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Journal of Nuclear Materials 258–263 (1998) 1264–1268

journal of nuclear materials

Void formation and microstructural development in oxide dispersion strengthened ferritic steels during electron-irradiation

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Abstract

ODS ferritic steels (13Cr–0.5Ti–0.2Y_2O_3) were prepared by the mechanical alloying method followed by the hot extrusion and several heat treatments including recrystallization. ODS steels with different heat treatment and a ferritic/martensitic (F/M) steel for the reference were irradiated to 12 dpa at 670–770 K in HVEM. After recrystallization, the dislocation density decreased with increasing grain size, however, the oxide particles did not show any obvious change in the size and the number density. During the electron-irradiation the microstructure was relatively stable, i.e. oxide particles showed good stability and the dislocation density remained almost constant. A limited void formation was observed in the specimens, and the suppressive effect due to dislocations with high number density was confirmed. From these results, the behavior of microstructure and the limited void formation in ODS steels have been discussed.

1. Introduction

Ferritic steels have good swelling resistance but have less creep strength at high temperature. To improve the high temperature properties, the oxide dispersion strengthened (ODS) method has been employed. ODS ferritic steels have more swelling resistance than austenitic steels and have excellent creep strength at high temperature due to the finely distributed stable oxide particles. Because of these properties, ODS ferritic steels are expected to be candidate materials for the first wall component in fusion reactors and as cladding materials in fast breeder reactors [1–3].

On the contrary, the ODS steel has a disadvantage in the microstructure from the fabrication process: the strong anisotropy of manufactured thin-walled cladding tubes is revealed, which causes degraded creep rupture strength and less ductility in transverse hoop direction perpendicular to the elongated grain structure due to the hot-extruding. In order to improve this strength anistropy, several ways have been tried, such as recrystallization [1–3].

In this study we performed electron-irradiation on ODS ferritic steels to clarify the effect of microstructural change, dislocations and oxide particles, after recrystallization on the void formation. Especially, changing of dislocations and oxide particles are important points we observed.

2. Experimental procedure

2.1. Materials

In this experiment, four types of specimens were used; the ferritic/martensitic steel (F/M), the as-extruded ODS ferritic steel (ODSE), the recrystallized ODS ferritic steel (ODSR) and the cold-worked ODS ferritic

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0022-3115/98/$19.00
PII: S0022-3115(98)00140-8

Table 1
The chemical composition of F/M, as-extruded ODS, recrystallized ODS and cold-worked ODS steels (wt%)

	C	Cr	Ti	W	N	V	Nb	Y_2O_3	O
F/M	0.13	11.2	–	2.1	0.05	0.2	0.05	–	–
ODSE	0.045	12.87	0.52	2.81	0.0152	–	–	0.34	0.186
ODSR	0.027	13.27	0.42	2.88	0.0098	–	–	0.216	0.074
ODSCW	0.031	11.38	0.10	1.99	0.017	–	–	0.2	0.12

steel (ODSCW). F/M, ODSE and ODSR are cladding tubes in the shape and ODSCW is a sheet. The manufacturing of the ODS steels involved the following process: The pre-alloyed powder was produced by argon gas atomization process. The ferritic powder and the yttrium oxide powder were mechanically alloyed (MA) using a high energy attrition ball mill with steel balls. The duration of processing was 173 ks at an agitator speed of 220 rpm in argon gas atmosphere. During this MA process, the metal powders might have been trapped between the grinding balls and then deformed, fractured, and cold welded. The MA powders were sealed in steel cans after degassing at 673 K in 10^{-4} torr and then hot-extruded at 1423 K. After the MA process and hot-extrusion, the rods were hot-worked (ODSE), and then were annealed at 1473 K for 3600 s for recrystallization after cold working (ODSR). Some of these steels were 40% cold-worked after recrystallization (ODSCW). The chemical composition of ODS steels and conventional F/M for reference are shown in Table 1.

2.2. Electron irradiation

To make TEM discs the specimens were mechanically thinned to 0.1 mm thickness, punched to discs with a diameter of 3 mm and electropolished at 50 V, 291–293 K with electrolytic solution of CH_3COOH: $HClO_4 = 19:1$. Electron irradiation was performed in a HVEM (H-1300) operated at 1000 keV to 12 dpa at 673–773 K. The damage rate was 2.2×10^{-3} dpa/s. The void swelling was measured in the micrographs by using a conventional digitizing method.

3. Results and discussion

3.1. Microstructures before irradiation

Fig. 1 shows typical microstructures in F/M and ODS steels which were as-extruded and recrystallized, respectively. F/M revealed lath structure with high dislocation density. The microstructure of the ODSE has high dislocation density introduced during MA and has homogeneously distributed dispersoid particles. The grains are drawn along the hot extrusion direction and have strong texture of {1 1 1} ⟨1 1 0⟩ [4]. On the contrary, in the microstructure of ODSR: a decreased dislocation density and an increased grain size were observed. It would appear that the strength anisotropy was improved by the recrystallization. Fig. 2 shows the

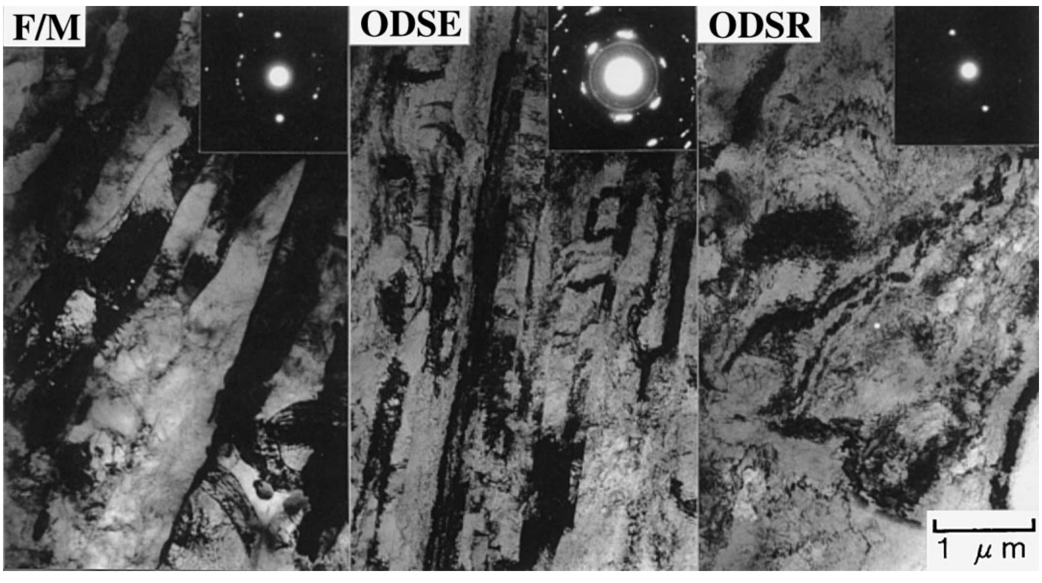

Fig. 1. Typical microstructures in ferritic/martensitic steel, as-extruded ODS steel and recrystallized ODS steel.

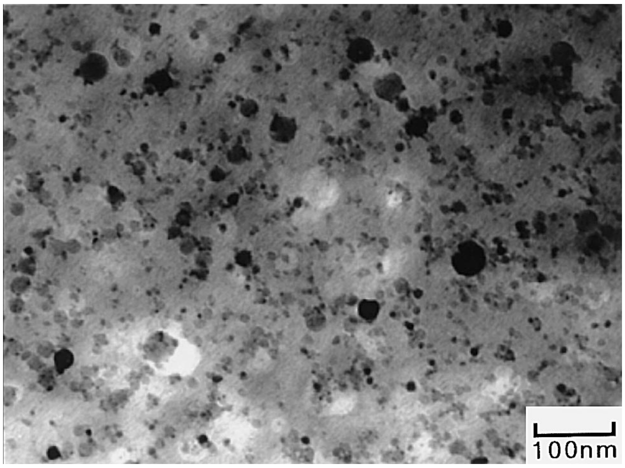

Fig. 2. Oxide particles in recrystallized ODS ferritic steel.

oxide particles after recrystallization. The size of distributed dispersoid particles was 4 nm in average. After the recrystallization the oxide particles did not show any obvious change in size. It indicates that the ODS particles are stable after the recrystallization.

3.2. Microstructures after irradiation

During electron irradiation, the increase of dislocation density and the formation of voids are confirmed in all specimens. A few voids are formed in F/M. In ODSE, void formation was rarely found. However, void nucleation and growth were obivous in ODSR during irradiation.

Fig. 3 shows the development of the microstructure in ODSR after irradiation up to 12 dpa at 698 K. The swelling was about 0.8% after irradiation to 12 dpa. In this case, the increasing of dislocation density was observed, but a change of the oxide particles could not be seen clearly because of the high density of dislocations.

3.3. Void number density

Fig. 4 shows the void number density as a function of the irradiation dose for each specimen. In the case of

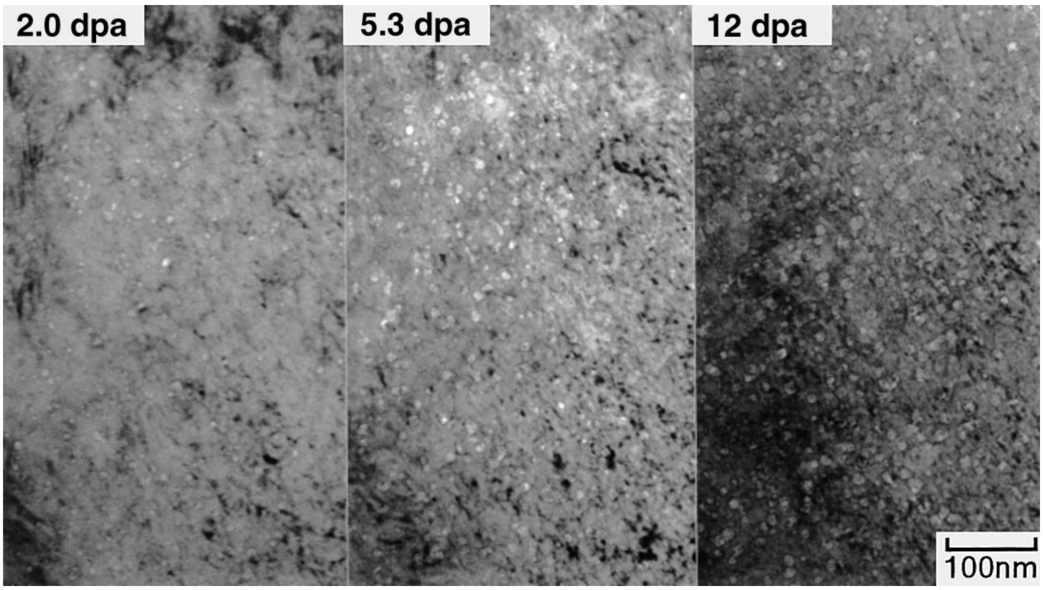

Fig. 3. Microstructural development during irradiation at 698 K in recrystallized ODS ferritic steel.

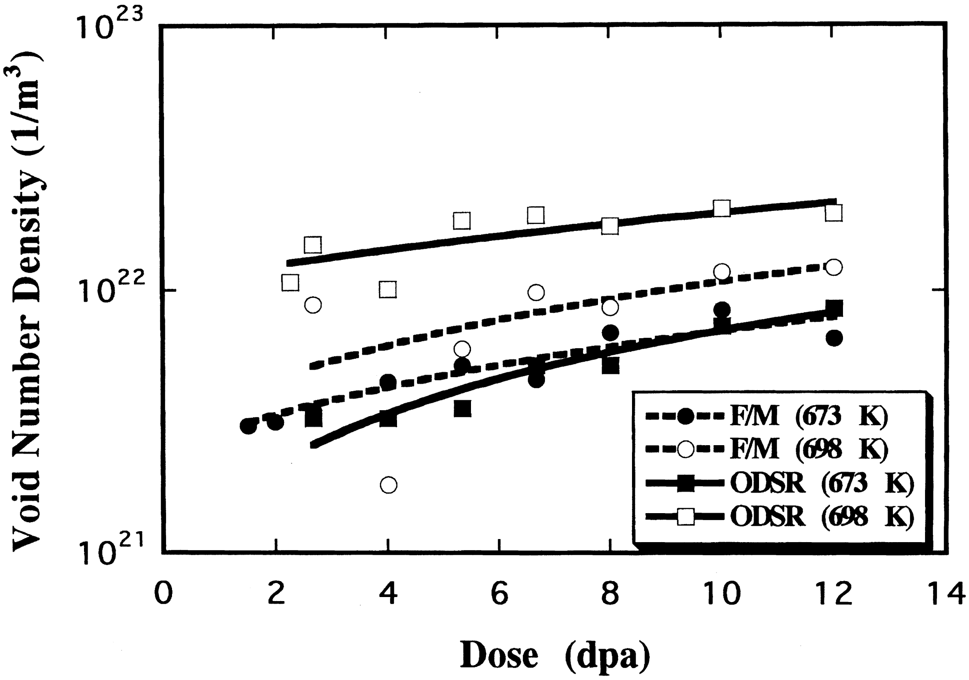

Fig. 4. Dose dependence of void number density up to 12 dpa.

typical austenitic steels, obvious void nucleation occurs only at the beginning of the electron irradiation, and the void number density sometimes decreases at high doses. This means that the concentration of point defects should be rapidly saturated in austenitic steels. In the case of F/M and both of ODS ferritic steels, the void number density increased with continuously increasing the irradiation dose, as shown in Fig. 4. This means that during the irraditation both void nucleation and growth occur at the same time. The microstructures of F/M and ODS steels are very complex and can act as sink sites for point defects. This suggests that a lot of time should be needed for the accumulation of point defects. In this case, the void formation occurs gradually at the sites where voids can be easily formed. Such nucleation sites could be the ODS dispersoids and gas bubbles.

3.4. Void swelling

The void swelling increased with increasing irradiation dose for each steel. Void swelling in ODSR irradiated at 698 K is remarkably high, however, other temperatures and specimens showed very low swelling levels.

Fig. 5 shows the temperature dependence of void swelling after the irradiation up to 12 dpa. The detectable swelling occurred between 673 and 723 K, and the peak swelling temperature was about 690 K. Though the swelling level reaches up to 0.8% in the ODSR, this level is lower than that of typical austenitic steels, in which swelling levels are more than 1% [5]. The swelling resistance of ferritic steels could be explained by several possible mechanisms; a high interactive recombination rate of point defects, strong trapping of point defects at solute atoms, and others [6,7]. Moreover, the role of cold working should be noted for reducing the swelling as shown in the data for ODSR and ODSCW.

3.5. Swelling resistance in ODS ferritic steels

In the ODSR, swelling is higher than that of ODSE and F/M (Fig. 5), which can be considered to be due to differences in their dislocation density. Lath martensitic structures and MA structures have dislocations with high density. The nucleation of both interstitial dislocation loops and voids is inhibited as a consequence of the dominance of point defect recombination at the dislocation sinks and then void swelling is suppressed [6,7]. On this point, introducing extra dislocations due to cold working can suppress the swelling, as shown in Fig. 5. Another reason of higher swelling in ODSR is considered to be due to the gas bubbles in the microstructure. The bubbles are attributed to be Ar gas contamination during the MA process and formed during the recrystallization process. During irradiation, void nucleation and growth probably occur at the gas bubble sites.

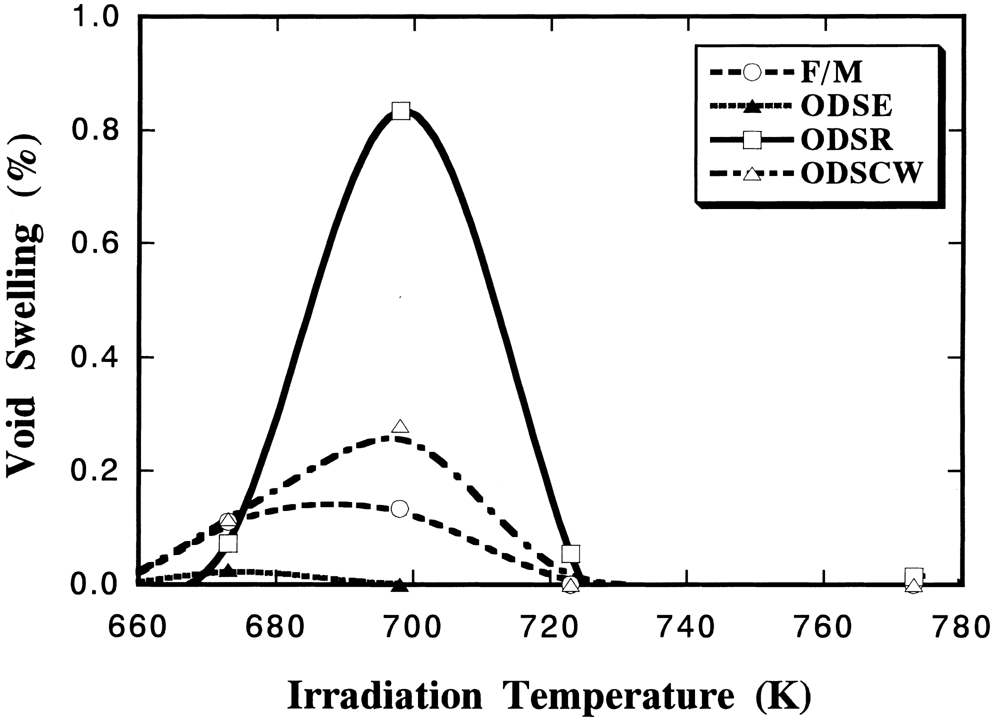

Fig. 5. Temperature dependence of void swelling after the irradiation to 12 dpa.

4. Summary

The electron-irradiation was performed on the several types of ODS ferritic steels and ferritic/martensitic steel, and the effect of heat-treatment and cold working on the microstructural evolution during the irradiation were investigated. The following conclusions are derived from the results and discussion.

1. In ODS ferritic steels and ferritic/martensitic steel, swelling levels were lower than conventional austenitic steels.
2. Swelling was increased in the ODS ferritic steel after recrystallization, however the swelling was reduced to a low level by the additional cold working.
3. The void formation of ODS ferritic steels strongly depends on the dislocation density.

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