kazakhstan*
[b] *Pacific Northwest National Laboratory, Richland, WA 99354, United States*

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 or less, when produced by neutron irradiation at dpa rates in the range 10^{-7}–10^{-8} dpa/s. Other studies yielded similar results for AISI 316 and the Russian analog of AISI 316. In the current study a blanket assembly duct from BN-350, constructed from the Soviet analog of AISI 321, also exhibits swelling at dpa rates on the order of 10^{-8} dpa/s, with voids seen as low as 281 °C and only 0.65 dpa. It appears that low-temperature swelling occurs at low dpa rates in 300 series stainless steels in general, and also occurs during irradiations conducted in either fast or in mixed spectrum reactors as shown in other studies.

1. Introduction

In a recently published study, it was shown that in annealed 12Cr18Ni10Ti, the Soviet analog of AISI 321 stainless steel, void swelling occurs in a temperature regime with a lower limit just at or above 300 °C, when irradiated in the BN-350 fast reactor at dpa rates on the order of 10^{-7}–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-type stainless steels irradiated in Japanese and European PWRs [4,5].

Previous fast reactor studies conducted in Western countries could not establish 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 fast reactors. One of the first-generation, the BN-350 fast reactor in Kazakhstan, was recently

* Corresponding author. Tel.: +1 509 376 4136, mobile: +1 509 531 2112; fax: +1 509 376 0418.
E-mail address: frank.garner@pnl.gov (F.A. Garner).

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decommissioned. It had an inlet coolant temperature of 280 °C.

Following an earlier prediction concerning higher-than-expected swelling in PWRs [6,7], a number of recent studies by Garner and coworkers have shown that void swelling in austenitic stainless steels actually increases at lower dpa rates [8–15], often allowing the observation of the lower swelling temperature limit at much lower dpa levels. This increase in swelling arises primarily 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.

Recently, it was shown that the Soviet analog of AISI 316, when irradiated as a hexagonal blanket assembly at relatively low dpa rates (on the order of 10^{-8} dpa/s) in BN-350 also exhibits swelling at unexpectedly low levels, with voids seen as low as 281 °C and only 1.3 dpa [14].

Another opportunity has recently arisen to provide additional confirmation concerning the generality of the lower temperature limit of void nucleation in austenitic stainless steels by examining another component from the BN-350 reactor that was irradiated at low neutron flux. This component was similar in irradiation history to that of the hexagonal blanket assembly constructed from the AISI 316 analog previously reported in Ref. [14].

2. Experimental procedure

A hexagonal blanket assembly designated H-214(1) was irradiated in the reflector region of the BN-350 reactor, reaching a maximum of 12.6 dpa at a maximum dpa rate of 3.8×10^{-8} dpa/s averaged over its lifetime in reactor. The hexagonal duct with faces 50 mm wide and 2 mm thick was constructed from 12Cr18Ni10Ti stainless steel, a Soviet analog of AISI 321 steel, and was produced with the final thermal–mechanical treatment of the duct being 15–20% cold deformation followed by annealing at 800 °C for 1 h.

The coolant temperature at the bottom of the assembly was 280 °C and the temperature at the top of the assembly was 430 °C. Specimens were chosen for examination between positions having calculated coolant temperatures between 281 and 333 °C. Since the inlet coolant temperature is controlled at 280 °C, the temperatures toward the bottom of the assembly are well known, but may be uncertain by ±5 °C at the upper elevations examined in this experiment. Due to the thinness of the duct wall, the internal temperature of the duct 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 any elevation.

Table 1
Dose and temperatures over the height of the H-214(1) blanket assembly

Distance from midplane (mm)	H-214(1)/12Cr18Ni10Ti		
	Dose (dpa)	Dpa rate (10^{-8} dpa/s)	Calculated temperature (°C)
−900	0.65	0.12	281
−375	7.3	1.36	294
0	12.3	2.3	313
+75	12.6	2.34	318
+375	7.3	1.35	333

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 and immersion density weighing.

The examination technique involved transmission electron microscopy (TEM), using a JEM-100 CX electron microscope operating at 100 keV. The density was measured using a hydrostatic weighing technique employing a KERN-770 electronic balance with methyl alcohol as the working liquid.

Disks of 3 mm diameter for microscopy studies were prepared from ⩽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.

3. Results and discussion

The results of the density change measurements are shown in Table 2. There definitely appears to be swelling at relatively low levels that increases gradually with axial position and thereby increasing temperature. The presence of voids can not be directly inferred from these measurements alone,

Table 2
Hydrostatic weighing results

Distance from midplane (mm)	Density (g/cm^3) (metallography specimen)	$\Delta\rho/\rho$ (%) (metallography specimen)
−900	7.888	−0.25
−375	7.856	0.15
0	7.852	0.20
+75	7.846	0.28
+375	7.817	0.65

however, since precipitation may be occurring to produce all or some portion of these density changes.

The density of non-irradiated steel 12Cr18Ni10Ti was listed in previous studies on this steel to be 7.868 g/cm^3. The point at −900 mm therefore indicates some densification has occurred. Note that this measurement was made after electrical polishing of the specimen. Another measurement made before polishing yielded 7.869 g/cm^3, indicating that no net densification had occurred.

It is known that stainless steels, especially those of lower nickel content, tend to interact during long-term exposure with sodium such that near-surface compositional changes occur, sometimes leading to the formation of a ferritic layer on the surface [16–20]. Perhaps this phenomenon accounts for the difference in density between polished and unpolished specimens, with the net density changing upon removal of the ferrite layer.

The microscopy results, however, confirm the presence of void swelling in the range 281–333 °C, as shown in Table 3 and Figs. 1–3. Most significantly, it is seen that even at 0.65 dpa and 281 °C voids are clearly visible, adding additional support to the growing body of evidence that void swelling can extend down to unexpectedly low temperatures and doses if the atomic displacement rate is low enough. Since helium generation rates in austenitic steels are comparable in light water and fusion spectra it is possible that a similar behavior of austenitic steels will be observed in fusion devices as well. For lower-flux, water-cooled designs such as ITER small amounts of void swelling may be observed.

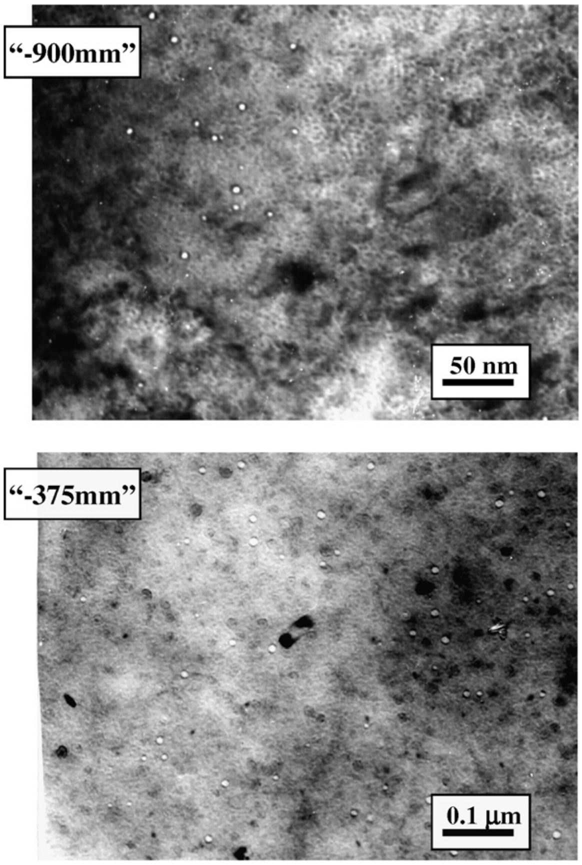

Fig. 1. Voids observed in 12Cr18Ni10Ti at 0.65 dpa, 281 °C (top) and 7.3 dpa, 294 °C (bottom) after irradiation in BN-350.

Based on the studies of Okita and coworkers at ~400 °C, it appears that the primary mechanism causing an increase in swelling at lower flux arises from the flux sensitivity of Frank loop formation and the subsequent stability of the loop ensemble against unfaulting [10,11]. It is not clear at present, however, if such a mechanism acts to move the boundary of the swelling regime to lower temperatures.

Table 3
Microstructural data on cavities for irradiated stainless steel 12Cr18Ni10Ti

Distance from midplane (mm)	Range for void sizes (nm)	Mean void diameter (nm)	Peak void diameter (nm)	Void density ($\times 10^{15}\ cm^{-3}$)	Swelling (%)
−900	<5–12	7.7	<5 nm/5–10	0.84	0.03
−375	<10–15	11.6	10	0.47	0.05
0	<10–20	11.2	10	2.9	0.25
+75	<8–18	9.0	8	8.2	0.33
+375	<10–35	15.3	15	1.0	0.23

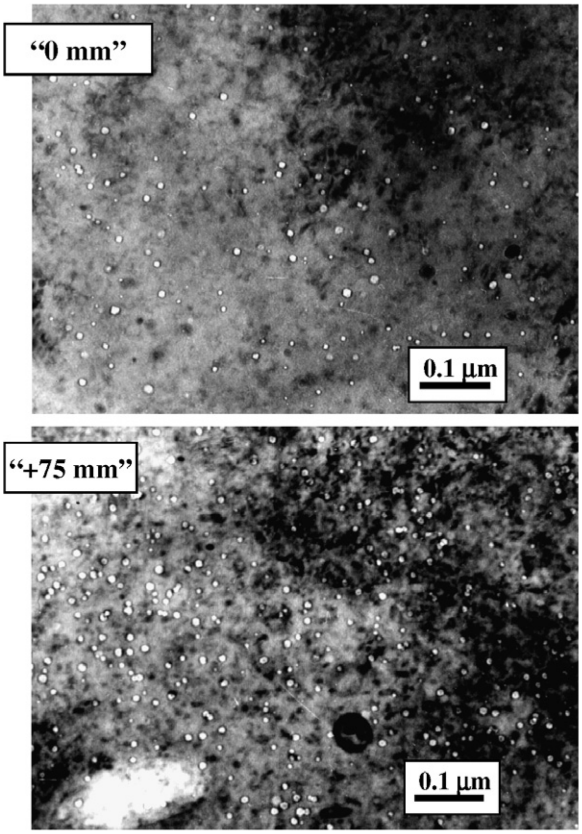

Fig. 2. Voids observed in 12Cr18Ni10Ti at 12.3 dpa, 313 °C (top) and 12.6 dpa, 318 °C (bottom) after irradiation in BN-350.

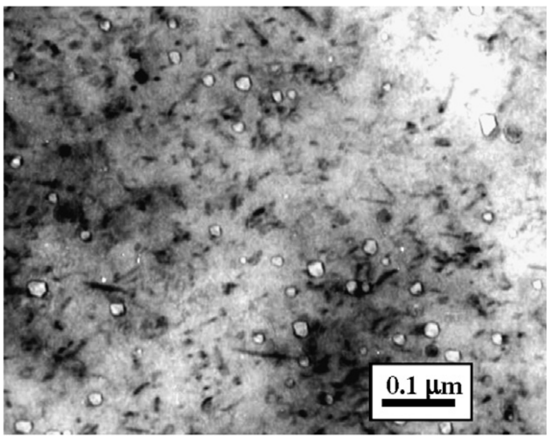

Fig. 3. Voids observed at +375 mm elevation in 12Cr18Ni10Ti at 7.3 dpa, 333 °C dpa after irradiation in BN-350.

4. Conclusions

Once again it appears that irradiation of stainless steels at progressively lower dpa rates leads to swelling occurring at lower doses and temperatures than previously expected. Such observations have been hampered in previous Western studies by the high dpa rates used in those studies and the relatively high inlet temperatures of Western reactors.

It also appears that low-temperature swelling at low dpa rates occurs in all 300 series stainless steels in general, whether Western or Russian in composition. The fact that such behavior is observed during irradiations conducted in both fast and mixed spectrum reactors implies that it will be observed in fusion devices as well.

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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, August 5–9, 2001 (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, September 14–18, 1998, Fontevraud, France, p. 261.

[3] V.S. Neustroev, V.N. Golovanov, V.K. Shamardin, Z.E. Ostrovskiy, A.M. Pecherin, in: Proceedings of 7th Russian Conference on Reactor Material Science, September 8–2, 2003 (in Russian).

[4] K. Fujii, K. Fukuya, G. Furutani, T. Torimaru, A. Kohyama and 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, J. Nucl. Mater. 329–333 (2004) 621.

[6] F.A. Garner, L.R. Greenwood, D.L. Harrod, in: Proceedings of the Sixth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, San Diego, CA, August 1–5, 1993, p. 783.

[7] F.A. Garner, Trans. Am. Nucl. Soc. 71 (1994) 190.

[8] F.A. Garner, M.B. Toloczko, J. Nucl. Mater. 251 (1997) 252.
[9] D.J. Edwards, E.P. Simonen, F.A. Garner, B.A. Oliver, S.M. Bruemmer, J. Nucl. Mater. 317 (2003) 32.
[10] T. Okita, T. Sato, N. Sekimura, F.A. Garner, L.R. Greenwood, J. Nucl. Mater. 307–311 (2002) 322.
[11] T. Okita, T. Sato, N. Sekimura, F.A. Garner, W.G. Wolfer, in: 11th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, August 2003 (issued on CD format).
[12] V.S. Neustroev, V.K. Shamardin, Z.E. Ostrovsky, A.M. Pecherin, F.A. Garner, Temperature-shift of void swelling observed at PWR-relevant temperatures in annealed Fe–18Cr–10Ni–Ti stainless steel irradiated in the reflector region of BOR-60, in: M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L. Grossbeck (Eds.), Effects of Radiation on Materials: 19th International Symposium, ASTM STP, vol. 1366, American Society for Testing and Materials, 2000, p. 792.
[13] F.A. Garner, N.I. Budylkin, Yu. V. Konobeev, S.I. Porollo, V.S. Neustroev, V.K. Shamardin, A.V. Kozlov, in: 10th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 2003, p. 647.
[14] O.P. Maksimkin, K.V. Tsai, L.G. Turubarova, T. Doronina, F.A. Garner, J. Nucl. Mater. 329–333 (2004) 625.
[15] S.I. Porollo, A.M. Dvoriashin, Yu.V. Konobeev, A.A. Ivanov, S.V. Shulepin, F.A. Garner, Microstructure and Mechanical Properties of Austenitic Stainless Steel 12X18H9T Irradiated in the Pressure Vessel of BR-10 at Very Low Displacement Rates, in this semiannual; Progress Report.
[16] W.F. Brehm, Interaction of sodium with breeder reactor materials, in: R.P. Agarwala (Ed.), Diffusion Processes in Nuclear Materials, Elsevier Science Publishers B.V., 1992, p. 323.
[17] P.T. Nettley, I.P. Bell, K.Q. Bagely, D.R. Harries, A.W. Thorley, C. Tyzack, Problems in the selection and utilization of materials in sodium cooled fast reactors, BNES, Fast Breeder Reactors, Pergamon, Oxford, 1967, p. 825.
[18] W. Charnock, C.P. Haigh, C.A.P. Horten, P. Marshall, CEBG Research, November 1979, p. 3.
[19] I.I. Balachov, F.A. Garner, Y. Isobe, M. Sagisaka, H.T. Tang, in: Eleventh International Conference on Environmental Degradation of Materials in Nuclear Systems – Water Reactors, 2003, p. 640.
[20] S. Ukai, E. Yoshida, Y. Enokido, I. Nihei, in: Proceedings of Materials for Nuclear Reactor Core Applications, BNES, London, 1987, p. 341.