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Temperature dependent dispersoid stability in ion-irradiated ferritic-martensitic dual-phase oxide-dispersion-strengthened alloy: Coherent interfaces vs. incoherent interfaces

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ABSTRACT

In this study, the microstructure of a 12Cr ferritic-martensitic oxide-dispersion-strengthened (ODS) alloy is studied before and after Fe ion irradiation up to 200 peak displacements per atom (dpa). Irradiation temperature ranges from 325 to 625 °C. Before irradiation, both coherent and incoherent dispersoids exist in the matrix. In response to irradiation, the mean sizes of dispersoids in both the ferrite and tempered martensite phases change to equilibrium values that increase with irradiation temperature. The evolution of dispersoids under irradiation is explained by a competition between athermal-radiation-driven shrinkage and thermal-diffusion-driven growth, with interface coherency affecting the growth mechanism. However, each coherency type exhibits different evolution behavior under irradiation. Coherent dispersoids, regardless of their initial size, change toward an equilibrium size at each temperature tested. On the other hand, incoherent dispersoids are destroyed at lower test temperatures but survive while shrinking in size at higher temperatures. This difference in behavior can be explained by the lower interfacial energy of coherent dispersoids in comparison with incoherent dispersoids. This study sheds light on the roles of interface configurations in maintaining dispersoid integrity under irradiation.

1. Introduction

Ferritic/martensitic (F/M) steels are being considered as candidates for cladding materials in fast reactors. F/M steels are preferred rather than austenitic steels not only because of their lower thermal expansion and higher thermal conductivity, but also because they are known to have overall higher radiation tolerance to some forms of radiation damage [1–7]. In particular, the swelling resistance of F/M steels is much better in general than austenitic steels, having longer incubation regimes for void nucleation, and a steady-state swelling rate of approximately one-fifth that of austenitic steels [6,8].

Swelling resistance of both austenitic and ferritic alloys is known to be further enhanced by introducing a high density of stable defect sinks, such as grain/sub-grain boundaries and precipitate-matrix interfaces [6–13]. Among F/M alloys, oxide-dispersion-strengthened (ODS) variants are also attractive for nuclear applications due to their strong contribution to creep resistance, especially at higher temperatures where ferritic alloys are much weaker than austenitic alloys [5,14,15]. The dispersoids pin and stabilize the grain boundaries against recrystallization and grain growth and may act as sinks for both point defects and transmutant helium atoms [13–18]. Trapping of defects and helium at the dispersoid/matrix interface was evidenced by comparing irradiation-induced microstructure changes of ODS and non-ODS alloys [18]. Such a trapping effect was believed to suppress helium bubble growth and enhance the resistance against helium embrittlement of the material [15,16,18].

Ukai et al. have developed a series of ODS alloys combining mesoscopic optimization of dual phase ferrite/tempered

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martensite with nano-scale optimization of ultra-fine oxide particles to strongly enhance high temperature creep resistance and void swelling resistance [19–22]. Among these alloys, a 12Cr ODS alloy was fabricated to have a very stable dual-phase microstructure by increasing the nickel content which is traditionally low in most F/M alloys [13,22]. This alloy, when compared to other 9Cr ODS alloys, also has greatly improved resistance to high temperature oxidation and corrosion. The combined roles of the tempered martensite phase at a large volume percentage and the fine-sized dispersoids in both phases make this alloy a promising material for nuclear applications, especially in terms of swelling and creep resistance [7,13].

The maintenance of the structural integrity of nano-sized dispersoids during irradiation is very critical to maintain the strength of an ODS alloy, especially at high temperatures. First, dispersoids stabilize the grain/sub-grain structures; second, they pin dislocations and restrain thermal creep; and third, they have been proposed to serve as defect-trapping sites, all of which may work to reduce void swelling. Thus, it is of great importance to understand the evolution of nano-sized dispersoids under various irradiation conditions. Previous studies on various ODS alloys under neutron and ion irradiation have observed a range of behaviors, showing that dispersoids can shrink and/or dissolve [13,20,23,24], coarsen and/or grow [25–27], undergo amorphization [28–30] or be preserved with essentially no changes [31–33]. An excellent review on current understanding of radiation effects on ODS alloys can be found in Refs. [17,20 and 34].

However, there is still a lack of knowledge on all factors governing dispersoid integrity during irradiation, especially concerning the wide range of dispersoid phases and interface configurations that combine to determine dispersoid stability. In the present study, we select the 12Cr dual-phase ODS due to the presence of distinct dispersoid distributions in both the ferrite and tempered martensite phases. Our particular interest is to compare not only the dispersoid stability under irradiation in both phases, but also the changes in their stability as a function of irradiation temperature, a factor we will show to be related to the dispersoid-matrix interface configuration.

2. Experimental procedures

The 12Cr ODS dual-phase alloy was supplied by Professor S. Ukai of Hokkaido University, Japan [22]. The sample had been normalized at 1050 °C for 60 min and tempered at 800 °C for 60 min. The resultant two phases, tempered martensite and ferrite, have a ratio of ~4:1 [13], a ratio which is controlled by the nickel content [22]. Y-Ti-O dispersoids were previously observed in both phases, but with distinctly different distributions [13]. The chemical composition of this alloy can be found in Table 1.

Irradiation was carried out using a 1.7 MV Ionex Tandetron accelerator located at Texas A&M University. Three temperatures, 325, 475 and 625 °C, were chosen for irradiation. The temperature was monitored throughout the irradiation using a thermocouple attached to the hot stage surface. Temperature fluctuation during irradiation was less than 5 °C. At each irradiation temperature, samples were irradiated with 3.5 MeV Fe ions to 100 and 200 peak dpa. A double charged Fe beam of 200 nA was defocused to irradiate a 6 × 6 mm area on the stage. Use of a defocused beam has been recommended as a better surrogate technique to simulate steady-state neutron damage than use of a rastered beams [35–37].

Fig. 1 shows the displacement and injected Fe distribution profiles caused by 3.5 MeV Fe ion irradiation in pure Fe, calculated using SRIM [38]. The displacement threshold energy for the Fe matrix was set to 40 eV; and the Kinchin-Pease option was used due to recent findings of overestimation of dpa using the full damage cascade mode [39,40]. The per-ion dpa profile peaks at a depth of ~1000 nm. The injected Fe distribution peaks at ~1200 nm.

Prior to irradiation, specimens were cut into 5 × 5 × 0.7 mm plates and polished mechanically. The surface damage layer resulting from mechanical polishing was removed using electro-polishing with a perchloric acid solution. In order to reveal the microstructure in the whole irradiation projected region as well as irradiation-free region, we prepared TEM specimens using focused-ion-beam (FIB) technique with a Tescan LYRA-3. TEM specimens ~10 μm wide by ~7 μm deep were lifted out and thinned to ~200 nm using 30 keV Ga beam. Next, a 5 keV Ga beam was used to polish the sample to a thickness of ~100 nm. To complete preparation, a 2 keV Ga beam was used to remove damage caused by higher energy Ga bombardment. Microstructure characterization was performed using a 200 kV Technai F20 Supertwin transmission electron microscope (TEM) and a JOEL 2100F 200 kV TEM. Bright field (BF) and weak beam dark field (WBDF) imaging techniques were used to show nano-dispersoid distributions. Energy dispersive X-ray spectroscopy (EDX) and energy-filtered transmission electron microscopy (EFTEM) were used for chemical composition studies. High resolution transmission electron microscopy (HRTEM) was used to reveal some details of the microstructure.

3. Results

As shown in Fig. 2a, the two phases of ferrite and tempered martensite have different grain sizes. The tempered martensite

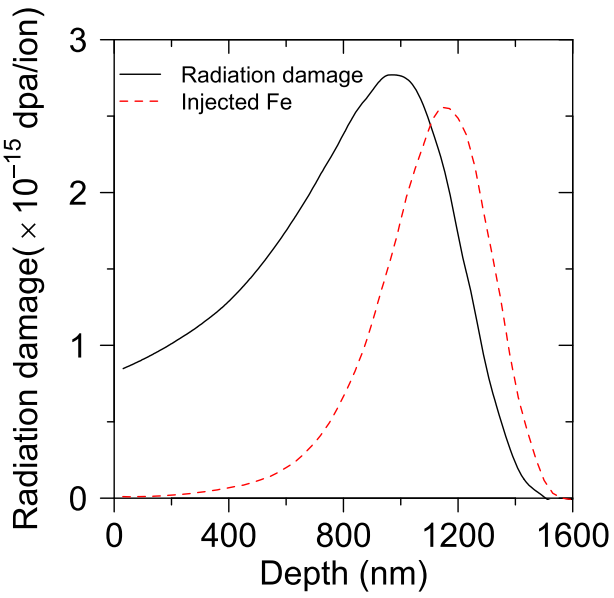

Fig. 1. SRIM calculation of 3.5 MeV self-ion irradiation of pure Fe.

Table 1
Chemical composition of the 12Cr ODS alloy investigated in this study, with Ex. O standing for excess oxygen.

Component:	Fe	C	Cr	Ni	W	Ti	N	Ar	Y_2O_3	Ex. O
Weight, %:	Bal.	0.16	11.52	0.34	1.44	0.28	0.007	0.006	0.36	0.144