MICROSTRUCTURAL EVOLUTION OF COLD-WORKED AUSTENITIC STAINLESS STEELS IRRADIATED TO 17 DPA IN SPECTRALLY TAILORED EXPERIMENTS OF THE ORR AND HFIR AT 400°C — E. Wakai (Japan Atomic Energy Research Institute), N. Hashimoto, J. P. Robertson (Oak Ridge National Laboratory), T. Sawai, and A. Hishinuma (JAERI)

OBJECTIVE

The purpose of this work is to summarize the microstructural evolution of cold worked austenitic steels irradiated at 400 °C to 17.3 dpa in the spectrally tailored experiments of the ORR and HFIR.

SUMMARY

The microstructural evolution of cold-worked JPCA, 316R, C, K steels irradiated at 400 °C in spectrally tailored experiments of the ORR and HFIR was investigated. The helium generation rates were about 12-16 appm He/dpa on the average up to 17.3 dpa. The number density and average diameter of dislocation loops in the steels had ranges of 3×10^{21} - 8×10^{21} m^{-3} and 14.4- 23.7 nm, respectively. Carbides were formed in all steels by the irradiation, and the number density and average diameter had ranges of 2×10^{21}- 1×10^{22} m^{-3} and 3.4- 17.7 nm, respectively. The number density and root mean cube of radius for cavities were 6×10^{21} to 2×10^{22} m^{-3} and 1.2 to 2.4 nm, respectively, in these steels, and the swelling was 0.007 - 0.1% in these steels. JPCA-CW which has the lowest swelling shows the highest number density of carbides, while K-CW which has the highest swelling shows the lowest density of carbides.

PROGRESS AND STATUS

1. Introduction

One of the favored first wall and blanket concepts for near term fusion systems such as the International Thermonuclear Experimental Reactor (ITER) is a low pressure water-cooled austenitic stainless steel structure [1]. The neutron sources with a maximum energy of 14 MeV in the D-T fusion reactor create displacement damage in the first wall materials, and also produce hydrogen and helium atoms from (n, p) and (n, α) reactions. In the absence of operating fusion reactors, the necessary irradiation experience has to be gained from a partial simulation of the fusion environment using fission reactors. For austenitic stainless steels, it is possible to reproduce the damage rate, neutron fluence, and helium generation rate typical of the fusion environment using spectral tailoring [2-6]. Spectral tailoring involves progressively changing the ratio of thermal to fast neutron flux through the use of removable shields surrounding the experimental assembly [2]. In this way the two-step thermal neutron reaction with ^{58}Ni [7] can be mainly manipulated so that the ratio of helium generation rate to displacement rate (He/dpa ratio) approximates that for fusion throughout the irradiation. In this study the microstructural evolution of several types of austenitic stainless steels has been examined under the controlled He/dpa ratio. This experiment is being conducted under the DOE/JAERI Collaborative Agreement.

2. Experimental Procedure

The spectrally tailored experiments were performed in two stages. The first stage of the irradiation was carried out in the Oak Ridge Research Reactor (ORR) in capsule ORR-MFE-7J (330 and 400°C) [8-11]. After accumulating approximately 7.4 dpa in the ORR, the 400 °C specimens were transferred to the High Flux Isotope Reactor (HFIR) in capsule HFIR-RB-400J-1 for the second stage [12-14]. In each reactor, the specimens were irradiated at 400 °C. Temperatures were continuously measured and controlled in these experiments during irradiation. The thermal and fast (E >0.1 MeV) neutron fluences in the ORR were 8.1 x 10^{25} and 9.5 x 10^{25} n/m^2 [12], and those of the HFIR were 4.0 x 10^{25} and 1.6 x 10^{26} n/m^2[8], respectively. The experiments achieved a total peak damage level of 17.3 dpa. The helium concentrations generated in type 316 and JPCA stainless steel were 200 and 280 He appm, respectively, and the controlled average ratios of He/dpa were 12 and 16 appm for the 316 and JPCA, respectively, in this irradiation. The summary is given in Table 1.

Transmission electron microscopy (TEM) disks of several different austenitic stainless steels were irradiated in these capsules. The steels are the JPCA, 316R, C, and K. Chemical compositions of these alloys are given in Table 1. The JPCA steel contains boron, phosphorus, and titanium. The 316R is a standard of type 316 stainless steel. The C and K stainless steels have low carbon concentration, and they are modified exploratory alloys with titanium and/or niobium. The JPCA, 316R, C, and K alloys were solution-annealed or 20 % cold-worked before irradiation.

Microstructures of these specimens were examined using a JEM-2000FX transmission electron microscope with a LaB_6 gun operated at 200 kV. In order to evaluate defect density the foil thickness of each TEM specimen was measured by thickness fringes or by the improved CSS method [15, 16].

Table 1. Damage levels, helium concentrations, and the ratios of He/dpa of type 316 and JPCA stainless steels irradiated in the spectrally tailored experiments of the ORR and HFIR

	316 (13 wt%Ni)			JPCA (16 wt%Ni)		
	Damage (dpa)	He (atppm)	He/dpa	Damage (dpa)	He (atppm)	He/dpa
ORR (MFE-7J)	7.4	100	14	7.4	155	21
HFIR (RB-400J-1)	9.9	100	10	9.9	125	13
ORR + HFIR	17.3	200	12	17.3	280	16

Table 2. Chemical compositions of austenitic stainless steels used in this study (wt%)

Alloy	Fe	Cr	Ni	B	C	N	P	S	Si	Ti	Mn	Nb	Mo
JPCA	Bal	14 2	15.6	0 003	0 06	0.0039	0.027	0.005	0.50	0.24	1.77	-	2.3
316R	Bal.	16 8	13 5	-	0.06	-	0.028	0.003	0 61	0.005	1.80	-	2.5
C	Bal.	15 4	15 6	-	0 02	0.0018	0.017	0 007	0 51	0.25	1.56	0.08	2.4
K	Bal	18 0	17 6	-	0 02	0 004	0.015	0 005	0 48	0 29	1.46	-	2.6

3. Results and Discussion

Microstructures of austenitic stainless steels under spectrally tailored experiments

The microstructural data obtained is summarized in Table 3-5. Dislocation loops, carbides, and cavities were formed by the irradiation at 400°C to 17.3 dpa The number density and average diameter of dislocation loops in the steels had ranges of 3×10^{21}- 8×10^{21} m^{-3} and 14.4-23.7 nm, respectively. In 316-CW steels, the loops were not observed in some area of high density dislocation prior to the irradiation. In the JPCA- CW steels, the number density was somewhat higher than those of the other steels. Carbides were formed in all the steels by the irradiation, and the number density and mean size of carbides in these steels had ranges of 2×10^{21}- 1×10^{22} m^{-3} and 3.4- 17.7 nm, respectively. The steels with the lowest and highest number density of carbides were K-CW and JPCA-CW alloys, respectively. The number density of cavities were 6×10^{21} to 2×10^{22} m^{-3} in these steels. The swelling was 0.007 - 0.1% in these steels. JPCA-CW which has the lowest swelling shows the highest number density of carbides, while K-CW which has the highest swelling shows the lowest density of carbides.

In many steels, the number density of dislocation loops, carbides, and cavities in these cold-worked steels are very close to those in the solution-annealed steels[17], but the swelling in most part of CW steels is lower than that of these SA steels.

Table 4. Mean size and number density formed in these steels irradiated at 400 °C to 17.3 dpa with a helium generation rate of about 12 -16 appm He/dpa on the average throughout this experiment. *data are previous data[17].

Alloy	Mean Size (nm)	Number Density (m^{-3})
JPCA-CW	19.3	8×10^{21}
JPCA-SA*	18.7	7×10^{21}
316R-CW	14.4	5×10^{21}
316R-SA*	20.2	1×10^{22}
C-CW	23.7	3×10^{21}
C-SA*	26.3	3×10^{21}
K-CW	20.2	4×10^{21}
K-SA*	22.1	5×10^{21}

Table 5. Mean size and density of precipitates formed in these steels irradiated at 400 °C to 17.3 dpa. *data are previous data[17].

Alloy	Mean Size (nm)	Number Density (m^{-3})
JPCA-CW	3.4	1×10^{22}
JPCA-SA*	3.4	8×10^{21}
316R-CW	17.7	5×10^{21}
316R-SA*	15.1	5×10^{21}
C-CW	4.3	5×10^{21}
C-SA*	5.3	6×10^{21}
K-CW	15.1	2×10^{21}
K-SA*	19.3	1×10^{21}

Table 6. Swelling data of the cavities formed in these steels irradiated at 400 °C to 17.3 dpa with a helium generation rate of about 12 -16 appm He/dpa on the average throughout this experiment. *data are previous data[17].

Alloy	Root Mean Cube of Cavity Radius (nm)	Number Density (m^{-3})	Swelling (%)
JPCA-CW	1.3	8×10^{21}	0.007
JPCA-SA*	1.5	1×10^{22}	0.01
316R-CW	2.4	6×10^{21}	0.04
316R-SA*	1.8	6×10^{21}	0.02
C-CW	1.2	3×10^{22}	0.02
C-SA*	2.5	1×10^{22}	0.07
K-CW	2.4	2×10^{22}	0.01
K-SA*	4.0	1×10^{22}	0.3

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REFERENCES

1. G. L Kulcinski, J. Nucl. Mater., **122 & 123**(1984)457.
2. M. L. Grossbeck et al., in: Proc. Conf. on Fast, Thermal, and Fusion Reactor Experiments, Salt Lake. City, Utah, April 1985, Vol. 1, pp. 199-210.
3. A. F. Rowcliffe, A. Hishinuma, M. L. Grossbeck, and S. Jitsukawa, J. Nucl. Mater., **179-181** (1991)125.
4. R. E. Stoller, P. J. Maziasz, A. F. Rowcliffe, and M. P. Tanaka, J. Nucl. Mater., **155-157** (1988)1328.
5. T. Sawai, P. J. Maziasz, H. Kanazawa and A. Hishinuma, Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1990, Office of Fusion Energy, DOE/ER-0313/9, 1990, p. 152.
6. T. Sawai, P. J. Maziasz, H. Kanazawa, and A. Hishinuma, J. Nucl. Mater., **191-194** (1992) 712.
7. A. A. Bauer and M. Kangilaski, J. Nucl. Mater., **42**(1972)91.
8. J P. Robertson, I. Ioka, A. F. Rowcliffe, M. L. Grossbeck, and S. Jitsukawa, Effects of Radiation on Materials: 18th International Symposium, ASTM STP 1325, R. K. Nastad, et. al. Eds., American Society for Testing and Materials, Philadelphia, 1997.
9. I. I Siman-Tov, Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1987, Office of Fusion Energy, DOE/ER-0313/2, 1988, p. 18.
10. L. R. Greenwood, Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1989, Office of Fusion Energy, DOE/ER-0313/6, 1989, p. 23.

11. L. R. Greenwood, Alloy Development for Irradiation Performance Semiannual Progress Report for Period Ending March 31, 1989, Office of Fusion Energy, DOE/ER-0045/16,1986, p. 17.
12. L. R. Greenwood, C. A. Baldwin and B. M. Oliver, Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1994, Office of Fusion Energy, DOE/ER-0313/17, 1995, p. 28.
13. A. W. Longest, J. E. Pawel, D. W. Heatherly, R. G. Sitterson, and R. L. Wallace, Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1993, Office of Fusion Energy, DOE/ER-0313/14, 1993, p. 14.
14. A. W. Longest, D. W. Heatherly, E. D. Clemmer, and J. E. Corum, Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1993, Office of Fusion Energy, DOE/ER-0313/14, 1993, p. 14.
15. T. Sawai, M. Suzuki, P. J. Maziasz, and A. Hishinuma, J. Nucl. Mater., 187(1992)146.
16. T. Sawai, and M. Suzuki, Scripta Metall., 24(1990)2047.
17. E. Wakai, N. Hashimoto, T. Sawai, J. P. Robertson, L. T. Gibson, I. Ioka, and A. Hishinuma, Fusion Reactor Materials Semiannual Progress Report for Period Ending June 30, 1997, Office of Fusion Energy, DOE/ER-0313/22, 1997, pp. 159-168.