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Effect of mechanical alloying parameters on irradiation damage in oxide dispersion strengthened ferritic steels

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Abstract

Issues for developing oxide dispersion strengthened (ODS) steel are anisotropic mechanical properties due to the bamboo-like structure, impurity pick up during the mechanical alloying (MA) process, stability of oxide particles, heat-treatment condition and chemical composition. Several ODS steels were fabricated with a changing gas environment during MA, heat-treatment condition and chemical composition, and were electron-irradiated to 12 dpa at 673–748 K in a high-voltage electron microscope. An ODS martensitic steel (M–Ar) with high dislocation density showed very good swelling resistance. Swelling levels of ODS ferritic steels depended on the gas environment during MA and the recrystallization condition. These indicated that a helium gas environment during MA was more effective to suppress swelling than an argon gas environment and that cold working after recrystallization reduced void formation and swelling. The effect of MA parameters, such as the gas environment, heat-treat condition and cold working on the swelling behavior was evaluated. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Oxide dispersion strengthened (ODS) ferritic steels have superior high-temperature creep strength as well as excellent swelling resistance. Due to these properties, they are considered to be one of the most promising candidate materials for the first wall in fusion reactors and as cladding materials in fast breeder reactors [1–5].

Issues for ODS ferritic steel are anisotropic mechanical properties due to the bamboo-like structure, impurity pick up during MA process, stability of oxide particles, heat-treatment condition and chemical composition. Recrystallization is useful for improving the anisotropic structure [3]. On the impurity effect, in the case of argon atmosphere during MA, it was found that argon bubbles could be the nucleation sites of defect clusters during irradiation [4].

In this paper, we performed an electron-irradiation to evaluate the effects of MA parameters on the radiation response of ODS steels, such as the inert gas MA environment and cold working on void formation. Specific microstructures and irradiation-induced damage, such as swelling, dislocation density, distribution of oxide particles and phase stability of ODS martensitic steel were studied.

2. Experimental

As shown in Table 1, the alloys used in the present experiment were five kinds of ODS steels (F–Ar0, F–Ar20, F–He0, F–He20, M–Ar) and one kind of ferritic/martensitic steel (F/M) as a reference material. All the alloys were fabricated as cladding tubes. The chemical composition for the ODS ferritic steels (F–Ar0, F–Ar20, F–He0, F–He20) was 0.06C–11.8Cr–0.26Ti–0.23Y_2O_3

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Table 1
Chemical composition of F/M, F–Ar0, F–He0, M–Ar steels (wt%)[a]

	C	Cr	Ti	W	N	V	Nb	Y_2O_3	Ex. O
F/M	0.18	11.40	–	2.77	0.02	0.01	0.06	–	–
F–Ar0	0.07	11.82	0.22	1.93	0.01	–	–	0.22	0.09
F–He0	0.06	11.78	0.30	1.93	0.01	–	–	0.24	0.04
M–Ar	0.13	9.00	0.20	1.94	0.01	–	–	0.30	0.04

[a] F–Ar20 and F–He20 are the same composition with F–Ar0 and F–He0, respectively.

(wt%); on the other hand, that for the ODS martensitic steel (M–Ar) is 0.13C–9Cr–0.2Ti–0.3Y_2O_3 (wt%). Excessive oxygen is defined as the oxygen content after subtraction of the oxygen coupled to Y_2O_3.

In the manufacturing process, ODS steels were fabricated in the following five ways:

1. Mechanical alloying (MA) in an Ar gas environment and recrystallization at 1423 K for 1.8 ks (F–Ar0).
2. MA in an Ar gas environment and cold rolled 20% after recrystallization at 1423 K for 1.8 ks (F–Ar20).
3. MA in a He gas environment and recrystallization at 1423 K for 1.8 ks (F–He0).
4. MA in a He gas environment and cold rolled 20% after recrystallization at 1423 K for 1.8 ks (F–He20).
5. MA in an Ar gas environment and normalization at 1323 K for 3.6 ks (M–Ar).

The specimens were sliced from the cladding tube and were mechanically thinned to about 0.2 mm thickness and punched to discs with a diameter of 3 mm. These discs were electro-polished with an electrolytic solution of $CH_3COOH:HClO_4 = 19:1$ to get thin foils for transmission electron microscopy.

These specimens were irradiated to 12 dpa in a high-voltage electron microscope (H-1300) operated at 1 MeV

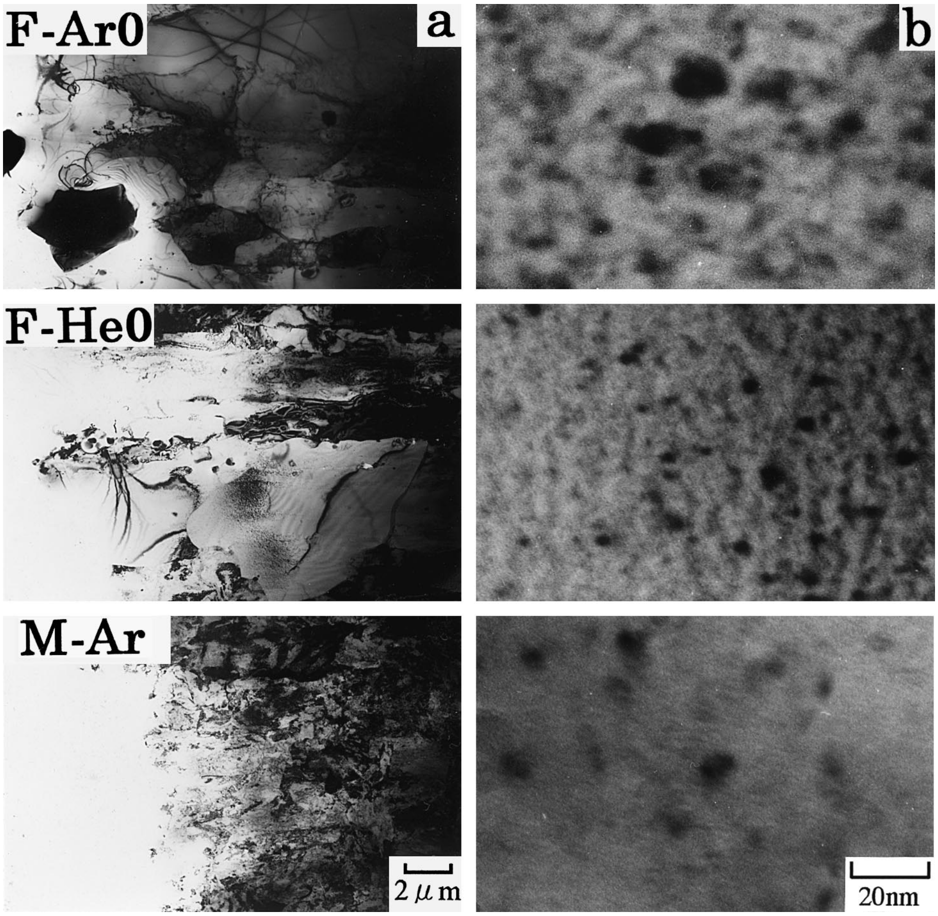

Fig. 1. Typical microstructure (a) and oxide particle (b) in F–Ar0, F–He0 and M–Ar before irradiation.

at 673–748 K. The damage rate was 2.0×10^{-3} dpa/s. The void swelling and the dislocation number density were measured in the micrographs.

3. Result and discussion

3.1. Typical microstructures before irradiation

Figs. 1(a) and (b) show typical images of dislocations and oxide particles in the alloys. F–Ar0 and F–He0 are composed of intermingled regions of recrystallized and unrecrystallized areas, while M–Ar has a typical lath martensite structure. There were obvious differences in the size of oxide particles (Fig. 1(b)) among F–Ar0, F–He0 and M–Ar; the particle sizes in F–He0 were lower than those in other alloys that were MA processed in an argon gas atmosphere. In the cold worked F–Ar20 and F–He20, it was found that the grains were elongated in the working direction and had a texture on {1 1 1} planes with ⟨1 1 0⟩ directions [4,6]. A dislocation cell structure was also observed in local areas (Fig. 2). F/M had a typical tempered martensite structure.

3.2. Void formation and swelling

Fig. 3 shows the development of the microstructure in F–Ar0, F–He0 and M–Ar during irradiation up to 12 dpa at 723 K. From the micrographs, it was found that voids in F–Ar0 had begun to nucleate at a low-dose level (<4.8 dpa), and grew continuously with increasing irradiation dose. Void formation in F–He0 was suppressed compared with that in F–Ar0. No voids were formed in M–Ar. The temperature dependence of void swelling is shown in Fig. 4. The peak temperature of the swelling seems to be about 723 K. Peak swelling levels at 12 dpa in F–Ar0 and F–He0 were about 1.3% and 0.5%, respectively. The swelling levels of F–Ar20 and

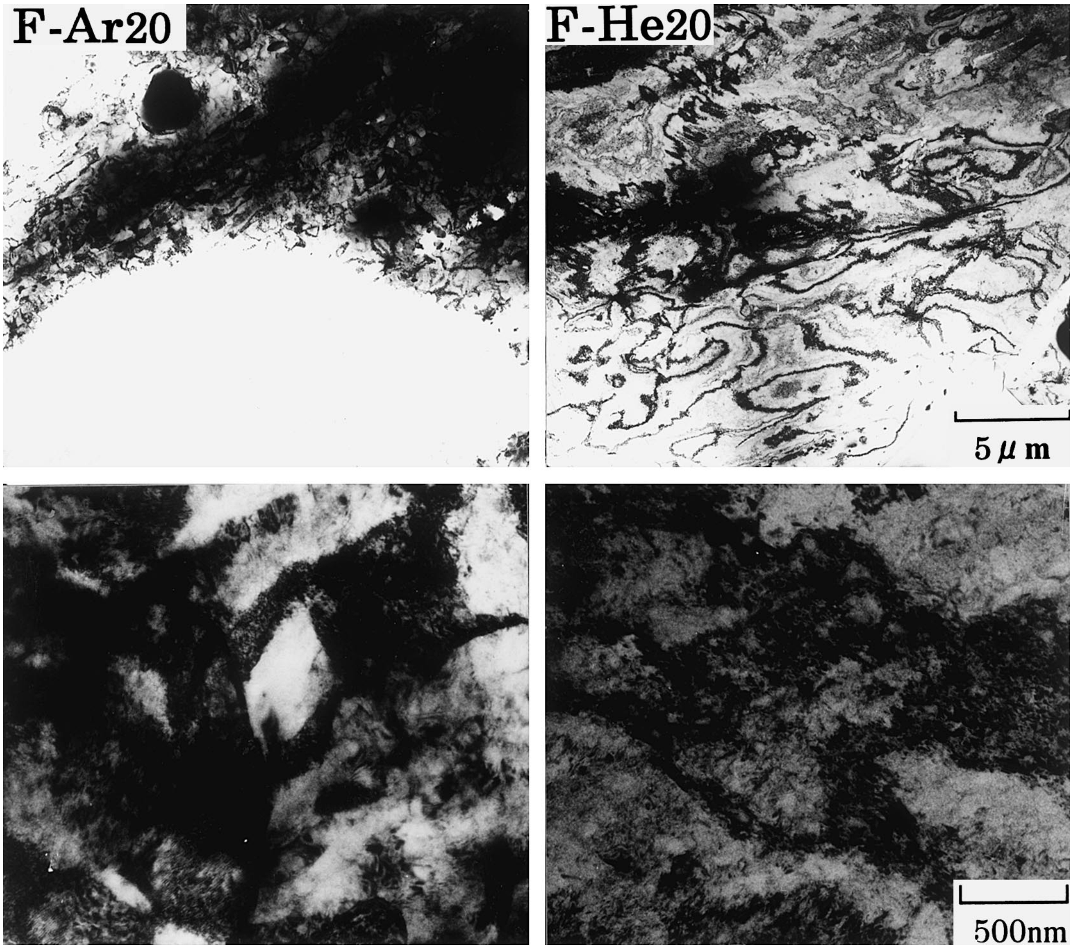

Fig. 2. Microstructure in F–Ar20 and F–He20 before irradiation.

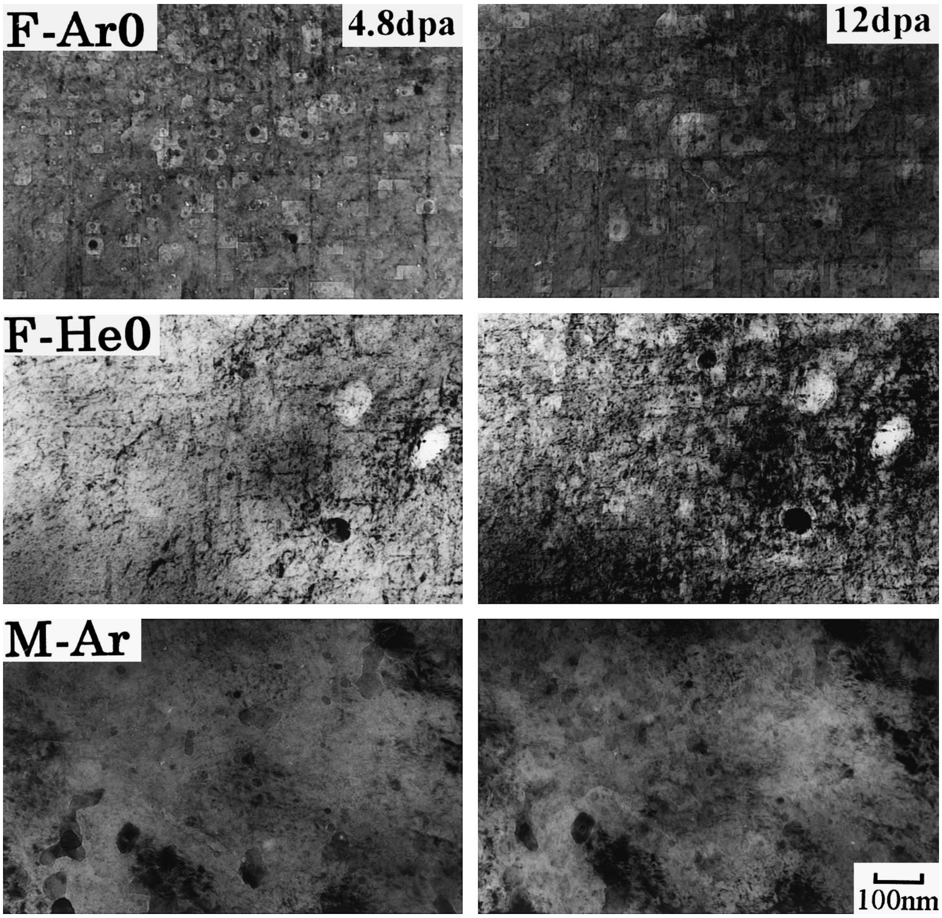

Fig. 3. Development of microstructure in F–Ar0, F–He0 and M–Ar after irradiation up to 12 dpa at 723 K.

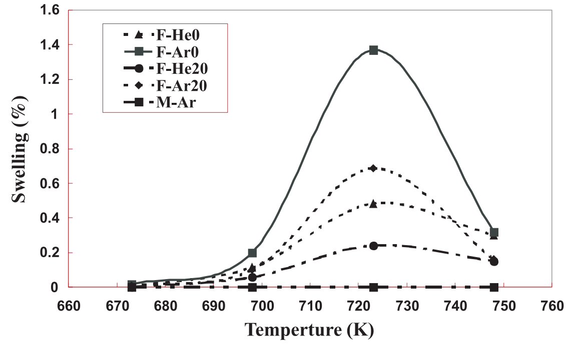

Fig. 4. Temperature dependence of void swelling in several types of specimens irradiated up to 12 dpa.

F–He20 were about half of those in F–Ar0 and F–He0, respectively. Except for F–Ar0, these swelling levels were lower than that of typical austenitic steels at the same conditions, in which the swelling levels are more than 1% [7].

3.3. Effect of oxide particle distribution

The primary purpose of the dispersed oxide particles is to improve the high-temperature strength. Results indicate that the oxide particles play another important role. Fig. 3 (F–Ar0) shows evidence that voids formed at the oxide particles, which indicated that the oxide particles had acted as point defect sinks and nucleation sites for defect clusters during electron-irradiation. The oxide particles might contribute to enhanced recombination to decrease point defect concentration in the matrix during electron-irradiation. This might cause the lower swelling in F–He0. It may be said that the oxide particles were stable, since there was no change in size and density of oxide particles before and after electron-irradiation.

3.4. Effects of MA parameters on swelling

Swelling resistance of the alloys is different, as shown in Fig. 4. In M–Ar, void formation was suppressed. It seems that the high-dislocation density was effective to suppress the void formation. On the other hand, recrystallization was not effective. Application of helium gas as MA environment and the cold working after recrystallization seem to be effective to reduce void swelling. As discussed in Section 3.3, swelling was also affected by the size and the density of the oxide particles.

4. Conclusion

Electron-irradiation was carried out on ODS ferritic steels and a martensitic steel, which were fabricated under different conditions:

1. Peak swelling temperatures of recrystallized ODS ferritic steels were about 723 K.
2. Cold working after recrystallization is effective in suppressing void swelling.
3. Application of helium gas as MA environment is also effective to reduce swelling.
4. ODS martensite exhibited the highest swelling resistance among the alloys examined.
5. An effective combination of MA parameters on swelling is the application of helium gas as MA environment and cold working after recrystallization.

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