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ScienceDirect
Journal of Nuclear Materials 356 (2006) 118–121
journal of nuclear materials
www.elsevier.com/locate/jnucmat

Microstructural evolution of SINQ irradiated austenitic stainless steels

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Abstract

A type 316 stainless steel 316F and Ti-modified type 316 stainless steel JPCA irradiated in SINQ were examined using transmission electron microscopy. Estimated irradiation temperatures for two 316F specimens were 250 and 300 °C and that for the JPCA specimen was 255 °C. Irradiation damage of these specimens is calculated to be about 10 dpa. In the 316F specimen irradiated at 300 °C, Frank loops up to 30 nm were observed and larger perfect loops up to 50 nm were observed. Numerous defect clusters smaller than 5 nm were also observed. Stacking fault tetrahedra up to 2 nm were also observed. Dislocation loops up to 20 nm were observed in the JPCA specimen. They are smaller than those observed in 316F and it is reasonable considering the lower irradiation temperature of the JPCA specimen and lower dislocation bias expected in JPCA.

1. Introduction

Type 316 stainless steels have been used in various nuclear applications, and their radiation response has been studied with various neutron sources. It has been shown that their microstructural evolution depends on the neutron spectrum [1] due to different rates of transformation products, such as helium and hydrogen. He is produced in reactor-irradiated austenitic stainless steels via two-step transmutation of Ni58 caused by thermal neutrons while displacement damage is caused by fast neutrons. Type 316 stainless steels are also considered as the candidate material for the beam window of spallation neutron sources, which will be subject to high-energy proton/neutron irradiation. Even higher He/H production than fusion condition is expected in spallation source windows due to high-energy protons. Production rates of these gas elements in a spallation source application are higher than in any other nuclear applications and our knowledge on the microstructural evolution under such a condition is not sufficient.

Two heats of Japanese type 316 stainless steels were irradiated in the SINQ target irradiation program (STIP-I) [2]. 316F is a typical type 316 stainless steel and JPCA is a type 316 stainless steel containing 0.22% Ti. Titanium in JPCA drastically reduces its swelling in various irradiation conditions [3–5]. In this study, microstructural evolution of these two 316 steels has been examined using transmission electron microscopy (TEM).

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0022-3115/$ - see front matter
doi:10.1016/j.jnucmat.2006.05.007

2. Experiment

The chemical composition of 316F is 16.79Cr, 13.95Ni, 2.34Mo, 0.23Mn, <0.001Co, 0.04C, 0.04Si, <0.003P, 0.002S, 0.011N, bal. Fe (wt%). The chemical composition of JPCA is 14.14Cr, 15.87Ni, 2.29Mo, 1.54Mn, 0.22Ti, 0.028Co, 0.004B, 0.058C, 0.50Si, 0.026P, 0.004S, 0.003N, bal. Fe. 316F was solution annealed at 1060 °C for 15 min and JPCA was solution annealed at 1100 °C for 60 min. JPCA is modified not only by the Ti-addition but also by changing the Cr and Ni contents to stabilize austenite. TEM disks with 3 mm in diameter and 0.25 mm in thickness were loaded in the 4th and the 3rd rod of SINQ-target 3. Specimens used in the present study and their irradiation conditions are summarized in Table 1. Calculated results [6] suggest the He and H production in these specimens would be 500 and 4000 appm, respectively. Irradiated disks were transferred from SINQ to Japan Atomic Energy Agency (JAEA) and from which TEM foils were prepared. Two disks were used to prepare TEM foil by a focused ion beam (FIB) with a micro-sampling system installed in JAEA Hot Laboratory. FIB-prepared specimens were further finished with 200 eV Ar sputtering to remove damaged layers. Another specimen was electrolytically polished to obtain TEM foil. Specimens were examined by TEMs operated at 200 kV. Bright and weak beam dark field images using $g = \langle 002 \rangle$ were used to identify dislocation microstructure and slightly-defocused bright field images in kinematical conditions were used to identify cavities.

Table 1
Specimens used in this study

ID	G^4	G^8	F^{10}
Material	316F	316F	JPCA
Irradiation position	4/MA	3/RD	3/RC
Irradiation dose	10.6 dpa	10.4 dpa	10.6 dpa
Irradiation temperature	300 °C	250 °C	255 °C
Thinned by	FIB	Electrolysis	FIB

3. Results and discussion

No cavities could be observed in these specimens. The resolution limit of cavities depends on foil conditions. Specimens of 316F were successfully prepared, and the foil thickness and resolution limit of cavities were estimated to be about 30 nm and 5 nm, respectively. The foil of JPCA prepared by a FIB was not as good as that of 316F and the foil thickness and cavity resolution were estimated to be somewhat larger, 60 nm and 10 nm, respectively. Cavities with a bimodal size distribution have been often observed in austenitic stainless steels irradiated at about 400 °C and higher. Present irradiation temperature (250–300 °C) is lower than that temperature regime. JPCA irradiated in HFIR at 300 °C up to 34 dpa contained only tiny bubbles of about 3 nm in diameter [3]. Present specimens may contain small cavities below the resolution limit. The cavity evolution in the SINQ-irradiated JPCA, which contains much more He and H than that irradiated in HFIR, is believed to be not large as can be seen in

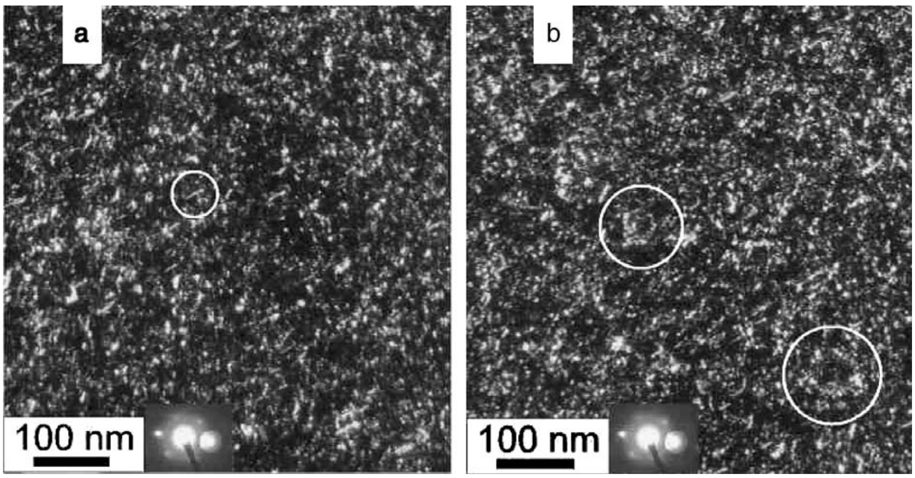

Fig. 1. Weak beam dark field images with $B = 011$, $g = 200$ showing dislocation microstructure of 316F irradiated at 300 °C (G^4). Faulted Frank-loops in an edge-on condition (a) and un-faulted perfect loops (b) are observed.

Ref. [8], because many tiny cavities stabilized by helium become the dominant sink [1].

In the 316F specimen irradiated at 300 °C (G^4), Frank loops up to 30 nm were observed and larger perfect loops up to 50 nm were observed (Fig. 1). Numerous defect clusters smaller than 5 nm were also observed. Most of them contain Moiré fringes and this suggests that they are precipitates. Stacking fault tetrahedra (SFT) up to 2 nm in diameter were observed only in the electrolytically polished specimen, G^8 (Fig. 2). SFT could also be formed in JPCA, as the quality of foil prepared with FIB might be not sufficient. Dislocation loops up to 20 nm were observed in the JPCA specimen, F^{10} (Fig. 3). They are smaller than those observed in the 316F specimen, G^4. It is reasonable considering the lower irradiation temperature of the JPCA specimen and lower dislocation bias expected in JPCA. The foil condition of the JPCA specimen was, unfortunately, not enough good to identify that the loops faulted ones or unfaulted ones. Small defect clusters observed in the 316F specimens were not observed in JPCA specimens. It is probably due to the foil condition of the JPCA specimen.

JPCA has been optimized to withstand severe neutron irradiation. Its advantage is more predominant at higher irradiation temperatures. At present irradiation conditions, no large difference was observed in the microstructure of JPCA and 316F. Present results are somewhat consistent with the previous report on fatigue tests where these two steels showed similar results [7].

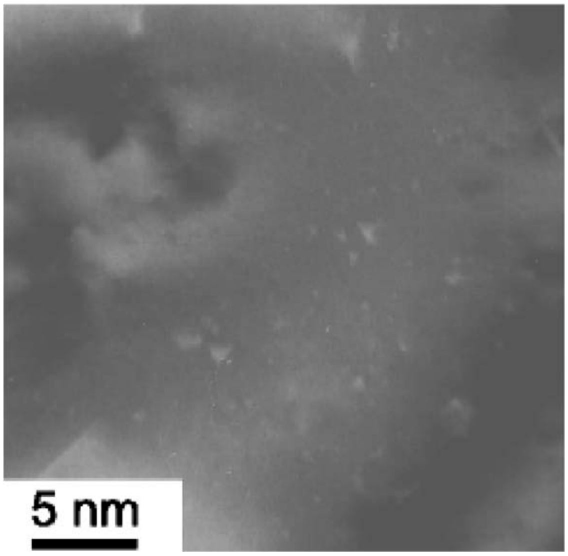

Fig. 2. Stacking fault tetrahedra (SFT) observed in electrolytically polished 316F specimen, G^8.

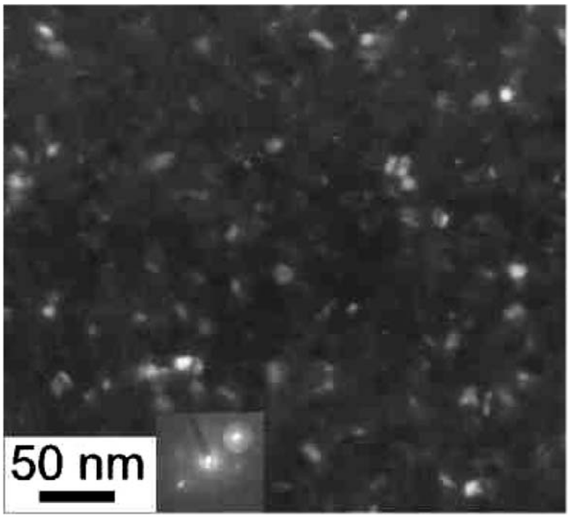

Fig. 3. Dislocation microstructure of JPCA irradiated at 250 °C. Diffraction condition was similar to that of Fig. 1.

4. Conclusions

A type 316 stainless steel 316F and Ti-modified type 316 stainless steel JPCA irradiated in SINQ at 250–300 °C were examined using transmission electron microscopy. In 316F specimen irradiated at 300 °C, Frank loops up to 30 nm were observed and larger perfect loops up to 50 nm were observed. Loops up to 20 nm were observed in JPCA irradiated at 255 °C. SFT up to 2 nm were observed in 316F specimen prepared by electrolytic polishing. These two steels, which will show quite different microstructural evolution at 400 °C and higher due to Ti in JPCA [5], showed little difference in the microstructural evolution in the present irradiation conditions.

Acknowledgement

The help provided by the Paul Scherrer Institute on irradiation in the SINQ target is highly acknowledged.

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