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SCIENCE DIRECT
Journal of Nuclear Materials 351 (2006) 223–227
journal of nuclear materials
www.elsevier.com/locate/jnucmat

Irradiated microstructure of alloy 800H

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Abstract

Austenitic alloy 800H has the same basic composition as INCOLOY alloy 800 (Fe–20Cr–32Ni) but with significantly higher creep-rupture strength due to a required 60 μm minimum grain size. It is one of the high temperature candidate alloys being considered for Generation IV nuclear reactor systems. The radiation resistance of 800H has not been previously studied. This work provides information on the microstructural changes in 800H after irradiation using 5.0 MeV Ni ions at 500 °C to 5 and 50 dpa. Following irradiation, changes in microstructure and phase stability were studied using transmission electron microscopy (TEM). At a dose of 50 dpa, no voids were found and the density and size of the faulted loops were measured to be 2.3×10^{16} cm^{-3} and 8.4 nm, respectively. There are fine precipitates distributed in 800H with an average size approximately 6 nm and a density greater than 9.1×10^{15} cm^{-3}. The high Ni content and the presence of precipitates are believed to be responsible for the resistance to void formation at dose up to 50 dpa.
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1. Introduction

The proposed generation IV nuclear energy systems are aimed at making revolutionary improvements in economics, safety and reliability, and sustainability. To achieve these goals, Generation IV systems will operate at higher temperatures and in higher radiation fields than current light water reactors. Metallic alloy components will experience unprecedented microstructural and mechanical property evolution as they progress to higher doses. Of the candidate alloy systems that could be considered for high temperature application, alloy 800H is expected to play an important role as structural components in Generation IV systems since it is code certified for temperature up to 760 °C for use in nuclear systems [1]. Alloy 800H has a composition of 31.6 wt% nickel and 20.4 wt% chromium. It has been shown that high Ni content (>30 wt%) in austenitic steel improves material resistance to swelling under neutron irradiation at 400 °C to a dose of 18.5 dpa [2]. The alloy also is expected to have good corrosion resistance with its high Cr content for systems such as the super critical water reactor.

Heavy-ion irradiation provides a unique approach in this exploratory task to evaluate material tolerance to radiation up to very high doses. Alloy 800H is a steel developed for high temperature application. Its microstructure evolution under irradiation up to high dose (~50 dpa) has not been

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0022-3115/$ - see front matter Published by Elsevier B.V.
doi:10.1016/j.jnucmat.2006.02.009

investigated in the past. This work was aimed at investigating the high dose effect on the irradiated microstructure of alloy 800H with focus on radiation induced dislocations, loops, cavities and precipitates. Follow-on studies are expected on materials currently being irradiated in the Advanced Test Reactor in USA and Phenix reactor in France.

2. Experiments

The composition for alloy 800H is listed in Table 1. The alloy is commercial grade and used in the as-received condition. The manufacturer of the alloy 800H specified a final heat treatment at temperature of 1177 °C for 2.25 h followed by a water quench. The thin sheet samples for irradiation were cut from a 6.35 mm thick plate and mechanically polished to a thickness of approximately 200 μm with a final finish of 0.1 μm.

Ni-ion irradiation was conducted at the Environmental and Molecular Science Laboratory at Pacific Northwest National Laboratory using 5 MeV Ni ions at 500 °C under a pressure less than 1×10^{-7} Torr with a damage rate of 1.39×10^{-3} dpa/s. The irradiation dose was calculated using TRIM program [3]. The details on calculation of atomic displacements by charged particles can be found in the reference [4]. The sample was mounted on an irradiation stage that allows temperature control through heating with electron beam and cooling with liquid nitrogen flow in a cooling channel. The ion beam was rastered over an irradiated area of 8×8 mm^2 on the sample. The irradiation temperature was monitored using both a pyrometer and a thermocouple. The 800H foils were irradiated to doses of 5 and 50 dpa. TEM discs were prepared from both unirradiated and irradiated samples.

The range for a 5 MeV Ni ion beam in stainless steel is approximately 1.5 μm. A thin layer of approximately 0.5 μm depth was removed from the irradiated side using a 5-s flash jet-polishing in a solution of 2% perchoric acid and 15% ethylene glycol in methanol at a polishing condition of 25 V and −65 °C. TEM discs were then jet-polished from the unirradiated side to perforation. Microstructure characterization was carried out using a 200 KV transmission electron microscope equipped with EDS for chemical analysis. Weak beam dark filed images of the edge-on faulted loops are recorded using rel-rod imagining technique near zone $\langle 011 \rangle$. The details of rel-rod imaging technique can be found in the references [4,5].

3. Results

The unirradiated microstructure of alloy 800H is shown in the Fig. 1. It has a dislocation density of $\sim 4 \times 10^{13}$ m^{-2}. There were no precipitates found in the unirradiated condition. For the 800H irradiated with Ni ion at 500 °C to a dose of 5 dpa, the microstructure was altered by the formation of faulted dislocation loops, shown in Fig. 2. No voids were found at 5 dpa. The irradiated microstructure is dominated by dislocation loops. The size of the faulted loops, averaged over 933 counts, is 12.5 ± 0.2 nm with a loop density of $(1.2 \pm 0.2) \times 10^{16}$ cm^{-3}. There are no radiation-induced precipitates found in the matrix of the 5 dpa sample.

For 800H irradiated to 50 dpa at 500 °C, the irradiated microstructure is dominated by faulted loops and small precipitates uniformly distributed throughout the sample. No cavities were detected under this irradiation condition. The bright field image ($g = 200$) and the rel-rod dark filed image of the faulted loops are shown in Fig. 3. The size of faulted loops, averaged over 335 counts, is 8.4 ± 0.2 nm with a loop density of $(2.3 \pm 0.4) \times 10^{16}$ cm^{-3}. The size distribution of faulted loops at 50 dpa in comparison with loops at 5 dpa is shown in Fig. 4. Note that the average loop size in the 50 dpa sample is less than that in 5 dpa sample.

The fine precipitates uniformly distributed in the matrix of 800H irradiated to 50 dpa are shown in Fig. 5. These precipitates were not seen in 5 dpa and the unirradiated samples. The size of these precipitates, averaged over 189 counts, is 5.9 ± 0.1 nm. The diffraction from these fine precipitates indicated they do not have a fixed orientation relationship with matrix. Since the dark field image of the precipitates in Fig. 5 was taken using the diffraction from the precipitates, not all the precipitates were visible in the picture. The estimated precipitate number

Table 1
Composition for alloy 800H (wt%)

Fe	C	Mn	P	S	Si	Ni	Cr	Ti	Cu	Al
Balance	0.069	0.76	0.014	0.001	0.13	31.59	20.42	0.57	0.42	0.50

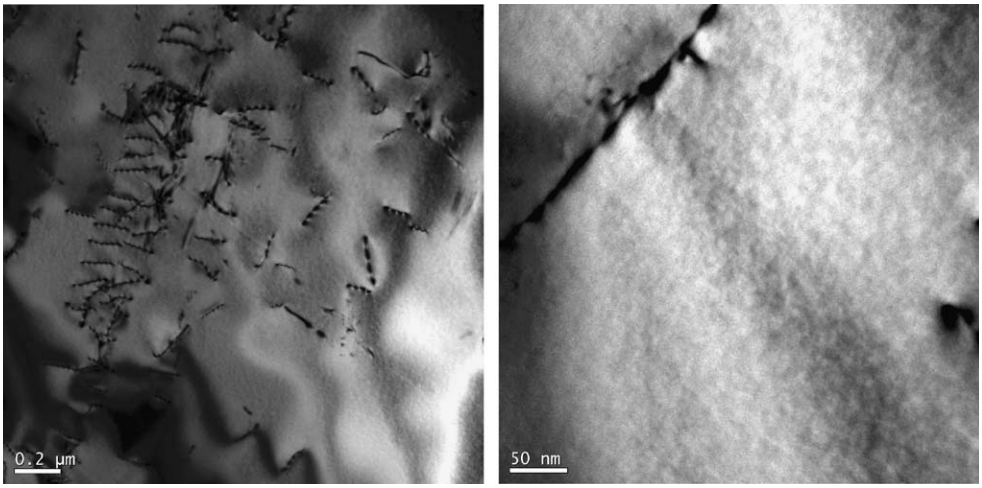

Fig. 1. Unirradiated microstructure of alloy 800H (imaged with $g = 200$ diffraction) reveals dislocations in low magnification (left) and high magnification (right). The alloy has a relatively high dislocation density considering it was heat-treated at 1177 °C for 2.25 h.

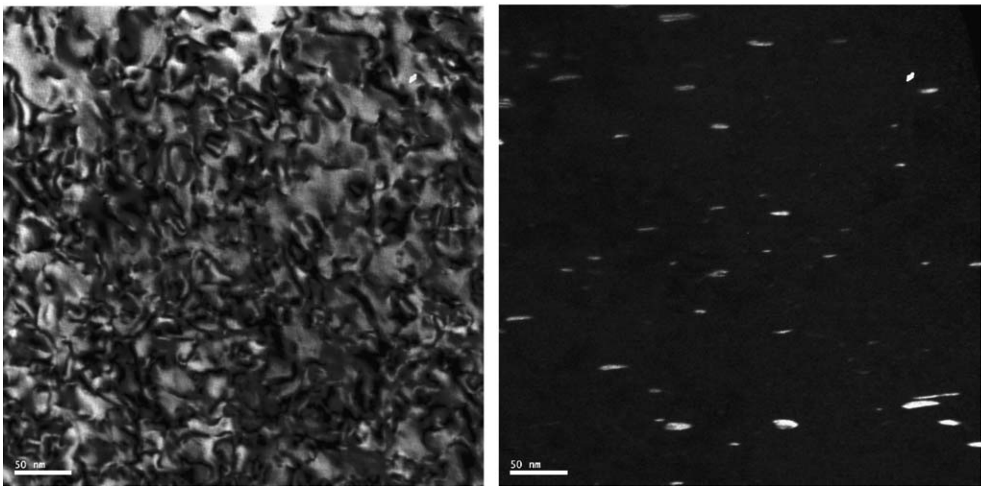

Fig. 2. Microstructure in alloy 800H irradiated with Ni ions at 500 °C to a dose of 5 dpa. Bright field image (left) showing dislocation loops under $g = 200$ and the rel-rod dark field image (right) showing 1/4 of the faulted dislocation loops.

density is greater than 9.1×10^{15} cm^{-3}. Both composition analysis and the crystal structure determination for these small precipitates in 50 dpa sample were not successful due to their small size.

4. Discussions

Alloy 800H is an austenitic solid-solution alloy. The estimated dislocation density ($\sim 4 \times 10^{13}$ m^{-2}) in the material heated, treated at 1177 °C for 2.2 h, is moderate. It was reported that 800H is free of precipitates in the solution-annealed condition [1]. However, a technical report issued by Special Metals Corporation Group, the company that invented 800H, specified that precipitates of titanium nitrides, titanium carbides and chromium carbides normally appear in the microstructure [6]. Although precipitates were not detected in the unirradiated 800H used in this work, it may just indicate the densities of these precipitates in the unirradiated condition are extremely low, likely below 10^{14} cm^{-3}. Generally the low limit for a reasonable number density measurement in microstructural characterization using TEM is approximately 10^{14} cm^{-3}.

The formation of faulted loops in austenitic steel is an important mechanism for radiation hardening [7]. In comparison to the 5 dpa sample, an increase in loop density at 50 dpa is expected but the

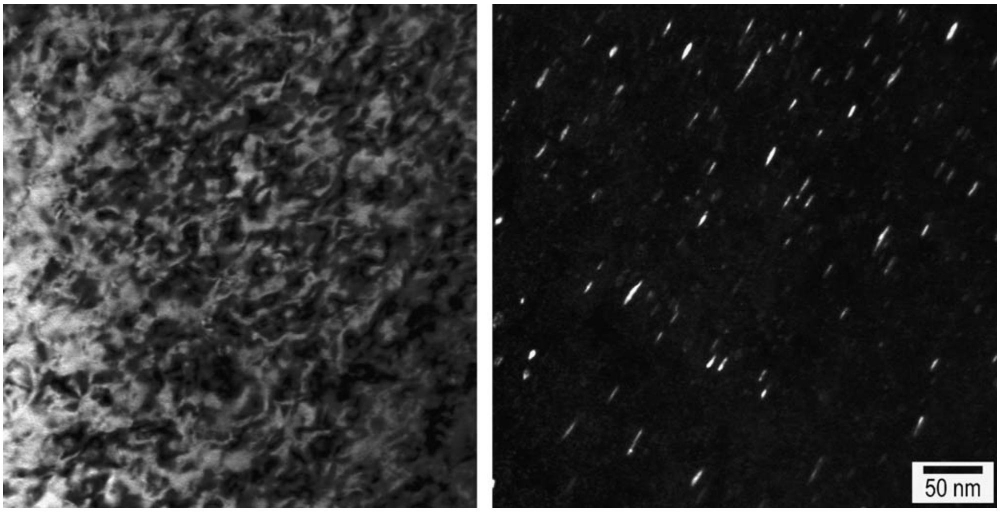

Fig. 3. Microstructure in alloy 800H irradiated with Ni ions at 500 °C to a dose of 50 dpa. Bright field image (left) showing dislocation loops under $g = 200$ and the rel-rod dark field image (right) revealing 1/4 of the faulted dislocation loops.

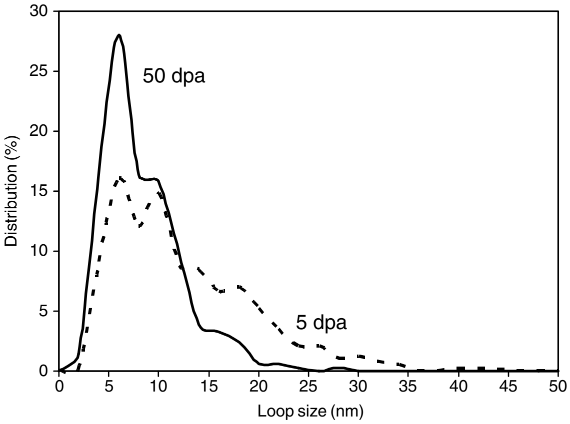

Fig. 4. Size distributions of faulted loops in alloy 800H irradiated with 5 MeV Ni ions at 500 °C to doses of 5 dpa (dot line) and 50 dpa (solid line).

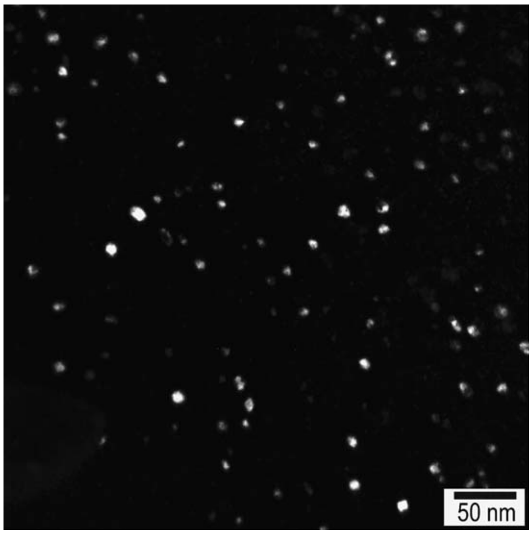

Fig. 5. Dark file image of finely distributed precipitates in 800H irradiated with Ni ions at 500 °C to 50 dpa. The image was taken using the diffraction from the precipitates.

decrease in loop size at 50 dpa was unexpected. The decrease in loop size may be due to the presence of finely distributed small precipitates developed at high dose. The uniformly distributed small precipitates are expected to play an important role in material mechanical properties. Once formed, these precipitates will act as sinks for point defects, altering the microstructural evolution under irradiation such as loop and cavity formation and growth possibly by promoting point defect annihilation at precipitates. It is believed that the presence of finely distributed precipitates may be responsible for the decrease in loop size at high dose compared to the 5 dpa sample.

At both irradiation dose of 5 dpa and 50 dpa, no voids were identified in alloy 800H. The early work on microstructure of Ni ion-irradiated 316 SS showed the presence of voids at dose as low as 2 dpa [8,9]. It appears that 800H has good resistance to void swelling. This may be attributed to the high nickel content in 800H and the presence of fine precipitates at high doses. The work by Garner and Kumar on Fe–15Cr–xNi irradiated in EBR-II at 400 °C to 18 dpa indicated that swelling dropped significantly as Ni content exceeds 30% [2]. An increase in vacancy diffusivity at higher Ni levels may be responsible for the resistance to cavity nucleation [10]. Similar to the effect of uniformly distributed fine precipitates on loops, the precipitates may act as neutral sinks to promote interstitial and vacancy recombination and therefore delay the void nucleation.

The use of Ni ion irradiation study provides an economic and quick screening test to evaluate the material microstructure evolution under a well-controlled irradiation temperature and doses due to its non-radioactive nature and high dose rate to reach high doses. However, the high damage rate requires a temperature up-shift which often limits its use to study temperature sensitive processes such as precipitation under irradiation. A neutron irradiation study is planned to confirm the ion irradiation results and understand the materials response under relatively long-term neutron irradiation. This work investigated two very limited irradiation conditions and more work on alloy 800H is necessary to further understand the microstructural evolution under various conditions relevant to its application in generation IV nuclear reactor systems.

5. Conclusions

Alloy 800H was irradiated with Ni ions at 500 °C to does of 5 and 50 dpa. Faulted loops were produced with a size and density of 12.5 nm and 1.2×10^{16} cm^{-3} at 5 dpa and 8.4 nm and 2.3×10^{16} cm^{-3} nm at 50 dpa. Irradiation to 50 dpa also produced a microstructure populated by fine precipitates uniformly distributed in the matrix with a size of 5.9 nm and density greater than 9.1×10^{15} cm^{-3}. No voids were found at either dose. The smaller loop size at high dose is believed due to the presence of fine precipitates which suppressed the loop growth. The lack of voids at 50 dpa is likely due to the combined effect of high Ni content and the presence of fine precipitates.

Acknowledgement

This work is supported by the US Department of Energy under NERI program award No. DE-FG07-03ID14542 and NERI project No. 02-110. Authors are grateful to Dr. Stephen M. Bruemmer, Sandy L. Webs and Kirk E. Bigelow for generous support for the access to their facility at Pacific Northwest National Laboratory for sample preparation.

References

[1] K. Natesan, A. Purohit, S.W. Tam, C.A. Greene., ‘Materials Behavior in HTGR Environments’, ANL-02/37 and NUREG/CR-6824, 2003.

[2] F.A. Garner, A.S. Kumar, in: Radition-Induced Changes in Microstructure: 13th International Symposium (part I), in: F.A. Garner, N.H. Packan, A.S. Kumar (Eds.), ASTM STP, 955, ASTM, Philadelphia, 1987, p. 289.

[3] J.F. Ziegler, J.P. Biersack, U. Littmark, TRIM97 program, IBM Corp., Yorktown, New York, 1997.

[4] J. Gan, PhD dissertation ‘Microstructure Evolution in Proton-irradiated Austenitic Fe–Cr–Ni alloys under Light Water Reactor Conditions’, The University of Michigan, 1999.

[5] Christine Brown, in: J.V. Venables (Ed.), Developments in Electron Microscopy and Analysis, Academic Press, 1976, p. 405.

[6] Alloy 800H technical report, Publication Number SMC-047, Special Metals Corporation, 2004 (March 04) p. 7.

[7] G.E. Lucas, J. Nucl. Mater. 206 (1993) 287.

[8] J. Gan, E.P. Simonen, D.J. Edwards, S.M. Bruemmer, B.H. Sencer, L. Founier, G.S. Was, Microstructure evolution in charged particle irradiated 316 SS modified to reduce radiation damage, in: G.E. Lucas, L. Snead, M.A. KirkJr., R.G. Elliman (Eds.), Proceedings of MRS, Symposium of Microstructural Process in Irradiated Materials, vol. 560, 2000.

[9] J. Gan, E.P. Simonen, S.M. Bruemmer, L. Fournier, B.H. Sencer, G.S. Was, J. Nucl. Mater. 325 (2004) 94.

[10] W.A. Coghlan, F.A. Garner, in: Effect of Radiation on Materials; 13th International Symposium, in: F.A. Garner, N.H. Packan, A.S. Kumar (Eds.), ASTM STP, 955, ASTM, Philadelphia, 1987, p. 315.