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Effect of cold work on void swelling in proton irradiated Fe–15Cr–20Ni ternary alloys

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Abstract

Effect of cold work on swelling and void microstructures was investigated for Fe–15Cr–20Ni alloy irradiated with 180 keV protons to 10 dpa at 773–873 K (500–600°C). The He preinjection level was 10 appm at room temperature. The introduced dislocations in Cold-worked (CW) specimen recovered thoroughly during the irradiation for all the irradiation temperatures. Although there was no significant difference in void microstructure between Solution-annealed (SA) and CW specimens at 773 K, larger and fewer voids were observed in CW specimens at temperatures above 823 K. The amount of swelling was similar in both SA and CW specimens at all the irradiation temperatures. Cold work treatment seems to be less effective in controlling swelling of the He preinjected Fe–15Cr–20Ni alloy in the present proton irradiation. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

The radiation-induced swelling is one of the most serious problems for fusion reactor materials. Considerable progress has been made in understanding the swelling behavior of the candidate materials such as Fe–Cr–Ni austenitic alloys. It is generally recognized that cold work treatment plays a significant role in suppressing the radiation-induced swelling. A recent study, however, showed that cold work treatment could actually increase swelling of simple solute-free Fe–Cr–Ni ternary alloys [1].

It is well known that the role of cold work is strongly modified by the presence of gaseous atoms in the materials. Particularly, it has been reported that helium (He) atoms promote the formation of He bubbles attached to the dislocation lines in the cold-worked 316 SS irradiated with the neutrons [2,3], 2.5 MeV $^{4}He^{+}$-ions [4], and 200 keV C^{+}-ions [5]. Since the 14 MeV neutrons will produce gaseous atoms due to (n, α) and (n, p) transmutation reactions in the fusion rector materials, it is important to consider the influence of gaseous atoms in order to evaluate the effect of cold work on swelling.

In this study, we performed 180 keV proton irradiation to 10 dpa at temperatures from 773 to 873 K for Fe–15Cr–20Ni alloy preinjected with 10 appm He in both solution-annealed (SA) and 20% cold-worked (CW) conditions. The objective is to investigate the interactive effect of cold work and preinjected He on swelling in the simple Fe–Cr–Ni alloy.

2. Experimental procedure

The chemical compositions of Fe–15Cr–20Ni alloy used in the present experiment is given in Table 1. Cold-rolled sheets of the alloy were annealed for 120 s at 1293 K SA. The CW sheets were prepared from the SA sheets by further rolling to 20% reduction in thickness. The SA and CW sheets were spark-cut into 3 mm diameter disks. Surface layer of the disk was removed by mechanical grinding and electropolishing. The final thickness was about 120 μm at the central area of the disks prior to the He-preinjection. Disk specimens were irradiated with 50–200 keV He^{+} at room temperature so that He atoms were injected uniformly to the level of about 10 appm in the range of 200–600 nm. After the He preinjection, the specimens were irradiated with 180 keV protons at

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Table 1
Chemical composition of Fe–15Cr–20Ni alloy (wt%)

Cr	Ni	C	Si	Mn	P	S	N	O	Cu	Fe
15.04	20.12	0.0061	0.0020	<0.001	0.0009	0.0020	0.0017	0.0087	<0.001	Bal.

temperatures from 773 to 873 K. The dose level was 10 dpa at a depth of 400 nm from the irradiated surface. The dose rate was 2.0×10^{-4} dpa/s at 400 nm in depth, and the dose rate varied less than ±30% at the depth ranging from 300 to 500 nm. The experimental details of He preinjection and proton irradiation have been described elsewhere [6]. The materials were removed by electropolishing from the irradiated surface by 300 nm in thickness, and then the disks were back-side thinned for transmission electron microscopic (TEM) observation. The TEM observation was performed in the foil area of 100–200 nm thick, corresponding to the depth range of 300–x nm ($x = 400$–500) from the irradiated surface. Foil thickness was evaluated by a stereographical technique [7]. When the foil over 200 nm in thickness was obtained due to the coarsening of void microstructure, data calibration was carried out by recounting voids in the area of 200 nm thick with addressing the spatial void distribution.

3. Results

Typical void microstructures of SA and CW specimens are shown in Fig. 1. Voids and dislocation network were formed, but no precipitates were observed in all of the specimens examined. Results on microstructure and swelling are summarized in Table 2.

Fig. 2 shows the temperature dependence of (a) average void diameter, (b) void number density, (c) dislocation density and (d) swelling in SA and CW specimens. There was no significant difference in void microstructure between SA and CW specimens at 773 K, but larger and fewer voids were observed in CW specimen at 823 and 873 K (Fig. 2(a) and (b)). Although the lower density of dislocations was observed in CW specimen at 823 K, dislocation density was similar in both SA and CW specimens at 773 and 873 K, as shown in Fig. 2(c). This indicates that the dislocations introduced by cold work recovered thoroughly during the irradiation. The amount of swelling shown in Fig. 2(d) was similar in both SA and CW specimens at all the irradiation temperatures.

4. Discussion

The effect of cold work on swelling of complex alloys has been well studied in many investigations. Qian et al. [8] have reported void swelling of SA and 20% CW 316 SS under 1 MeV electron irradiation in HVEM at temperatures ranging from 673 to 993 K. The amount of swelling in 316 CW was lower than that in 316 SA at all the irradiation temperatures. Smaller voids with higher number density were observed in 316 CW, and dislocation density of 316 CW was higher than that of 316 SA. Similar tendency has also been reported for the CW complex alloys in case of neutron [2,3], He-ion [4] and C-ion irradiations [5]. Liu et al. [9] have explained the influence of cold work through the interaction between dislocations and the radiation-induced point defects. Higher dislocation density provides more void nucleation sites at the early stage of irradiation where the dislocation can climb, because the dislocations act as bias sinks for the radiation-induced interstitials. However at the later stage of irradiation where the dislocation network is formed, the interstitials and vacancies tend to annihilate as a result of recombination at dislocations. Since void grows and swelling increases substantially at the later stage, the higher dislocation density causes resistance to void growth and swelling due to the lower concentration of the radiation-induced free point defects. The radiation-induced precipitates formed during the irradiation play a significant role of maintaining the higher dislocation density in CW specimen through pinning the dislocations. Therefore, it is generally accepted that the role of cold work is to suppress void growth and swelling of complex alloys, while void nucleation can be enhanced by cold work.

As compared with the studies for complex alloys, only a few studies have explored the effect of cold work on swelling of Fe–Cr–Ni ternary alloys. Garner et al. [1] have recently pointed that the role of cold work was not always to suppress swelling of Fe–Cr–Ni ternary alloys irradiated in the FFTF reactor to about 30 dpa at 793–873 K. Although swelling was suppressed by cold work at 693 K, CW specimens swelled more than SA specimens at 793 and 873 K. They have suggested that the enhanced void nucleation by cold work would increase swelling at higher temperatures where void nucleation is retarded in SA specimens. In these experiments, however, the quantitative data of void number density, void size and dislocation density were not reported so that the more detail mechanism could not be clarified.

In the proton irradiation for the Fe–15Cr–20Ni alloy, however, another effect of cold work different from Garners suggestion [1] must be acting to modify microstructure development, since lower density of voids

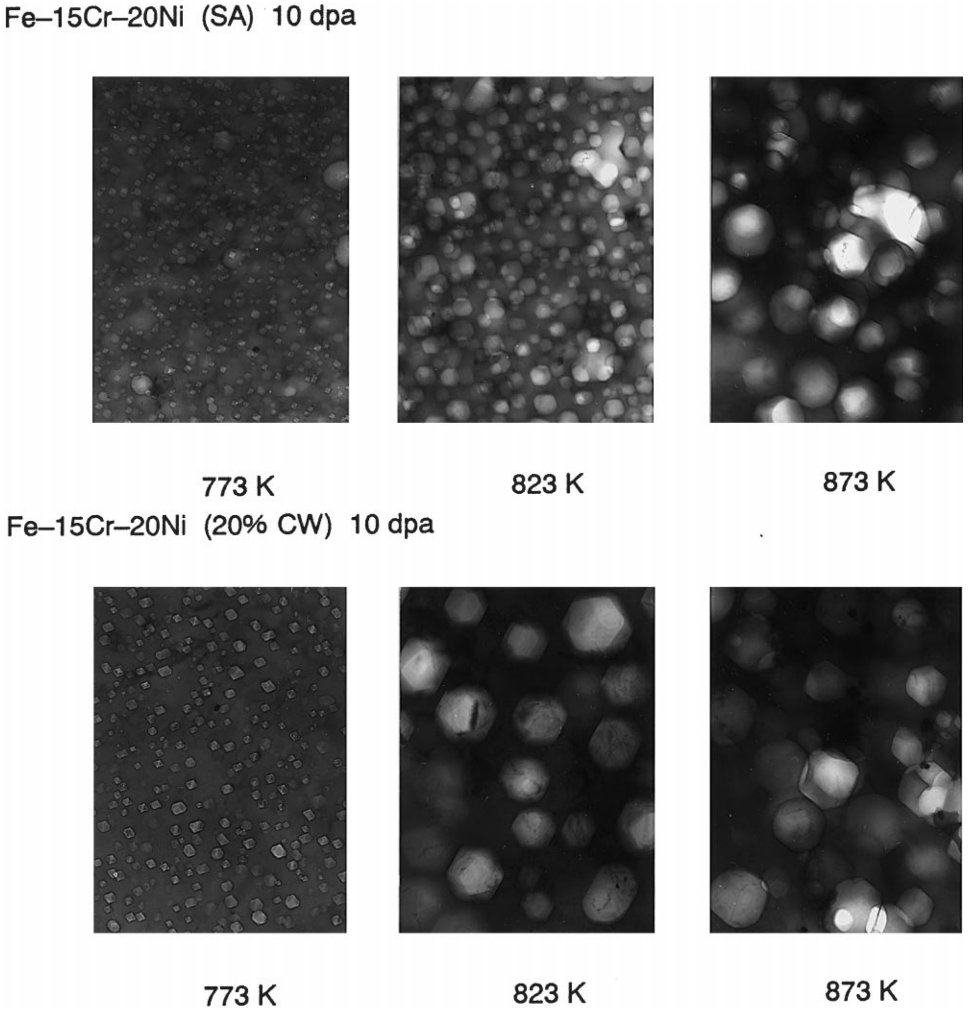
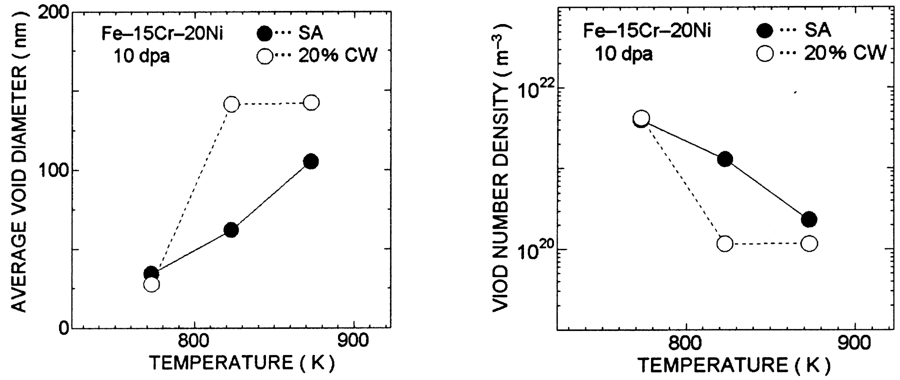
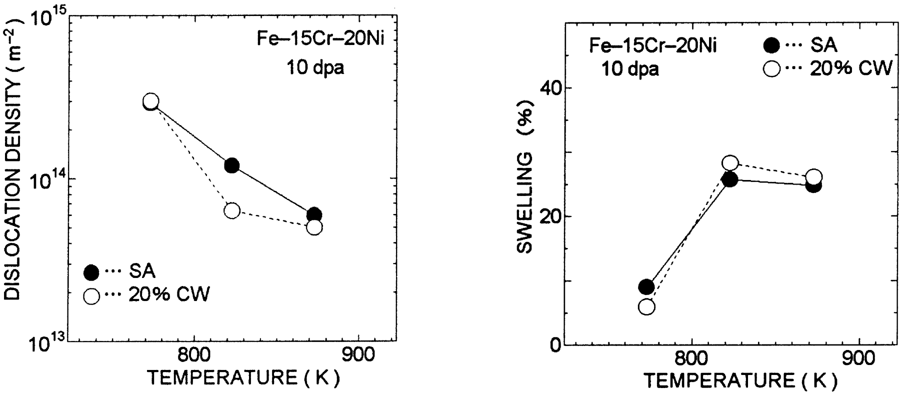


Fig. 1. Typical void microstructures of SA and CW specimens.

was observed in CW specimen at temperatures above 823 K (Fig. 2(a) and (b)). Chen et al. [4] have pointed out the interaction between He-bubbles and dislocations in microstructure development. They have performed 2.5 MeV $^4He^+$-ion irradiation for 20% cold worked 316L SS and reported the drastic coarsening of void microstructure at 873 K, while spherical voids with high density and small size were observed at 773 K. In the specimen irradiated at 873 K, dislocation belts produced by cold work were reported to disappear partly in the area with dense He-bubbles, but not in the area with few He-bubbles [4]. This interaction between He and dislocations would be asserted to the present results of CW specimens above 823 K. The introduced dislocations by cold work are likely to drag the preinjected He during the recovery of dislocations. Redistributed He induces localized dense voids and subsequently frequent void coalescence occurs in the process of void growth. Therefore, the drastic coarsening of void microstructure of CW specimens at 823 and 873 K (Fig. 2(a) and (b)) is suggested to be caused by void coalescence as a result of the redistributed He through the interaction between He and dislocations. In contrast, similar void microstructure was observed in both SA and CW specimens at 773 K (Fig. 2(a) and (b)). The less interaction between He and dislocations has been suggested by Chens experiment [4] at such temperature. The effect of cold work on void microstructure may be suppressed by the preinjected He at 773 K where the preinjected He is more stable in the material. The amount of swelling was little dependent on cold work at all the irradiation temperatures (Fig. 2(d)), irrespective of the remarkable difference in void microstructure between SA and CW specimens at 823 and 873 K. Cold work treatment seems to be less effective in controlling swelling in the present proton irradiation. It is difficult to follow the diminishing



(a) average void diameter (b) void number density



(c) dislocation density (d) swelling

Fig. 2. Temperature dependence of void microstructure parameters in SA and CW specimens.

effect of cold work on swelling, because some complexity remains in evaluation how the dynamic change of sink strength (i.e. recovery of dislocations, void coalescence) influences the amount of swelling during the irradiation. However at least, the extensive recovery of the introduced dislocations in CW specimens gives similar amount of dislocation density in both SA and CW specimens. Furthermore, the preinjected He is working to facilitate the formation of stationary dislocation network even in SA specimens, because the nucleation of dislocation loops and subsequent formation of tangled dislocations was reported to be promoted by He [10].

Table 2
Microstructure and swelling data of SA and CW specimens at 10 dpa

Irradiation temperature (K)	Average void diameter (nm)	Void number density ($\times 10^{21}$ m^{-3})	Dislocation density ($\times 10^{14}$ m^{-3})	Swelling (%)
SA specimens				
773	34.2	3.97	2.9	9.0
823	61.8	1.29	1.2	25.7
873	105.0	0.23	0.59	24.8
CW specimens				
773	27.3	4.19	3.0	6.0
823	141.4	0.12	0.64	28.2
873	142.1	0.12	0.50	26.0

The early establishment of the similar dislocation network in both SA and CW alloys may contribute to the reduction in the effect of cold work on swelling.

There were some reports claiming that H play the same role as He in promoting void nucleation [6,11,12]. In the previous paper [6], it was reported that the H effect persists up to 773 K. Although the H effect cannot be distilled from the present experimental results, the He effect may be emphasized by the H effect at 773 K. Even at higher temperatures above 823 K, however, the H may affect microstructure development, since H was expected to be trapped at He-vacancy clusters and promote their growth at such higher temperatures [13]. More detail studies on the synergistic effect of H and He are definitely needed to improve the understanding of the effect of cold work in the present proton irradiation.

5. Conclusions

The 180 keV proton irradiation to 10 dpa was conducted in the temperature range of 773–873 K for Fe–15Cr–20Ni alloy preinjected with 10 appm He in both solution-annealed (SA) and 20% cold-worked (CW) conditions. The following results (1)–(3) can be drawn from the TEM examination of void and dislocation microstructures.

1. Dislocation microstructure was similar in SA and CW specimens at 773 and 873 K, although the lower dislocation density was observed in CW specimen at 823 K. The introduced dislocations by cold work appear to be recovered thoroughly during the irradiation.
2. Void microstructure was similar in both SA and CW specimens at 773 K, while larger and fewer voids were observed in CW specimens at 823 and 873 K. The preinjected He seems to control void microstructure at 773 K. The coarsening of void microstructure in CW specimens above 823 K would be caused by frequent void coalescence as a result of the redistribution of the preinjected He through their interaction with the introduced dislocations.
3. The amount of swelling was similar in both SA and CW specimens at all the irradiation temperatures. Cold work treatment seems to be less effective in controlling swelling of the He preinjected Fe–Cr–Ni ternary alloy in the present proton irradiation.

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