Stability of Nanoparticles in an Oxide Dispersion Strengthened Alloy

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Abstract. Oxide Dispersion-Strengthened (ODS) reduced-activation ferritic/martensitic steels are strong candidates for structural applications in the first wall and blanket of future fusion reactors. In the present work, a model ODS alloy based on a Fe-Cr matrix has been produced in order to gain insight into the processes which occur during processing and after high-energy ion irradiation. The model ODS alloy has been characterized by Energy Dispersive X-Ray and Electron Energy Loss Spectroscopy and Energy Filtered Transmission Electron Microscopy, focusing on the evolution of the nanoparticle dispersion and the chemical composition of individual nanoparticles. The chemical analyses reveal that there are three different kinds of nanoparticles in the alloys. Particle sizes, distributions and morphologies are not changed significantly by irradiation up to doses of ~ 7 dpa.

1. Introduction

Oxide dispersion-strengthened reduced-activation ferritic/martensitic steels (ODS-RAFMS) are being considered as structural materials for the first wall and blanket of future fusion reactors. An homogeneous dispersion of hard oxide nanoparticles in the steel could block the motion of dislocations and allow an increase in their maximum service temperature of more than 100°C compared with conventional RAFMS [1]. It has been demonstrated that a dispersion of ~ 0.3wt% of Y2O3 particles results in better tensile and creep properties than those of the base material [2]. A major concern with these materials has been their poor impact behaviour, but this could be improved by applying suitable thermo-mechanical treatments [3]. The nanoparticles could also help to trap helium atoms and point defects, which would reduce the swelling and hardening effects which cause the deterioration of the steels under irradiation [4]. ODS steels are normally produced by pulvimetallurgical routes that include mechanical alloying (MA) of the initial powders and consolidation by hot isostatic pressing (HIP) or hot extrusion. Recent studies have shown that the chemistry of the oxide nanoparticles in ODS steels can evolve with respect to the starting powders [5, 6] and their crystallographic phase may also change. For example, the milling conditions may lead to dissolution of the initial particles. Re-precipitation of particles with different crystallography or chemical composition than the starting particles could significantly affect their dissolution behaviour under irradiation and the mechanical performance of the material.

In order to gain insight into these processes, we focus here on the study of a simplified Fe12%Cr ODS alloy. This work reports the evolution of the nanoparticle dispersion after ion irradiation at 500°C up to doses of ~ 7 dpa.
2. Experimental methods
The model ODS alloy had a nominal composition Fe-12Cr-0.4 Y₂O₃ (wt%). The Y₂O₃ starting powders were spherical and had sizes ranging from ~9 to 100 nm. The lattice parameters obtained from X-ray diffraction of the powders were consistent with a monoclinic cell [7]. The alloy was produced by MA followed by HIP as described in [8]. After consolidation, the alloy was tempered at 1023 K for 4h. Disks of diameter 3 mm and thickness ~60 μm were then cut and prepared for irradiation by flash electropolishing for ~1 s to remove possible superficial damage produced during the grinding process. The disks were irradiated at 500°C to doses of 2.5 and 7 dpa. Both 0.5 and 2 MeV Fe⁺ ions were used in order to obtain a flat damage profile within about 1 μm of the ion-entry surface. The irradiated disks were subsequently prepared for TEM by flash electropolishing for ~2 s to remove about ½ μm from the irradiated surface, followed by back thinning by jet electropolishing in a TENUPOL 5 twinjet polisher using 5 % HClO₄ + 95 % CH₃OH as electrolyte.

TEM characterisation of the particles before and after irradiation was carried out by high angle annular dark field (HAADF) scanning TEM (STEM), energy dispersive spectroscopy (XEDS) and electron energy loss spectroscopy (EELS) using a JEOL 3000F TEM operated at 300kV. Energy filtered TEM (EFTEM) maps were obtained with a JEOL 2200MCO prototype microscope equipped with two spherical aberration (Cs) correctors.

3. Results and discussion
EFTEM maps in figure 1 show the Y-rich nanoparticle dispersion before and after irradiation. Details of the extraction of Y maps can be found in [9].

![Figure 1. BF images and corresponding Y N₂,₃ EFTEM maps of the ODS/Fe12Cr showing the distribution of Y-rich ODS particles before (a), (d) and after irradiation at 2.5 dpa (b), (e) and 7dpa (c), (f).](image)

Particle sizes, distribution and densities were similar in the three conditions, suggesting that they had not undergone dissolution under irradiation. The particle size distribution varied from one region...
to another, with individual ODS particle sizes in the range 1-50 nm. The particles were not homogeneously distributed. Particle densities were calculated for different regions with average volumes of 250x250x100 nm³. The foil thickness was measured using the log-ratio technique where the Malis formula was used to estimate the inelastic mean free path [10]. Densities were found to vary between 0 and 1.1×10²³ m⁻³. The particles were frequently found within the grains, but they can also be aligned along grain boundaries as shown in Figure 1 (a) and (d).

The chemistry of the particles was studied before irradiation in order to analyze their evolution during processing and after irradiation, with the aim of looking for possible changes caused for example by irradiation-enhanced diffusion. Nanoparticles with different chemical compositions were found. Figure 2 (a) is a STEM-HAADF image showing nanoparticles in the non-irradiated alloy. The chemical compositions of the particles marked as A, B and C were analyzed by EELS and XEDS, see figure 2 (b). Particle B has a higher contrast than A and C. Particles A and B are attached, appearing as halves of a bigger particle. Contrast changes in HAADF images can be due to differences in atomic number, density or thickness. The linescans reveal that the change in contrast found for particle B is due to a difference in chemical composition, as shown in figure 2 (b). This particle contains Y, Cr and O, while particles A and C, with a lower contrast, contain Cr and O but no Y. The high O content found in the matrix is attributed to the formation of an oxide film on the sample. The increment in the O concentration in the matrix in the direction of the linescan is probably an artefact arising from a decrease in thickness, which is suggested by the decrease in the HAADF signal of the image. Thinner regions would have less Fe and Cr content, but the same surface oxide thickness, forcing the quantitative O signal to increase.

![Figure 2](image1.png)

Figure 2. (a) STEM-HAADF image of a region in the non-irradiated ODS alloy, and (b) EELS elemental profile of O, Fe and Cr and EDS elemental profile of Y across the line marked in (a).

Other nanoparticles present in the alloy consist of an Y-O rich core and a Cr-rich shell. This core-shell structure is shown in figure 3. The chemical compositions of the particles do not seem to change
after irradiation, at least up to a dose of 2.5 dpa, and the Cr shell can be still observed [9]. Analyses of the particle composition, especially the core-shell structure at 7 dpa, are ongoing.

![Figure 3. (a) Y N\textsubscript{2,3} EFTEM map showing Y-rich nanoparticles in the non-irradiated alloy and (b) Cr M\textsubscript{2,3} EFTEM map showing that a Cr shell forms around some of the particles.](image)

### 4. Conclusions
This work describes the nanoparticle dispersion present in an oxide dispersion-strengthened ODS/Fe\textsubscript{12wt%Cr} alloy and its evolution after Fe\textsuperscript{+} irradiation at 500°C at doses of 2.5 and 7 dpa. An inhomogeneous dispersion of nanoparticles with sizes < 50 nm was found. Particle sizes, distributions and morphologies appear unchanged up to an implantation dose of 7 dpa, suggesting that they have not undergone dissolution under irradiation. The chemical composition of the particles was studied by STEM-EELS-EDS and EFTEM. Some Y-rich particles consist of an Y-O rich core and a Cr rich shell, while others contain Y-Cr-O. Particles containing only Cr and O were also detected. The chemistry of the particles appears to be unaltered at least up to a dose of 2.5 dpa, confirming their stability under these irradiation conditions. Compositional analyses at 7 dpa are in progress.

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### Acknowledgments
The authors thank Prof. R. Pareja’s group at the Universidad Carlos III de Madrid for providing the ODS alloy. V. de Castro acknowledges the FP6 Euratom Research and Training Programme on Nuclear Energy for funding this research. S Lozano-Perez is grateful to INSS (Japan) for sponsoring his research.