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Radiation response of oxide-dispersion-strengthened alloy MA956 after self-ion irradiation

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ABSTRACT

We studied the radiation-induced microstructural evolution of an oxide-dispersion-strengthened (ODS) ferritic alloy, MA956, to 180 dpa using 3.5 MeV Fe^{2+} ions. Post-irradiation examination showed that voids formed rather early and almost exclusively at the particle-matrix interfaces. Surprisingly, voids formed even in the injected interstitial zone. Comparisons with studies on other ODS alloys with smaller and largely coherent dispersoids irradiated at similar conditions revealed that the larger and not completely coherent oxide particles in MA956 serve as defect collectors which promote nucleation of voids at their interface. The interface configuration, which is related to particle type, crystal structure and size, is one of the important factors determining the defect-sink properties of particle-matrix interfaces.

1. Introduction

Oxide-dispersion-strengthened (ODS) ferritic-martensitic steels are being considered as candidate materials for advanced nuclear reactors primarily because of their superior high temperature creep resistance and improved strength and radiation tolerance compared with traditional ferritic-martensitic steels [1–4]. Recent studies have shown that oxide particles embedded in the matrix may also benefit the overall radiation tolerance of the alloy by providing additional defect-trapping sites for both point defects and helium at particle-matrix interfaces, thereby reducing void swelling [3–8]. However, there are very limited results showing direct evidence of oxide particle-defect interactions. Most ODS alloys examined to date are nano-structured and therefore contain a high density of grain boundaries (GBs) that strongly interact with the radiation-induced defects [3,8–11], thus camouflaging the role played by the oxide particles in interacting with point defects.

In this study, we used the ferritic ODS alloy MA956 with rather large grains and a low density of GBs to reduce the GB contribution to total defect annihilation [9–11]. We used self-ion irradiation, an increasingly well-understood technique that has been applied to nuclear material research for decades [12–16]. We investigated the radiation response of this alloy to acquire evidence of particle-defect interactions and to illuminate the mechanisms involved.

2. Experimental procedures

MA956 was received in final form from Special Metals Corporation with composition given in Table 1. It was sectioned into 3 mm disks that were successively ground with 600, 800 and 1200 grade grit and then finally polished with 50 nm colloidal silica and alumina solution. The disks were then irradiated at 450 °C with 3.5 MeV Fe^{2+} ions to 60, 120, and 180 peak displacements per atom (dpa), using a rastered beam at 15.63 and 0.156 Hz in X and Y directions, respectively. The peak dpa rate was in the order of 10^{-3} dpa/s. No gas injection was employed in this study. The profiles for atomic displacement and injected Fe distributions for pure Fe were employed, calculated using the SRIM code [17]. These calculations assumed a displacement energy of 40 eV and used the Kinchin-Pease option to provide a damage estimate comparable to that of neutron irradiation [18,19].

TEM samples were prepared using a FEI Quanta 3D FEG dual beam secondary-electron-microscope (SEM)/focused-ion-beam (FIB) system. Cross-sectional samples were lifted-out from the irradiated surfaces, revealing microstructural changes through the ion range and into the unirradiated region. A FEI TECNAI F20 SUPER-TWIN transmission electron microscope (TEM) with energy dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS) applications was used. The microstructural

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Table 1
Nominal composition of MA956 ODS from Special Metals Corporation (at.%).

Fe	Cr	C	Si	Mn	Ni	Al	Cu	P	S	Ti	Y_2O_3
Bal.	19.4	0.02	0.04	0.1	0.05	4.8	0.02	0.01	0.008	0.38	0.51

development was observed using conventional bright-field TEM. EDS and EELS were used to characterize the microchemistry and thickness of the samples, respectively. The thickness of the TEM samples is typically about 100 nm.

The sizes of grains in this MA956 variant are on the order of millimeters, much larger than the typical FIB size of ~10 × 10 μm². Multiple samples were lifted-out and showed that the variance in microstructure between different grains is minimal. As shown in both previous and current studies, TEM investigations on FIB samples of ferritic-martensitic alloys provide good statistics to demonstrate effects of radiation on microstructural evolution, especially for void formation and growth, as well as changes to nano-particles [3,20–22].

3. Results and discussion

Fig. 1(a) shows the microstructure of MA956 before irradiation. The dark circular features are oxide particles appearing in different contrasts that we attribute to differences in particle-matrix orientations. Most of the nano-particles were found to have a diameter less than 60 nm, with a mean diameter of ~18 nm. EDS investigations show that the particles are Y-Al-O rich. Some particles larger than 200 nm exist, with compositions typical of either aluminum oxide or titanium carbide. The density of all nano-particles is $2.7 \pm 0.3 \times 10^{15}\ cm^{-3}$.

Fig. 1(b-d) show the microstructures of the self-ion-irradiated samples with peak damage levels of 60, 120 and 180 dpa, respectively, with the ions entering from the left. The most significant change induced by irradiation are voids growing exclusively on the particle-matrix interface and possibly growing into both the matrix and the oxide particles. No voids appear to be free of particles. A similar linkage between voids and precipitates in austenitic steels was labeled by Mansur et al. as the "precipitate collector" effect [23–25].

The sizes and densities of voids appear to increase with dose. In Fig. 1(d), large voids formed near a depth of 1000 nm where the dpa reaches its maximum as shown by the superimposed damage profile represented by the solid line. The dashed line in Fig. 1(d) represents the distribution of injected interstitials known to suppress void formation through the defect imbalance effect [12,14]. Unlike most ferritic alloys where voids are totally absent in the injected interstitial zone, Fig. 1(d) shows there are voids observed between 1000 and 1200 nm where the density of injected interstitials is high, but they are generally smaller compared to those formed between 800 and 1000 nm, where the density of injected interstitials is relatively low. The irradiation also led to an increased dislocation density in the matrix, as seen in Fig. 1(b-d) and the insert in Fig. 1(c). The oxide particles in the present study are not effective in enhancing interstitial-vacancy recombination.

Fig. 2(a) and (b) present void swelling and void densities as a function of depth for the irradiated samples, showing that both the volume fractions of the voids and the position of peak void swelling increase with dpa. This phenomenon has been observed in other ODS and non-ODS ferritic-martensitic alloys when irradiated by self-ions [3,10–12,21,22]. The defect imbalance effect and the "internal temperature shift" due to the relatively large variation in the dpa rate over the ion range have been invoked to explain the shape of these distributions [14,26]. Void swelling from the depth interval of 600–1000 nm was used to produce Fig. 2(c), a

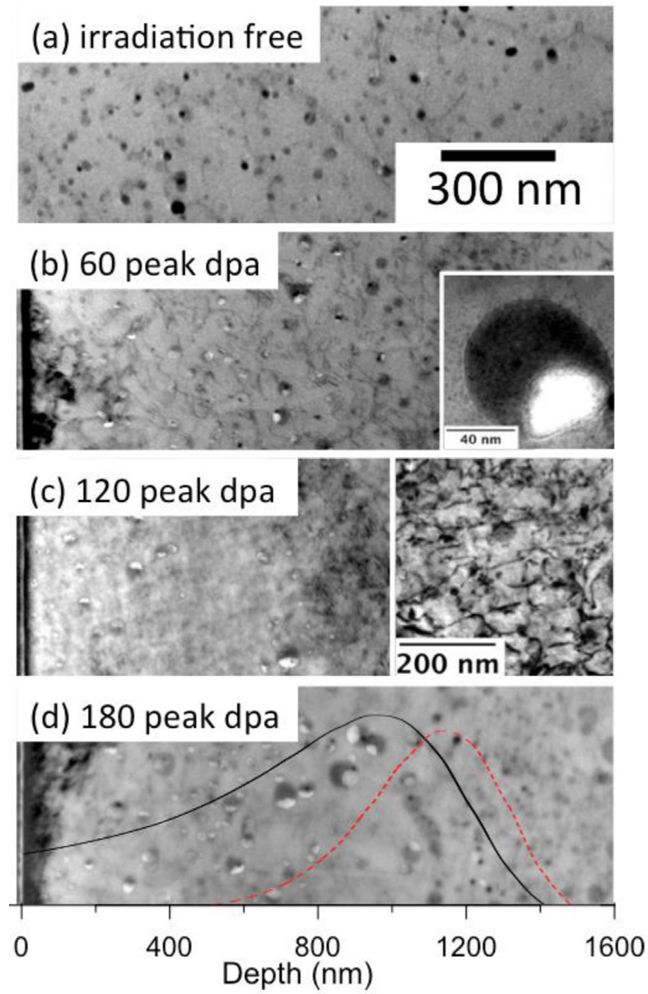

Fig. 1. Conventional bright field images of (a) the unirradiated and (b-d) irradiated specimens with 60, 120 and 180 peak dpa, respectively, showing that essentially all voids are associated with oxide particles. The insert in (b) shows a void formed on the side of an oxide particle in the 60 peak dpa sample. Note that the insert also shows a cloud of small precipitates surrounding the particle that was probably formed by recoil dissolution and reprecipitation. The insert in (c) shows the irradiation-induced dislocation network at the depth of 1000 nm in the 120 peak dpa sample. The lines superimposed on (d) are the calculated dpa and injected interstitial profiles as a function of depth.

plot of void swelling as a function of the average dpa level in that interval. Fig. 2(b) shows that specimens at 120 and 180 peak dpa have higher void densities than the 60 peak dpa sample, indicating nucleation of new voids with increasing damage. On the other hand, from 120 to 180 peak dpa, the changes in void density are not as significant, suggesting that from 120 to 180 peak dpa, the growth of existing voids plays the most important role in increasing swelling.

Fig. 2(d) shows the density- and diameter-depth profiles of oxide particles for the 180 peak dpa sample, as solid and empty circles, respectively. The density of oxide particles is slightly reduced in the irradiated region, as compared with that beyond the project range of the iron ions, illustrating the instability of oxide particles

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