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Microstructure, hardness and defect structure of the He irradiated ODS ferritic steel

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ABSTRACT

In this paper, the microstructure, hardness and defect structure of the oxide dispersion strengthened Fe-16Cr-2W-0.5Ti-0.4Y_2O_3 (designated as ODS-16Cr) and Fe-16Cr-2W-0.5Ti-0.4Y_2O_3-4Al-1Zr (ODS-16Cr-Al/Zr) steels irradiated with different doses of He ions were investigated by means of TEM, nanoindentation and positron annihilation spectroscopy. In the both alloys, nano-scale bubbles appeared in the vicinity of the precipitates as well as the grain boundaries. High density dislocation networks were observed as well. Moreover, the hardness of the irradiated steels increased distinctly with increasing He implantation. The irradiation hardening in the ODS-16Cr-Al/Zr alloy is more prominent in comparison with the ODS-16Cr alloy. The positron annihilation results illustrated that concentration of vacancy clusters of ODS-16Cr-Al/Zr steel is higher in the near surface under the larger dose, which is one reason of stronger irradiation hardening below the surface of 300–400 nm. The higher concentration of vacancies or vacancy clusters in ODS-16Cr-Al/Zr samples can be attributed to the fact that the solute Al atoms can effectively trap interstitials, which suppresses the annihilation of interstitials with vacancies.

1. Introduction

The oxide dispersion strengthened (ODS) ferritic steels have been developed as one of the most promising candidates of blanket materials for fusion power systems [1,2]. These nanoscale oxide particles act as pinning points for dislocations and grain boundaries, and thus improve the high temperature creep strength. Meanwhile, because the structural materials for future fusion reactors are exposed to the high-energy neutron irradiation, severe displacement damage and high He/H concentration could be generated in these materials, resulting in the microstructure change and irradiation embrittlement [3]. Therefore, nanoparticles dispersed in materials are expected to act as active sites trapping the irradiation induced point defects and He atoms [4]. It was reported that the nano-scale He bubbles could nucleate on the surfaces of nanoclusters, for example the Ti (N,C) precipitates, and along grain boundaries [5]. These observations demonstrate that ODS steel could exhibit significant improvement of radiation resistance.

Energetic ions have long been used to research irradiation response of structural alloys due to the high production rate of cascade damage and low induced radioactivity in specimens. However, damage regions are restricted to the surface of bulk specimens due to the short penetrating depth of ions. Therefore, nanoindentation has been widely used to characterize the properties of the thin ion implanted regions [6]. A model (known as Nix-Gao model) [7] is suggested to extrapolate the experimentally obtained nanoindentation hardness to bulk-equivalent hardness of ion-irradiated alloys and illustrates the irradiation hardening effect during the ions implantation. Moreover, the positron annihilation spectroscopy (PAS) is frequently employed in recent years to investigate the microstructural changes in materials designed for irradiation resistance [8]. Especially, it is a very powerful technique to detect and to identify vacancy clusters ranging from one vacancy to about 20 vacancies which constitute the first stage of the He bubble formation [9].

In our previous studies, the ODS ferritic steel with nominal composition of Fe-16Cr-2W-0.5Ti-0.4Y_2O_3 (weight percent) (designated as ODS-16Cr) was proven to have attractive mechanical

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properties [10,11]. Later, the Fe-16Cr-2W-0.5Ti-0.4Y_2O_3-4Al-1Zr (designated as ODS-16Cr-Al/Zr) were studied, where the addition of 4 wt% Al was effective to improve corrosion resistance in lead-bismuth eutectic (LBE) system [12] and minor Zr addition can prevent the Al element induced coarsening of the oxide particles [13]. The presence of different small-size precipitates in these two alloys leads to a high interface-to-volume ratio, which is considered to be effective for significant improvement of radiation resistance of the material. To check such effects, the samples of ODS-16Cr and ODS-16Cr-Al/Zr were irradiated by He^{2+} ions and the irradiation hardening and defect structure of these two kinds of ODS steels were comparatively analyzed in this paper by nanoindentation and PAS methods.

2. Experimental

The ODS-16Cr and ODS-16Cr-Al/Zr steels used in this study were fabricated by powder metallurgy. The mechanically alloyed powders were vacuum (~10^{-3} Pa) canned and consolidated by hot isostatic pressing (HIP) at 1150 °C for 3 h under 150 MPa. Then, the samples were cut from those ingots and annealed at 800 °C for 2 h. The details of preparation were presented in our previous papers [12,13].

The irradiation experiment was performed at an irradiation terminal of 320 kV high-voltage platform in the Institute of Modern Physics (IMP) in Lanzhou, China. Specimens with size of 10 × 10 × 0.8 mm^3 were cut from the annealed ingots and one surface was mechanically polished to a 0.25 μm finish. The polished surfaces were implanted with 500 keV He^{2+} ions to three increasing fluences of 7 × 10^{15}, 6 × 10^{16} and 6 × 10^{17} ions/cm^2 at room temperature under 7 × 10^{-5} Pa. Variation of the damage dose (solid lines) and ion concentration (dot lines) with implantation depth were calculated by the SRIM 2008, and plotted in Fig. 1 for the two kinds of samples. The maximum damage dose induced by He implantation is up to 0.53, 4.58, 45.89 displacement per atom (dpa) for ODS-16Cr sample at the three He ions fluences, respectively; and the maximum implantation depth of He is 1.2 μm. For ODS-16Cr-Al/Zr alloy, the peak of dpa value is slightly lower than that of ODS-16Cr samples at comparative dose and the predicted He concentration profile reveals a relatively sharp peak at a depth of 1 μm, extending from 600 to 1200 nm (Fig. 1b).

Microstructural characterization was performed with a transmission electron microscope (TEM, JEM-2000FX). The surfaces of the two irradiated specimens were glued face-to-face and then the glued parts were embedded in a brass tube with outer diameter of 3 mm using the same epoxy glue. Next, this tube was cut into 300 μm thick plates. Subsequently, these plates were manually thinned and polished on grinding paper to a thickness of 50 μm. Finally, they were prepared by ion milling from the unirradiated side in a Gatan-691 precision ion polishing system with beam energy of 5 keV at beginning, which was gradually decreased to 3 keV, in order to achieve electron transmission.

Nanoindentation (Nano Indenter G200, Agilent Corp.) was carried out on the irradiated sample surface to investigate the hardening behavior of materials under irradiation. The testing method of Continuous Stiffness Measurement (CSM) was used to obtain the hardness-depth profile continuously up to the depth of about 2 μm. The CSM method was advantageous to obtain the depth profile with only a single indent.

The generation and evolution of defects were studied by the positron annihilation spectroscopy (PAS). Positrons emitted from a ^{22}Na source are accelerated and injected into the samples with a kinetic energy ranging from 1 to 20 keV. Each positron eventually annihilates with an electron in the material resulting in the emission of two 0.511 MeV photons in almost the opposite directions.

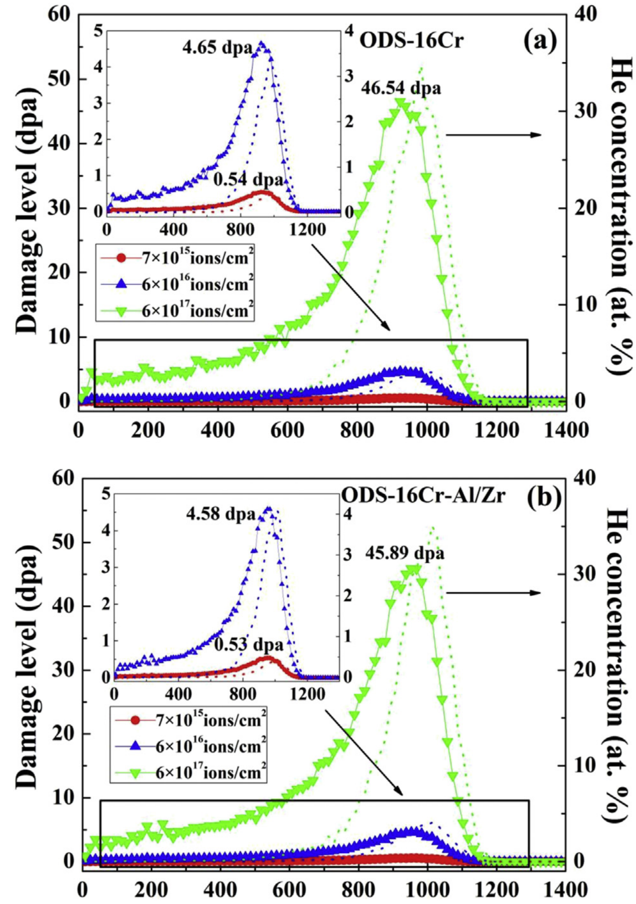

Fig. 1. Depth profiles of the displacement damage and the implanted helium calculated for the investigated irradiation samples (a) ODS-16Cr and (b) ODS-16Cr-Al/Zr samples.

Due to the momentum component of the electron in the direction of the emission (p_L), the measured energy of emitted photons is shifted from 0.511 MeV by an amount given by $\Delta E = 1/2\, cp_L$, owing to the Doppler effect, where c is the light velocity. These shifts lead to a broadening, which is typically a few keV and is quantified by the line-shape parameters S (sharpness) and W (wing). Moreover, the mean positron implantation depth z depends upon its kinetic energy as given by

$$z = (A/\rho)E^n \tag{1}$$

where A is a material-independent constant and can be taken as 40 according to the empirical formula [14], ρ is the density of the materials and E is the positron kinetic energy, n is chosen as 1.62 according to reference [15]. In the present case, the density of the ODS-16Cr and ODS-16Cr-Al/Zr alloys is 7.76 g/cm^3 and 7.51 g/cm^3, respectively, and the maximum mean implantation depth at 20 keV is about 700 nm.

3. Results and discussion

3.1. Microstructure

The typical TEM images of the two samples were presented in Fig. 2. In the ODS-16Cr steel after 6 × 10^{16} ions/cm^2 irradiation high-density dislocations can be detected (Fig 2a). A nano-scale oxide particle (about 30 nm) is shown in Fig. 2b. A lot of small