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A preliminary investigation of high dose ion irradiation response of a lanthana-bearing nanostructured ferritic steel processed via spark plasma sintering

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ABSTRACT

A nanostructured ferritic steel with nominal composition of Fe-14Cr-1Ti-0.3Mo-0.5La_2O_3 (wt.%) was irradiated with Fe^{+2} ions at 475 °C for 100, 200, 300 and 400 dpa. Grain coarsening was observed for the samples irradiated for 200–400 dpa resulting in an increase of the average grain size from 152 nm to 620 nm. Growth of submicron grains at higher radiation doses is due to decreased pinning effect imparted by Cr-O rich nanoparticles (NPs) that underwent coarsening via Ostwald ripening. Dislocation density consistently increased with increasing irradiation dose at 300 and 400 dpa. The mean radius of lanthanum-containing nanoclusters (NCs) decreased and their number density increased above 200 dpa, which is likely due to solutes ejection caused by ballistic dissolution and irradiation-enhanced diffusion. Chromium, titanium, oxygen and lanthanum content of nanoclusters irradiated at 200 dpa and higher got reduced by almost half the initial value. The reduction in size of the nanoclusters accompanied with their higher number density and higher dislocation density led to significant radiation hardening with increasing irradiation dose.

1. Introduction

In recent years, interest has been rekindled in oxide dispersion strengthened steels (ODS) for use in advanced reactor applications, which demand materials to serve under harsh environments such as high temperatures and radiation doses [1]. Classical ODS steels, such as MA957 (Fe–14Cr–1Ti–0.25Mo–0.25Y_2O_3) [2] and nanostructured ferritic steels (NFS) such as 14YWT (Fe–14Cr–0.4Ti–3W–0.25 Y_2O_3) [3], are typically produced by mechanical alloying (MA) of pre-alloyed or elemental powder mixtures and consolidated via hot extrusion or hot isostatic pressing (HIP).

Microstructural characteristics of these alloys are unique. The dispersion of oxide nanoparticles (NPs) and nanoclusters (NCs) within the ferritic matrix are intended to provide high temperature strength as well as radiation damage tolerance [4,5]. The NPs are the oxide particles with diameter larger than 5 nm whereas the NCs are clusters of complex oxide (Y-Ti-O) smaller than 5 nm [6]. According to the density functional theory (DFT) calculations by Barnard et al. [7], NCs with composition and structure of pyrochlore ($Y_2Ti_2O_7$) are more stable than clusters coherent with the Fe lattice. Sakasegawa et al. [8] demonstrated that non-stoichiometric clusters enriched in Y-Ti-O are higher in number density and smaller in diameter and thus have higher thermal stability. However, non-stoichiometric clusters will transform to $Y_2Ti_2O_7$ NPs after annealing at 1200 °C for an hour.

In nuclear applications, ODS materials are expected to undergo

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irradiation doses of more than 150 dpa at temperatures ranging from 400 to 700 °C for a prolonged period of time in Gen IV reactors [4,9]. It is critical to evaluate thermal and irradiation stability of the oxide nanoparticles, their composition and number density. In general, the irradiation stability of the oxide NCs in irradiated ODS alloys has been investigated up to 150 dpa at 400–700 °C, and conflicting results on NCs have been reported in several publications [10–15]. Allen et al. [10] reported that the average size of NCs decreases but the number density of NCs increases in 9Cr-ODS ferritic steel at 500–700 °C and up to 200 dpa. On the contrary, Lescoat et al. [9] reported a slight increase in the particle size and slight decrease in the density of the oxide NPs due to the Ostwald ripening mechanism in ODS steels irradiated with Fe ions at 500 °C up to 150 dpa.

In Fe-9Cr ODS, under ion irradiation dose of 3 dpa at 500 °C, the oxide NCs exhibited a reduction in size and number density of oxide particles with an increase in Y:Ti ratio [16]. Rogozhkin et al. [17] irradiated ODS Eurofer alloy (Fe-13.5 Cr-0.3 Ti-0.3Y_2O_3) at 300 °C for 2.4 dpa neutron irradiation and observed a decrease in the mean oxide particle size; and 2–3 times increase in number density of NCs using atom probe tomography (APT) studies. The reduction in size was attributed to irradiation-cascade-induced dissolution of larger oxide particles, and an increase in number density was likely due to nucleation of new oxide precipitates due to freely migrating irradiation-induced defects. The elemental composition of NCs remained unchanged suggesting that good irradiation resistance can be achieved by these NCs being homogeneously distributed in the matrix and serving as defect recombination sites.

London et al. [13] utilized X-ray diffraction (XRD) to study the stability of the oxide particles in 14Cr ODS self-ion irradiated up to 150 dpa at 500 and 700 °C. The study reported that the oxide NCs were substantially refined, amorphized or dissolved at high dpa levels, indicating a strong dependence of NC stability on temperature [13].

Certain et al. [11,18] studied NC stability in 14YWT and Fe-9Cr ODS alloy under proton (1–3 dpa at 400 °C), heavy ion (up to 100 dpa at −75 °C and 300–600 °C) and neutron irradiation (3 dpa and 500 °C) by APT. They observed a “dynamic stability” of NCs at high temperatures, where ejected solute atoms often diffuse back to the parent clusters, resulting in overall apparent stability of these NCs. A comprehensive review article on irradiation response of dispersed oxide particles in the bcc Fe-Cr based system has been recently published by Wharry et al. [19]. They reported ballistic dissolution, Ostwald ripening, irradiation-enhanced diffusion, and homogeneous nucleation as four mechanisms responsible for irradiation-induced changes in chemistry, size, and number density of NPs oxide phases. More than one mechanism (combination of Ostwald ripening and irradiation-enhanced diffusion for example) can be active simultaneously and control the evolution of NPs and NCs in these ODS steels [19].

In our previous studies [20–23], a novel ODS steel was developed by dispersing nanopowders of lanthana (La_2O_3) via high energy ball milling of elemental powders for 10 h and spark plasma sintering (SPS) at 950 °C for 45 min under an applied pressure of 88 MPa. A high number density of NCs (1.2×10^{24} m^{-3}) enriched in Cr-La-Ti-O with radius of 1.5 ± 0.2 nm was obtained in lanthana-bearing Fe-14Cr based ODS alloy denoted as 14LMT [15]. The shape, morphology, and number density of NCs did not reveal any significant changes after Fe^{2+} ion irradiation at 30 and 500 °C at 10, 50 and 100 dpa. However, radius of NCs decreased slightly after irradiation for 100 dpa sample. Upon irradiation at 500 °C for 100 dpa, the Cr and La concentrations of NCs increased from 8.9 to 12.2 and 6.9 to 8.8 (at%), respectively, and the Ti concentration of NCs decreased from 17.8 to 15.4 [15].

As the cladding materials, ODS alloys are proposed to be used under higher radiation doses up to 600 dpa [5], the focus of the current study was to gain fundamental understanding of the effects of self-ion (Fe^{2+}) irradiation (from 100 to 400 dpa) on the irradiation stability of dispersed oxide NCs (mainly La-Ti-Cr-O) at a relevant temperature (here 475 °C). The work utilized a combination of transmission electron microscopy (TEM) and APT to observe the microstructural evolution and used nanoindentation to probe the localized mechanical property of the irradiated materials. The results are discussed in the light of prevailing theories.

2. Material and methods

2.1. Material processing

The starting constituent powders including Fe (99.9 wt%, average particle size of 40 μm), Cr (99.8 wt%, average particle size of 5 μm), Ti (99.7 wt%, average particle size of 26 μm), La_2O_3 (99.99 wt %, average particle size of 40 nm) and Mo (99.9 wt%, average particle size of 1–2 μm) were mixed in the nominal proportion of Fe–14Cr–1Ti–0.3Mo–0.5La_2O_3 (wt.%). The Mo powder was procured from the Micron Metal Powder Inc., while the rest of the powders were procured from the American Elements Inc.

High energy ball milling was performed in an air-cooled SPEX 8000M mixer/mill for 10 h using 316 stainless steel balls (8 mm in diameter) as the milling media. A milling batch consisted of 100 g steel balls and 10 g powder giving a ball to powder ratio (BPR) of 10:1. The milled powder was consolidated via SPS by using a Dr. Sinter Lab SPS-515S machine (SPS Syntex Inc., Kanagawa, Japan). The milled powder was sintered at 950 °C for 45 min under vacuum (7×10^{-3} Torr or 0.9 Pa). A pulsed DC current with pattern of 12–2 (current on for 12 ms and off for 2 ms), a heating rate of 100 °C/min and a pressure of approximately 88 MPa (10 kN force) were used here to produce the specimens. The final product was in the form of a disk with diameter of 12.5 mm and thickness of 8 mm.

2.2. Heavy ion irradiation

The irradiation experiments using Fe^{2+} ion beam were conducted at the Texas A&M Ion Beam Laboratories using 1.7 MV Tandetron accelerator at temperature of 475 °C. The specimens were irradiated to doses of 100, 200, 300 and 400 dpa (1 dpa was equal to 5.75×10^{14} ions.cm^{-2}) with an ion flux of ~2×10^{12} ions cm^{-2} s^{-1}. Further details of the irradiation experiment has been explained in further details elsewhere [15]. The radiation damage profile (i.e. dpa as a function of depth) is estimated using the Stopping and Range of Ions in Matter (SRIM) 2008.04 software [24].

2.3. Microstructural studies

A focused ion beam (FIB) FEI Quanta 3D Field Emission Gun Scanning Electron Microscope (FEG-SEM) instrument with a Ga-ion source was used to prepare samples for TEM studies. The final thickness of lamella varied between 50 and 100 nm in different specimens. For TEM studies, a FEI Tecnai TF30 FEG STEM operating at 300 kV was utilized. Details of sample preparation has been explained elsewhere [15].

Specimens for APT studies were prepared by the above-mentioned FIB. The APT analysis was aimed at understanding the size and composition of the NCs present in the 14LMT alloy specimens. The APT experiments were performed with the help of a CAMECA LEAP 4000X HR operating in voltage mode at the specimen temperature of 50–60 K and 20% of the standing voltage pulse fraction. The atom maps were reconstructed using CAMECA IVAS 3.6 software. The maximum separation cluster algorithm was used to identify the composition of NCs. This was applied to APT