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THE INFLUENCE OF ¹⁰B ADDITION ON THE SWELLING SUPPRESSION BY PHOSPHORUS AND TITANIUM IN Fe-Cr-Ni

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Phosphorus and/or titanium modified Fe-16Cr-17Ni austenitic alloys doped with ¹⁰B were irradiated in FFTF/MOTA at 684 K for the purpose of investigating the influence of helium generation on the swelling suppression effects by phosphorus and titanium. The increase in void density caused by ¹⁰B addition resulted in swelling increase in phosphorus-modified alloys. The close relation between the dislocation density and the void swelling implied the enhancement of dislocation density as a possible mechanism of the swelling suppression by phosphorus addition at the present temperature.

KEY WORDS: austenitic alloys, void swelling, phosphorus effects, boron addition, helium effects

1. Introduction

Recent irradiation experiments with fast neutrons showed that the void swelling of Fe-Cr-Ni austenitic alloys was reduced by phosphorus addition at ~700 K and below without remarkable phosphide formation[1-3]. Some of the models proposed of the phosphorus effects, e.g., the helium dilution model[4], are based on the role of phosphorus as phosphide precipitates. These models may be applied at higher temperatures where copious phosphides are formed. As mechanisms unrelated to precipitation, interaction of phosphorus with interstitials[5,6] and vacancies[5,7] were proposed. The influence of helium in the precipitate-unrelated swelling suppression process by phosphorus is, however, not yet clear.

The boron addition technique has been applied to helium generation simulation for fusion conditions[8-11]. Verification studies of this technique were performed by separating boron's transmutative and chemical effects using isotopically controlled boron[9,10].

In the present study, the boron addition technique has been applied to the phosphorus and/or titanium modified Fe-Cr-Ni austenitic alloys. The combined addition of phosphorus and titanium was shown to be even more effective in suppressing the swelling[12,13]. The objective of this study is to examine the effects of helium on the role of phosphorus and titanium addition in microstructural evolution at a temperature where mechanisms unrelated to precipitation are operating.

2. Experimental Procedure

Fe-16Cr-17Ni austenitic ternary and those with addition of 0.25Ti, 0.024P, 0.1P or 0.1P+0.25Ti, doped with either 64 appm or 522 appm ¹⁰B, were produced by arc melting in pure Ar atmosphere. First, Ni-¹⁰B alloys with two ¹⁰B levels were made and then final alloys were produced out of pure Fe, Cr, P and Ti, and the Ni-10B alloys. This procedure made it possible to keep ¹⁰B in solid solution in the matrix. Because the 91% isotopically enriched ¹⁰B was used for the boron doping, the 64 appm and the 522 appm ¹⁰B doped specimens contain ~6 appm and ~52 appm ¹¹B as well. respectively. The alloys were then solution annealed at 1323 K for 30 min. The effects of 20 % cold work were also examined for the pure ternary and +0.1P+0.25Ti alloy. Irradiation was carried out in the Fast Flux Test Facility (FFTF) Below Core Canister at 684 K during cycle 11 and 11+12 to the fluences of 2.3 and $3.4 \times 10^{26} \text{ n/m}^2 (>0.1 \text{ MeV})$, which are roughly 8.4 and 12.4 dpa, respectively. The microstructures of boron-free alloys irradiated in the same condition to 8.4 dpa are available elsewhere[13]. The helium content and He/dpa ratio calculated based on the previous helium analysis[14] are listed in Table 1. After irradiation microstructures were observed with JEM-2000FX electron microscope at Oarai Facility of Tohoku University and Pacific Northwest Laboratory.

Table 1. Helium content and He/dpa ratio in the present experiment.

¹⁰ B concentration (appm)	Cycle 11 (8.4 dpa)		Cycle 11+12 (12.4 dpa)	
	He(appm)	He/dpa	He(appm)	He/dpa
<5	2	0.24	3	0.24
64	16.3	1.9	22.2	1.8
522	116.9	13.9	159.6	12.9

3. Results

Voids were observed in all specimens. The boron precipitates and related heterogeneous void distribution as observed in a previous study of Fe-Cr-Ni[11] were not observed. Fig. 1 shows voids observed at 8.4 dpa in 64 appm and 522 appm ¹⁰B doped alloys. The void size and density are very different in different alloys and at different ¹⁰B levels. Figs. 2 and 3 show void density and swelling, respectively as a function of the He/dpa ratio. The void

density and swelling are similar between the He/dpa ratio of 0.24 and 1.9 but changed significantly between 1.9 and 13.9. The alloys can be grouped into high swelling (pure ternary, +0.25Ti), medium swelling (+0.024P) and low swelling (+0.1P, +0.1P+0.25Ti) alloys. The swelling of 'high swelling' alloys decreases and that of 'low swelling' alloys increases with increasing the He/dpa ratio. The swelling decreases drastically with the increase of the phosphorus content. Titanium addition does not change significantly the microstructure.

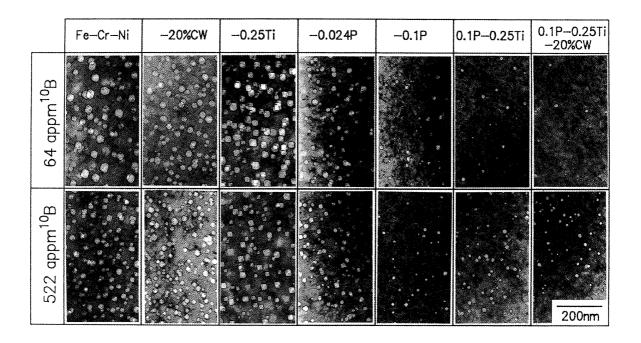
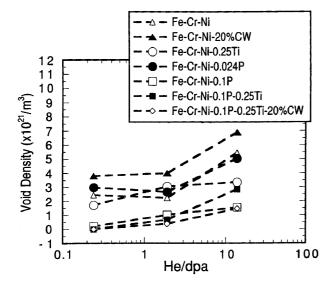
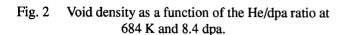


Fig. 1 Voids observed at 684 K and 8.4 dpa.





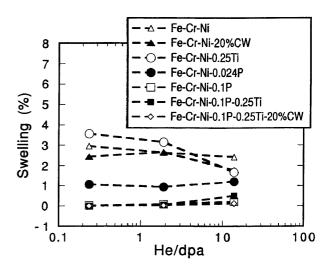


Fig. 3 Void swelling as a function of the He/dpa ratio at 684K and 8.4 dpa.

The dislocation images of the alloys doped with 64 appm¹⁰B irradiated to 8.4 dpa are shown in Fig. 4. The dislocation density increases with the increase in phosphorus content. Fig. 5 shows voids at 12.4 dpa. Figs. 6, 7 and 8 show void density, swelling and dislocation density, respectively. Figs. 7 and 8 imply that the swelling of alloys with high dislocation density is generally low. The void densities of +0.1P and +0.1P+0.25Ti alloys increase significantly with the increase of dose and ¹⁰B content. The cold work to +0.1P+0.25Ti alloy is effective in keeping void density low.

4. Discussion

The obvious relation between the swelling and the dislocation density shown in Figs. 7 and 8 suggests that the enhancement of dislocation density is one of the possible mechanisms of the swelling suppression by phosphorus addition at the present temperature. Phosphorus was shown to interact strongly with interstitials reducing their effective mobility and enhancing the loop density[5,12]. A high density of dislocation loops formed initially would result in

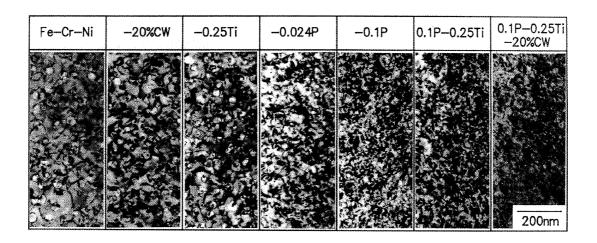


Fig. 4 Dislocation images of alloys with 64 appm¹⁰B at 684 K and 8.4 dpa.

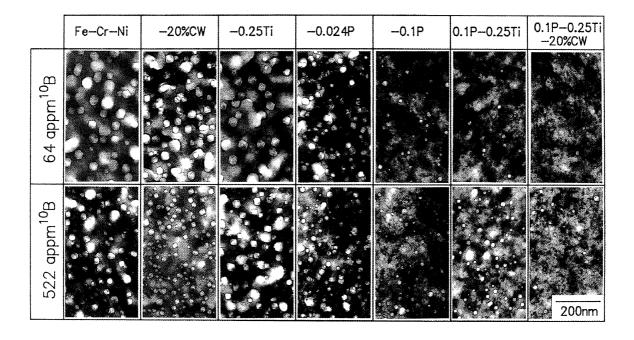


Fig. 5 Voids observed at 684 K and 12.4 dpa.

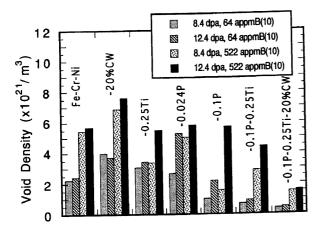


Fig. 6 Void density of the present alloys.

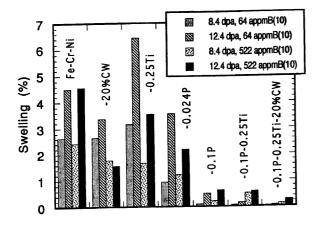


Fig. 7 Swelling of the present alloys.

a high density of dislocation network succeedingly. The increased level of helium would enhance bubble-void conversion where the void nucleation otherwise is difficult under the influence of high sink strength by dense dislocations[15].

The 20 % cold work was found to be beneficial in reducing the void density and the swelling in +0.1P+0.25Ti alloy. The dislocation density of the cold worked specimen was not much different from that of annealed +0.1P+0.25Ti alloy at either fluence. Thus it is expected that void nucleation in the annealed specimens took place at initial period of irradiation when the dislocation density was not yet too high. It was shown previously that the void formation in Fe-Cr-Ni during fast neutron irradiation at a similar temperature to the present study took place mostly at very low doses, when the dislocation network was not yet formed[16]. The cold work to the pure ternary was not effective in reducing the swelling probably because the dislocation density decreased quickly during irradiation without any pinning effects of phosphorus in solution[17].

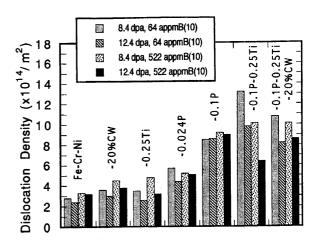


Fig. 8 Dislocation density of the present alloys.

The boron addition as a helium generation simulation has been shown to cause some side effects on microstructures. The co-generated lithium also enhances void formation in austenitic alloys[18]. Boron has, as its chemical role, suppressive effects on void formation, which is competing with its transmutative effects, in Ni and Ni-Si[9,10]. These issues are remaining to be solved in the future.

5. Conclusion

Void swelling of Fe-16Cr-17Ni was significantly reduced by addition of phosphorus, or phosphorus and titanium, without remarkable precipitation. The enhancement of dislocation density is a possible mechanism of the swelling suppression by phosphorus addition at the present temperature. The addition of ¹⁰B resulted in enhancement of void density in all alloys and of swelling in alloys with 0.1% phosphorus. The cold work to alloys with phosphorus and titanium was beneficial in keeping the void density low.

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