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Swelling of cold-worked austenitic stainless steels irradiated in HFIR under spectrally tailored conditions

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Abstract

The effects of cold working and impurities on swelling behavior in austenitic stainless steels irradiated at 400 °C to 17.3 dpa under spectrally tailored conditions in the Oak Ridge research reactor and high flux isotope reactor were investigated. The specimens were 20% cold-worked JPCA, 316R, K (low carbon (0.02%)) and C (low carbon (0.02%) and doped with 0.08% niobium). The helium generation rate was about 15 appm He/dpa. Cavities, dislocation loops and carbides were formed by irradiation in these steels. The swelling in the JPCA-CW and 316-CW was 0.003% and 0.004%, respectively and in the C-CW and K-CW was 0.02% and 0.01%, respectively. Swelling in K and C steels was strongly reduced by 20% cold-work, and the swelling in JPCA-CW and 316R-CW steels was comparable to JPCA-SA and 316R-SA steels. The synergistic treatments of addition of some impurities and cold working are very effective for the suppression of swelling at 400 °C in austenitic stainless steels.

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 14 MeV neutrons 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 manipulated so that the ratio of helium generation rate to displacement rate (He/dpa ratio) approximates that for fusion neutrons throughout the irradiation. Recently, the swelling behavior of solution-annealed several austenitic stainless steels was examined in specimens irradiated under the high flux isotope reactor (HFIR)/Oak Ridge research reactor (ORR) spectrally tailored experiments at 400 °C to 17 dpa [8]. In this study the microstructural evolution of several types of cold-worked austenitic stainless steels has been examined under the controlled He/dpa ratio.

2. Experimental procedure

The spectrally tailored experiments were performed in two stages. The first stage of the irradiation was carried out in the ORR in capsule ORR-MFE-7J (330 and 400 °C) [9–12]. After accumulating approximately 7.4 dpa in the ORR, the 400 °C specimens were transferred to the HFIR in capsule HFIR-RB-400J-1 for the second stage of irradiation [13–15]. In each reactor, the

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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 8.1×10^{23} and 9.5×10^{23} n/m^2, respectively [13], and those in the HFIR were 4.0×10^{25} and 1.6×10^{26} n/m^2 [9], respectively. The experiments achieved a total damage level of 17.3 dpa (7.4 dpa in the ORR and 9.9 dpa in the HFIR). The damage rates in the ORR and the HFIR were 1.8×10^{-7} and 2.2×10^{-7} dpa/s, respectively. The helium concentrations generated in type 316 and JPCA stainless steel were 200 and 280 appm He, respectively, and the controlled average ratios of He/dpa were 12 and 16 appm He/dpa for the 316 and JPCA, respectively, in this irradiation.

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 316 type 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 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 [16,17].

3. Results and discussion

Fig. 1 shows cavities in 20% cold-worked JPCA, 316R, C and K (low carbon 316 + Ti + Nb and 316 + Nb) austenitic stainless steels irradiated at 400 °C to 17.3 dpa in spectrally tailored experiments in the ORR and HFIR reactors. Larger size cavities were formed in the C and K steel, but not in the JPCA-CW and 316R-CW steels. In Fig. 2, the size distributions of cavities in the irradiated CW and SA steels are given. Note that the size distributions of cavity formed in C-CW and K-CW steels are bi-modal. The number densities of cavities were 6×10^{21}–3×10^{22} m^{-3} in these steels, and the mean cavity sizes were 1.2–2.4 nm, and they were reduced by CW. The swelling was 0.003–0.1% in the CW steels. The cavity data are summarized in Table 2.

Dislocation loops were formed by the irradiation. The loops observed in the steels were Frank type faulted loops {1 1 1} planes, which were identified by reflection from stacking faults in the weak beam dark-field images. In the JPCA-CW steel, the number density was somewhat higher than those in the other steels. The number

Table 1
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	Balance	14.2	15.6	0.003	0.06	0.0039	0.027	0.005	0.50	0.24	1.77	–	2.3
316R	Balance	16.8	13.5	–	0.06	–	0.028	0.003	0.61	0.005	1.80	–	2.5
C	Balance	15.4	15.6	–	0.02	0.0018	0.017	0.007	0.51	0.25	1.56	0.08	2.4
K	Balance	18.0	17.6	–	0.02	0.004	0.015	0.005	0.48	0.29	1.46	–	2.6

JPCA and 316R steels were solution-annealed at 1100 °C for 1 h, and C and K steels were solution-annealed at 1050 °C for 1 h before 20% cold working.

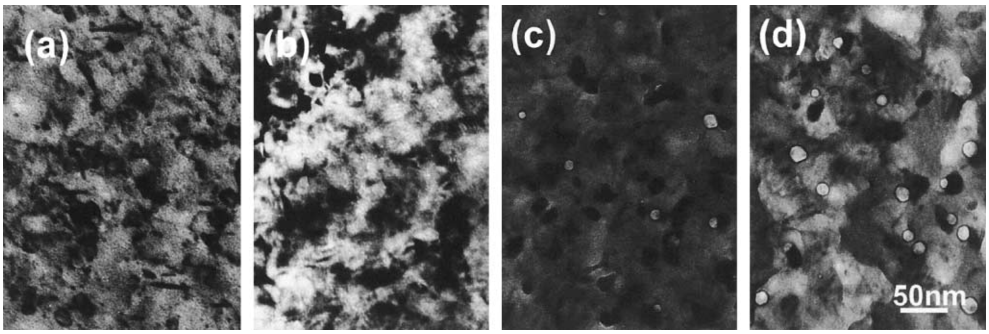

Fig. 1. Cavity formed in the CW steels irradiated at 400 °C to 17.3 dpa under spectrally tailored conditions in the ORR and HFIR: (a) JPCA-CW, (b) 316R-CW, (c) C-CW and (d) K-CW.

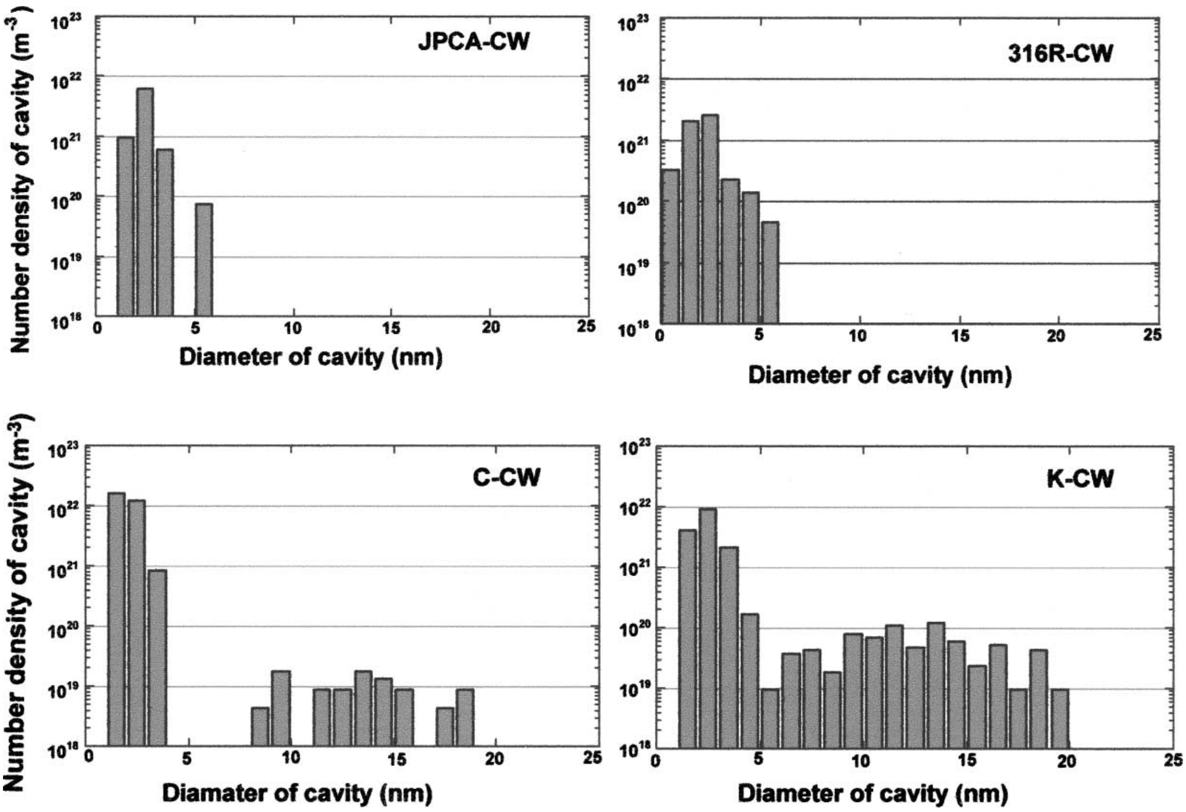

Fig. 2. Size distribution of cavities in the CW and SA steels irradiated at 400 °C to 17.3 dpa under spectrally tailored conditions in the ORR and HFIR.

Table 2
Cavity size and density and void swelling for the steels irradiated at 400 °C to 17.3 dpa under spectrally tailored conditions in the ORR and HFIR

Alloy	Root mean cube of cavity radius (nm)	Number density (m^{-3})	Swelling (%)
JPCA-CW	1.3	7.7×10^{21}	0.003
JPCA-SA[a]	1.5	1.1×10^{22}	0.016
316R-CW	1.2	5.4×10^{21}	0.004
316R-SA[a]	1.8	6.2×10^{21}	0.015
C-CW	1.2	3×10^{22}	0.02
C-SA[a]	2.5	1.1×10^{22}	0.079
K-CW	2.4	2×10^{22}	0.01
K-SA[a]	4.0	1.0×10^{22}	0.27

[a] From Ref. [8] (irradiation conditions: 400 °C, 17.3 dpa in ORR and HFIR).

density and average diameter of dislocation loops in the steels ranged between 3×10^{21}–8×10^{21} m^{-3} and 14.4–23.7 nm, respectively (see Table 3). Total length of dislocations in CW steels, which was a sum of line lengths of dislocations and dislocation loops, was higher than that in the SA steels. The lengths in the CW steels were similar, except for JPCA-CW. The increase in the total length due to 20% cold-working is likely to be not enough for the strong reduction in the swelling in austenitic stainless steels, and the difference in the swelling reduction between the steels may be related to some impurities.

Carbides were formed in the matrix of steels by irradiation. The number density and mean size of carbides in these steels ranged between 2×10^{21}–1×10^{22} m^{-3} and 3.4–17.7 nm, respectively (see Table 4). In JPCA steel, only small carbides were formed. Carbides were also formed at grain boundaries in these steels. The size

Table 3
Summary of data on dislocation loops and dislocation line density in different steels irradiated at 400 °C to 17.3 dpa. Total line density is the sum of length of dislocation loops and line dislocations

Alloy	Mean size (nm)	Number density (m^{-3})	Dislocation density of loops (m^{-2})	Dislocation density of line dislocations (m^{-2})	Total line density (m^{-2})
JPCA-CW	19.3	8×10^{21}	4.9×10^{14}	3.8×10^{14}	8.7×10^{14}
JPCA-SA[a]	18.7	7×10^{21}	4.1×10^{14}	–	4.1×10^{14}
316R-CW	14.4	5×10^{21}	2.2×10^{14}	$> 3.3 \times 10^{14}$	$> 5.6 \times 10^{14}$
316R-SA[a]	20.2	1×10^{22}	6.3×10^{14}	–	6.3×10^{14}
C-CW	23.7	3×10^{21}	2.2×10^{14}	2.9×10^{14}	5.1×10^{14}
C-SA[a]	26.3	3×10^{21}	2.5×10^{14}	–	2.5×10^{14}
K-CW	20.2	4×10^{21}	2.5×10^{14}	2.6×10^{14}	5.1×10^{14}
K-SA[a]	22.1	5×10^{21}	3.5×10^{14}	–	3.5×10^{14}

[a] From Ref. [8].

Table 4
Summary of data on carbides formed in these steels irradiated at 400 °C to 17.3 dpa

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

[a] From Ref. [8].

of carbides formed at grain boundaries was larger than that in the matrix. According to the study of the solution-annealed JPCA, 316R, C and K austenitic stainless steels [8], the carbides were identified as M_6C and MC types. The steels containing titanium as a swelling inhibitor were expected to form MC carbides on dislocation loops and to provide effective traps for helium and mediate the cavity distribution. However, carbides did not form on dislocation loops but in the matrix. Hence, the effect of titanium addition on the bias factor of dislocation loops cannot be expected in these steels.

The number density of dislocation loops, carbides, and cavities in the cold-worked steels were very similar to those in the solution-annealed steels, and therefore the influence of cold working on the nucleation of these defect clusters could be neglected. However, the increase in sinks due to dislocations induced by CW affected the cavity growth and swelling in the C and K steels. As regards the relation between swelling and impurities, very low swelling was observed in JPCA-CW and 316R-CW steels, which contain considerable amounts of carbon and phosphorus. Large size cavity hardly formed in the JPCA-CW and 316R-CW. However, in the lower carbon and phosphorus steels, i.e., C and K steels, the swelling was relatively reduced by the CW, but larger size cavities still remained, especially in K-CW steel which was doped with Ti and not doped with Nb. Considering the present study of CW steels and previous study of SA steels [8], it seems likely that the suppression of swelling may be influenced by the synergistic effects of cold working and the presence of various impurity elements.

4. Conclusion

The effects of cold working and impurities on swelling behavior in austenitic stainless steels irradiated at 400 °C under spectrally tailored conditions in the ORR and HFIR were investigated. Carbides were formed in all steels by the irradiation, and the number density and average diameter ranged between 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}–2×10^{22} m^{-3} and 1.2–2.4 nm, respectively, in these steels. The swelling in the JPCA-CW and 316-CW was 0.003% and 0.004%, respectively and in the C-CW and K-CW was 0.02% and 0.01%, respectively. Cold working reduced the swelling in all alloys by an approximate factor of 4 with the exception of K steel. For K steel, the swelling was reduced by a factor of 27. The synergistic effects of addition of some impurities and cold working in austenitic stainless steels are very effective in suppressing swelling at 400 °C.

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