In-situ $\text{Fe}^+$ Ion Irradiation of an Oxide Dispersion Strengthened Steel

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Abstract. Oxide Dispersion Strengthened (ODS) reduced activation ferritic steels are promising candidate materials for structural components of both nuclear fission and fusion reactors. However, when irradiated with energetic particles, they may suffer changes on their microstructures that degrade their mechanical performance. In-situ transmission electron microscopy studies on ion-irradiated ODS steels can give remarkable insights into fundamental aspects of radiation damage allowing dynamic observations of defect formation, mobilities, and interactions during irradiation. In this investigation, a commercially available PM2000 ODS steel was in-situ irradiated with 150KeV $\text{Fe}^+$ at room temperature and 700ºC. These experiments showed that the oxide nanoparticles in these steels remain stable up to the higher irradiation dose ($\sim$ 1.5 dpa), and that these particles seem to be effective sinks for irradiation induced defects.

1. Introduction

Oxide Dispersion Strengthened (ODS) ferritic steels are one of the best considered materials for structural components in advanced fission power plants and future fusion reactors [1]. In these steels, nanometer sized Y-rich oxide particles homogeneously dispersed in the matrix strengthen the alloy providing better mechanical and creep resistances which would allow higher operating temperatures to be used, resulting in enhanced efficiencies [2]. ODS ferritic and ferritic-martensitic steels also show a high resistance to void swelling and better thermal properties than austenitic steels [3]. It is well known that irradiation induces undesirable microstructural changes that may lead to severe degradation of mechanical properties. It is expected that ODS ferritic steels will present a lower damage accumulation rate than the base steels, due to the oxide particles acting as trapping sites for irradiation induced defects and transmutation gasses [4]. However, there has not been much systematic work to determine the effect of irradiation on ODS steels.

This investigation reports the microstructural characteristics of a commercially available PM2000 ODS ferritic steel in-situ irradiated with $\text{Fe}^+$ ions at room temperature (RT) and 700ºC up to calculated doses of $\sim$ 1.5 dpa. These $\text{Fe}^+$ ions simulate the damage produced by energetic neutrons.
2. Experimental methods

The PM2000 ODS steel used in this study was produced by Plansee Industries in form of bars. The nominal chemical composition given in the Plansee datasheets is Fe-19Cr-5.5Al-0.5Ti-0.5Y$_2$O$_3$ (wt%).

Before irradiation, disks of diameter 3 mm were cut perpendicular to the bar axis, thinned to a thickness of ∼60 µm to minimise magnetic effects within the transmission electron microscope (TEM) and finally JET electro-polished to get electron transparency. Electro-polishing was carried out in a TENUPOL 5 polishing unit using 5 % HClO$_4$ + 95 %CH$_3$OH as electrolyte, voltages of ∼20 V and temperatures below -30°C. The disks were in-situ irradiated with 150KeV Fe$^+$ ions at RT and 700°C at the intermediate voltage electron microscope (IVEM) facility available at Argonne National Laboratory (ANL) [5]. One of the advantages of using this facility compared to other in-situ irradiation facilities is that it allows the angle between the sample axis and the ion beam to be as low as 15°, also permitting continuous observation and data recording during irradiation. The ion energy (150KeV) was chosen to mimic high-energy fusion cascades and for comparison with previous work conducted by Jenkins and co-workers [6-8]. The maximum irradiation dose was 4x10$^4$ ions/cm$^2$. This fluence corresponds to a damage of ∼1.5 dpa in the region of the maximum damage (∼30 nm from the irradiated surface) as calculated with the SRIM program [9].

During the experiment, TEM characterization was carried out on a Hitachi 9000 NAR microscope operated at 300 kV. TEM studies before and after irradiation were carried out by bright-field (BF), dark-field weak beam (DF-WB) and energy dispersive spectroscopy (EDS) with a CM20 TEM operated at 200 kV. Energy filtered TEM (EFTEM) maps were acquired using a JEOL 3000F operated at 297 kV.

3. Results and discussion

Figures 1(a) to (d) show BF-TEM images of an area of the PM2000 steel irradiated to different damage levels at RT. This area was recorded during irradiation. Irradiation induced defects start to be visible at doses of ∼0.4 dpa, see figure 1 (b). They are revealed as small black dots which appear to be associated to ODS nanoparticles. No damage is observed in the matrix below ∼0.8 dpa (figure 1(c)). At this irradiation dose, the few defects present in the matrix have sizes < 5 nm. The defect density increases as the dose does, up to the maximum irradiation dose of ∼1.5 dpa (figure 1(d)).

Figure 1. Damage evolution at different doses at RT ((a) to (d)) and at 700°C ((e) to (h)).
Figures 1 (e) to (h) show the evolution of the PM2000 steel under irradiation at 700ºC. Irradiation induced defects could not be studied as surface degradation due to high temperature oxidation prevented their visualization at this temperature.

BF and DF-WB micrographs of two other areas irradiated to the maximum dose are presented in Figure 2. The area shown in figures 2 (a) and (b) contains a higher particle density while the particle density is lower in the area depicted in figures (c) and (d). In the high particle density region there are few defects present in the matrix; most defects are observed at particles. The number of defects present in the matrix is higher in the area containing a lower particle density. These results reveal that ODS particles act as sinks for irradiation induced defects, absorbing the vacancies or interstitials formed during the irradiation and subsequently expelling them as dislocations giving rise to the debris detected especially in the area containing a high particle density. This sequence of events was also observed during irradiation.

A previous investigation of as received PM2000 steel sheets produced by the same distributor revealed that the $Y_2O_3$ dispersoids added to the starting powder are not retained in the final product. They react with Al during high temperature pressing and form Y–Al–O ternary oxides with sizes < 40 nm [10]. In the present work, the ODS particle dispersion was analysed before and after irradiation using EFTEM to detect possible irradiation induced changes. The particle distribution before and after the RT irradiation is depicted in the elastic images, Y N$_{2,3}$ and O K EFTEM elemental maps presented in figure 3.

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![Figure 2. BF-TEM and WB-DF micrographs showing loop formation on ODS particles in PM2000 irradiated at RT. The grains are oriented along g (110).](image)

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![Figure 3. Elastic images, and Y N$_{2,3}$ and O K EFTEM maps of PM2000 showing the distribution of ODS particles before ((a) to (c)) and RT after irradiation ((d) to (f)) at 1.5 dpa.](image)

Figure 3. Elastic images, and Y N$_{2,3}$ and O K EFTEM maps of PM2000 showing the distribution of ODS particles before ((a) to (c)) and RT after irradiation ((d) to (f)) at 1.5 dpa.
The Al L elemental map was too noisy and is not presented here, but point EDS analyses reveal that the particles contain Al. EFTEM analyses did not reveal any differences in the particle morphology, sizes or chemical composition due to irradiation. Particles are nearly spherical and have (Y, Al, O) – rich compositions. The size distributions are similar before and after irradiation at both RT and 700ºC. Average particle sizes are 20 ± 10 nm and 22 ± 11 nm for the PM2000 steel before and after RT irradiation and 21 ± 10 nm after irradiation at 700ºC.

4. Conclusions
This work analyses the microstructure of a PM2000 steel in-situ irradiated at RT and 700ºC up to doses of ~1.5 dpa. The experiments showed that the ODS nanoparticles present in these steels are effective sinks for irradiation induced defects. The number of defects present in the matrix was lower in regions containing a higher particle density. Ion irradiation up to the higher irradiation dose had no measureable effect on the sizes of the ODS particles, caused no measureable change in particle composition, and moreover the particles kept their spherical shapes. ODS particles are therefore very stable in this material under these experimental conditions.

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