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Radiation response of ODS ferritic steels with different oxide particles under ion-irradiation at 550 °C

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HIGHLIGHTS

- Morphology of oxide particle dispersion, rather than the O/M interface structure, is the main factor to suppress swelling.
- No hardening is induced in the three ODS steels irradiated with both single Fe^{3+} and dual (Fe^{3+} and He^{+}) ions at 550 °C.
- Negligible hardening by He bubbles is probably dueto their high fractions (~74%–82%) adjacent to the oxide particles.

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ABSTRACT

In order to investigate the effects of oxide particles on radiation response such as hardness change and microstructural evolution, three types of oxide dispersion strengthened (ODS) ferritic steels (named Y-Ti-ODS, Y-Al-ODS and Y-Al-Zr-ODS), mostly strengthened by Y-Ti-O, Y-Al-O and Y-Zr-O dispersoids, respectively, were simultaneously irradiated with iron and helium ions at 550 °C up to a damage of 30 dpa and a corresponding helium (He) concentration of ~3500 appm to a depth of 1000–1300 nm. A single iron ion beam irradiation was also performed for reference. Transmission electron microscopy revealed that after the dual ion irradiation helium bubbles of 2.8, 6.6 and 4.5 nm in mean diameter with the corresponding number densities of 1.1×10^{23}, 2.7×10^{22} and 3.6×10^{22} m^{-3} were observed in Y-Ti-ODS, Y-Al-ODS and Y-Al-Zr-ODS, respectively, while no such bubbles were observed after single ion irradiation. About 80% of intragranular He bubbles were adjacent to oxide particles in the ODS ferritic steels. Although the high number density He bubbles were observed in the ODS steels, the void swelling in Y-Ti-ODS, Y-Al-ODS and Y-Al-Zr-ODS was still small and estimated to be 0.13%, 0.53% and 0.20%, respectively. The excellent swelling resistance is dominantly attributed to the high sink strength of oxide particles that depends on the morphology of particle dispersion rather than the crystal structure of the particles. In contrast, no dislocation loops were produced in any of the irradiated steels. Nanoindentation measurements showed that no irradiation hardening but softening was found in the ODS ferritic steels, which was probably due to irradiation induced dislocation recovery. The helium bubbles in high number density never contributed to the irradiation hardening of the ODS steels at these irradiation conditions.

1. Introduction

Oxide dispersion strengthened (ODS) steels owe their high performance to the ultrahigh density of nanoscale oxides and fine grains. Because of their superior tensile, creep and fatigue strength at elevated temperatures [1,2] and excellent neutron radiation tolerance [3], ODS steels can be used as a fusion blanket structural material to elevate the operating temperature and the overall thermal efficiency of fusion plants [4]. In addition, ODS ferritic steels with sufficient Al addition, which possess high corrosion/oxidation resistance, have been considered as a candidate fuel cladding for the next generation fission reactors [1,5] and light water reactors with accident tolerant fuel [6]. In harsh nuclear service environments, especially in fusion components, helium atoms generated through (n, α) nuclear reactions or directly from in-core plasma lead to the formation of interstitial helium atoms and helium-vacancy clusters, such as helium bubbles or voids in

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structural materials, which may result in the degradation of mechanical properties, such as hardening and embrittlement, as well as swelling [7].

Concerning ODS steels, it is commonly accepted that helium effects are mitigated by numerous dispersed oxide particles, acting as effective sinks providing sites for defect accumulation, combination or annihilation [3,4]. Previous studies confirmed the trapping effects of oxide particles on nanometer-sized cavities by means of transmission electron microscopy (TEM) [8], scanning TEM [9,10] and atom probe tomography (APT) [11] after various types of irradiations and post-irradiation annealing treatments in the temperature range of 400–750 °C. Meanwhile, two dominating nucleation styles of cavities in association with oxides were suggested by G.R. Odette [12]: one is the oxide particle that contains cavities and the other is the oxide particle attached with cavities in a side to side way. To our knowledge, the former was only found in amorphous Y-Al-O particles [13]. The previous experimental results revealed that not only oxide particle/matrix (O/M) interfaces could provide preferential nucleation sites for cavities, but also the oxides themselves might have some solubility for helium or helium bubbles. The effect of oxide type on the helium solubility was investigated by related simulation studies on various oxides (e.g. Y-Ti-O, Y-Al-O oxides) [14,15]. Finally, as expected, He-induced swelling was effectively suppressed by oxide particles and was well demonstrated by comparing ODS steels and non-ODS steels [16,17]. Ion-irradiation experiments of ODS steels with different oxide particle types, sizes and number densities were mainly studied by single He ion irradiations at relatively low displacement damages, indicating that finer oxide particles with a higher number density contributed to a smaller swelling [18,19]. Regarding the effect of simultaneous dual beam (Fe and He ions) irradiation, the amount of research is limited.

As for the peak swelling temperature, several previous studies indicated that the swelling in ferritic/martensitic (F/M) alloys peaked at around 420–470 °C in ion irradiations [20] and neutron irradiations [21] and is affected by the damage rate. Since helium may reduce the mobility of vacancies and increase the stability of voids, helium effects are expected to be enhanced at a higher temperature than 470 °C. In the previous research on helium effects in ODS ferritic steels, the targeted irradiation temperatures were ranging up to 700 °C and the maximum swelling rate was at 500–600 °C [22] because the mobility of helium might also become larger at these high temperatures [23]. It is considered that the peak swelling is determined as a result of a balance between the mobility of vacancies and thermal stability of voids.

Helium-induced hardening is another key concern for the structural materials in fusion systems. At room temperature, the implantation of about 750 appm He to an ODS ferritic steel gave rise to an obvious hardening of 21% with homogeneously distributed helium nanobubbles [24]. Likewise, hardening became larger with increasing helium concentration obeying a 1/2-power law [25]. But in another ODS ferritic steel implanted with about 3400 appm He, as the temperature increased from 100 to 500 °C, hardening decreased continuously from ~15% to ~3.5% [22]. These results elucidated that the hardening behavior was affected by implantation temperature and even materials. Furthermore, the difference in hardening between single Fe ion and dual (Fe and He) ion beam irradiations was significantly influenced by irradiation temperature: at 300 °C, the hardening caused by dual beam irradiation was evidently larger than that by single Fe ion irradiation [26], while at 500 °C, the hardening was almost the same between the two [27]. A simulation study indicated that helium bubbles of 2 nm in diameter with more than 5 helium atoms per vacancy were strong obstacles to the glide motion of an edge dislocation in iron [28], and significant hardening has been observed in reduced-activation ferritic/martensitic (RAFM) steels irradiated up to 11.3 dpa/1175 appm He in the Swiss Spallation Neutron Source (SINQ) [29]. In contrast to these hardening effect of helium, R. Kasada et al. reported that the hardening induced by helium ion irradiation (580 at. ppm He/0.226 dpa at below 423 K) in a reduced-activation martensitic steel was interpreted simply in terms of displacement damage, suggesting that there is no significant effect of helium on the hardening [30]. Therefore, in fusion relevant temperature region (~400–700 °C for F/M steel) [31], helium effects on irradiation hardening are still uncertain.

Since the material performance of ODS steels depends on the morphology of oxide particle dispersion, dispersion control is essential for development of the steel. The Al-addition to FeCr-ODS steels altered the main particles from $Y_2Ti_2O_7$ pyrochlore to YAH and YAP [32], and a small additions of Zr to FeCrAl ODS steels altered the majority of oxide particles from Y-Al-O dispersoids to Y-Zr-O dispersoids [33], resulting in an enhanced high-temperature strength of the steel [1].

In this research, we investigated the radiation response of three types of ODS ferritic steels mostly strengthened by Y-Ti-O, Y-Al-O and Y-Zr-O oxides, respectively, focusing on the roles of helium implantation in the hardness change and swelling under dual beam irradiation at 550 °C.

2. Experimental

2.1. Materials

The materials used in this research were three types of ODS ferritic steels, Fe-13.6Cr-1.9W-0.16Ti-0.33Y_2O_3 (Y-Ti-ODS), Fe-15.42Cr-3.8Al-1.85W-0.1Ti-0.36Y_2O_3 (Y-Al-ODS) and Fe-14.59Cr-3.46Al-1.84W-0.14Ti-0.27Zr-0.33Y_2O_3 (Y-Al-Zr-ODS), which were produced by mechanical alloy processing. After hot extrusion and forging were performed at 1150 °C, the materials were finally annealed at 1150 °C for 1 h and followed by air-cooling. More details of the material conditions and processing were described in Refs. [19,33,34].

Specimens were sampled so that the specimen surface was perpendicular to the extrusion direction. Specimen surface was mechanically ground with SiC papers, and subsequently polished with successive grades of diamond spray down to 0.25 μm. Finally, electrolytic polishing was conducted in a solution of 10% (vol.) $HClO_4$ and 90% (vol.) CH_3COOH at room temperature except for Y-Al-ODS.

2.2. Irradiation conditions

Specimens were simultaneously irradiated with 6.4 MeV Fe^{3+} and 1 MeV He^{+} at the DuET facility in Kyoto University [35]. A single Fe^{3+} irradiation was also carried out for comparison. The beams were raster scanned at a frequency of 1000 Hz in a horizontal direction and 300 Hz in a vertical direction. In the dual beam irradiation, the He^{+} beam was declined with 45° to the normal of the specimen surface and maintained a simultaneous injection with Fe^{3+} beam. A rotating energy degrader foil was applied for obtaining a rather homogenous distribution of helium atoms in specimens. The fluxes of He^{+} and Fe^{3+} ion beams were approximately 1.5×10^{16} ions m^{-2} s^{-1}, 1.7×10^{16} ions m^{-2} s^{-1}, respectively. The irradiation temperature was measured by an infrared thermography to be 550 °C within a fluctuation of ±10 °C. All three types of ODS steels were irradiated together for about 8 h.

The injected helium concentration and displacement damage (displacements per atom, dpa) were obtained by SRIM [36] simu-