Helium irradiation hardening in Fe, F82H-IEA and F82H-ODS steel

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(Received: 21 October 2014 / Accepted: 19 January 2015)

Helium implantation at room temperature was performed to investigate irradiation hardening of ferritic/martensitic steels. Bulk hardness was extracted from Nix-Gao model using a Berkovich nano-indentor. The correlation between irradiation hardening and helium concentration ranging 500 - 2000 appm was examined. Nano-hardness increases as a function of helium concentration. The hardening of Fe is saturated when the concentration of helium exceeds 1000 appm. No saturation tendency is observed for F82H-IEA and F82H-ODS. F82H-ODS, having a higher hardness, provides a lower irradiation hardening than F82H-IEA. Transmission electron microscopy (TEM) reveals that cavities with a uniform distribution are formed after helium implantation to 2000 appm, showing a mean size of 1.4 nm with an average number density of 5.8×10^{23} m^{-3} in Fe, 1.1 nm with 4.9×10^{23} m^{-3} in F82H-IEA and 1.3 nm with 7.4×10^{23} m^{-3} in F82H-ODS. The results reveal that the formation of defects by helium irradiation at room temperature is independent from the as-received microstructure, while the change of mechanical property is dependent on the microstructure of the as-received materials. This suggests the same type of irradiation induced defect may have a quite different hardening effect on materials with different microstructure.

Keywords: helium, irradiation hardening, cavity, oxide particle, nanoindentation

1. Introduction

Neutron with 14 MeV energy can produce transmuted helium by reacting with constituent atoms in structural materials. The effect of transmuted helium, which will cause the mechanical property degradation, is a critical issue for the application of structural materials in fusion reactors. An increasing attention has been paid to the increase of strength – irradiation hardening.

F82H-IEA and F82H-ODS steels are candidate materials for structure applications in fusion reactors due to their high radiation resistance. Oxide dispersion strengthened (ODS) reduced activation ferritic/martensitic (RAFM) steels attract an increasing attention because of its advantage over high temperature strength[1-3]. In addition, it is suggested that high density of nano-scale oxide particles are effective trapping sites for helium atoms, as well as vacancies, to resultantly suppress helium effect[4-6]. However, further investigation is required for understanding the suppression effect of the oxide particles.

In this work, we report helium irradiation hardening of Fe, F82H-IEA and F82H-ODS based on Nix-Gao model. The micostructure with cavities was observed for as-irradiated samples. This work investigates the mechanical property change and microstructure evolution with helium irradiation at room temperature.

2. Experimental procedure

Pure iron (purity better than 99.95%, produced by Johnson Matthey Chem. Ltd.), F82H-IEA and F82H-ODS steels (both provided by Japan Atom Energy Agency) were punched into 3 mm diameter disks. The punched out specimens of Fe were placed in a vacuum in a quartz tube (1×10^{-4} Pa) and then annealed at 873 K for 1 h. Then the specimens were removed from the tube, and electrochemically polished with Acetic acid: Perchloric acid = 19:1 to reduce stress concentrations on the surface and obtain a smooth surface for helium implantation. 70 - 250 kV He ion implantation (in Hokkaido University) was performed at room temperature. Chemical compositions and heat treatment conditions are shown in Table 1[7, 8]. The average helium concentration was 500 - 2000 appm. Fig. 1 shows Stopping and Range of Ions in Matter (SRIM) calculations of helium and displacement profile, showing an approximately constant He level of 0.2 at.% and the displacement damage of 0.1 dpa. The displacement energy used in the SRIM calculation is 25 eV. The implanted region reaches to 800 nm from the surface with a relatively homogeneous distribution of helium in the range of 200 - 650 nm.

Nano-hardness test was applied using an Elionix ENT-1100a (Elionix Inc. Japan) nano-indenter with Berkovich-type tip. 5×6 array indents with 30 μm interval and the hardness was calculated using Oliver and Pharr method[9].

A JEOL JIB-4601F focused ion-beam (FIB) micro-processing device, operated at 30 keV, was used to prepare XTEM foils. Then the foils were polished using a

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Table 1.
Chemical compositions and heat treatment conditions of Fe, F82H-IEA and F82H-ODS steel (mass%).

	C and N	Si	Cr	W	V	Ta	Ti	Y_2O_3	Ex. O	Fe
Fe	0.04	–	–	–	–	–	–	–	–	bal.
F82H-IEA	0.10	0.11	7.71	1.95	0.16	0.02	–	–	–	bal.
F82H-ODS	0.17	0.013	7.85	1.9	0.18	0.10	0.19	0.368	0.092	bal.

Fe: tempered at 600 °Cfor 1h
F82H-IEA: normalized at 1040 °Cfor 38min and tempered at 750 °Cfor 1h
F82H-ODS: normalized at 1050 °Cfor 1h and tempered at 750 °Cfor 1h

precision ion polishing system (PIPS) at 1 keV to remove the damage layer induced by FIB. The micro-structure was observed by using a 200 kV transmission electron microscope (TEM).

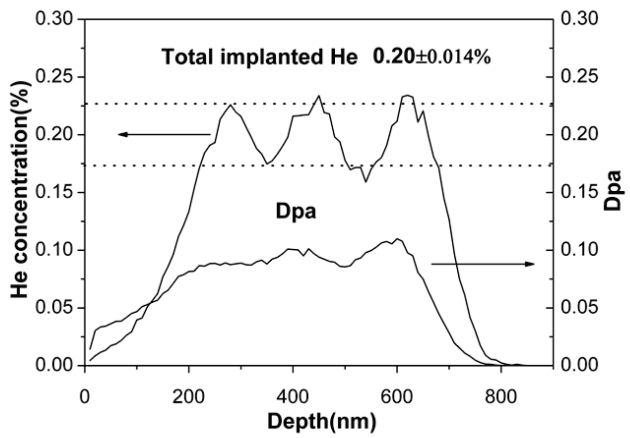

Fig. 1 SRIM calculations of He and displacement depth profile, where concentration of He is ~0.2 at.% and displacement damage is ~0.1 dpa.

3. Results and discussion

Fig. 2 shows typical microstructure of Fe, F82H-IEA and F82H-ODS before He irradiation. Fe contains few pre-existing defects. The number density of dislocation is less than 10^{12} m^{-2}. F82H-IEA contains martensite laths and precipitates along lath boundaries. The number density of dislocation is $(5\pm1)\times10^{14}$ m^{-2}. Relative to F82H-IEA, F82H-ODS contains a large number ($(1.1 \pm 0.04)\times10^{23}$ m^{-3}) of oxide particles with mean size of (3.8 ± 1.3) nm[10]. The number density of dislocations is $(1.1\pm0.8)\times10^{14}$ m^{-2}.

Fig. 3 shows helium concentration dependence of bulk hardness extracted from Nix-Gao model and irradiation hardening for Fe, F82H-IEA and F82H-ODS. Bulk hardness increases as a function of helium concentration. The bulk hardness of Fe is saturated when

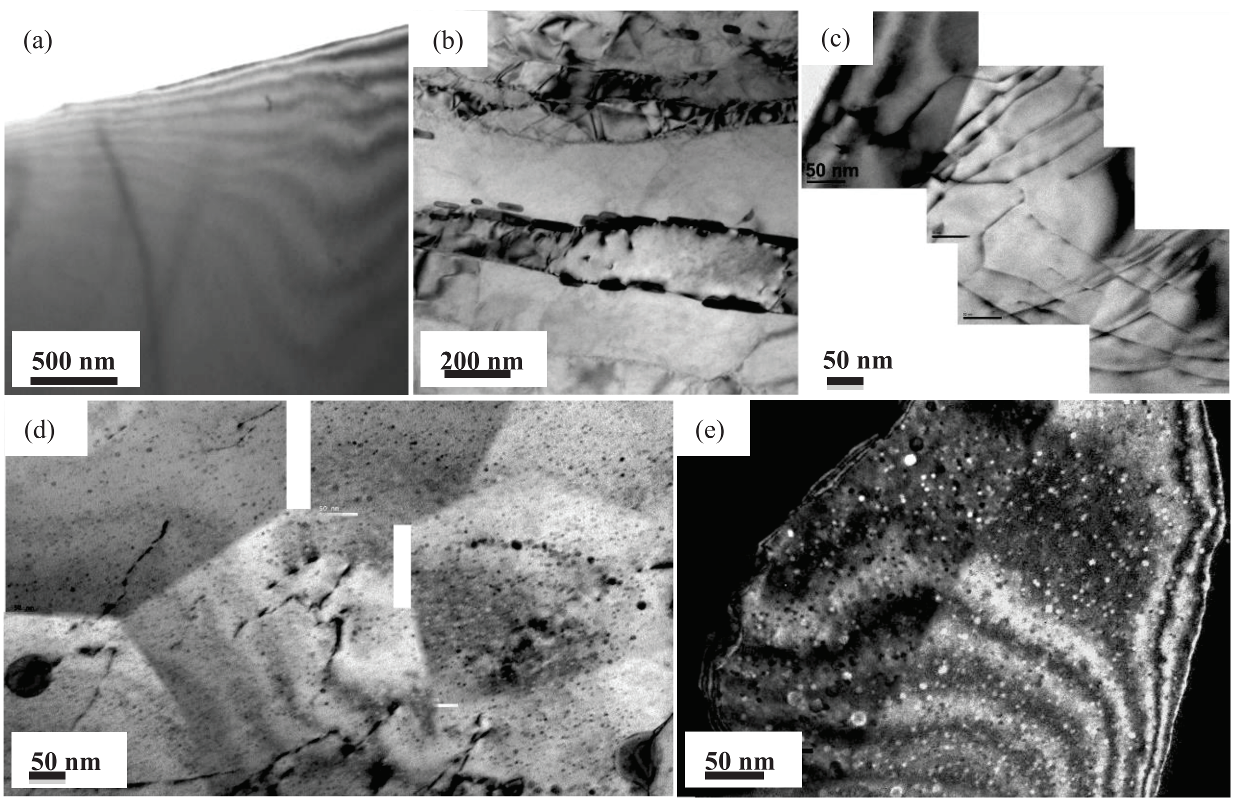

Fig.2 Bright field micrographs of Fe (a), F82H-IEA showing structure of matensite lath (b) and dislocation (c), and F82H-ODS showing dislocation (d) and weak-beam dark field image showing nano-scale oxide particles(e).

the He concentration is higher than 1000 appm. F82H-ODS with a higher hardness shows a smaller increase in bulk hardness than F82H-IEA. Irradiation hardening is defined as the bulk hardness difference between samples before and after helium irradiation. Irradiation hardening (ΔH) is 3-5, 1-3, and 0.5-1 GPa with helium concentration of 500-2000 appm for Fe, F82H-IEA and F82H-ODS, respectively.

Fig. 4 shows under-focused (Δf = -500 nm) TEM images of 0.2 at.% He implanted Fe, F82H-IEA and F82H-ODS. The images indicate that cavities uniformly appear as white dots surrounded by a dark Fresnel fringe. The mean size of the cavities is 1.4 nm with an average number density of 5.8 × 10^{23} m^{-3} for Fe, 1.1 nm with 4.9 × 10^{23} m^{-3} for F82H-IEA and 1.3 nm with 7.4 × 10^{23} m^{-3} for F82H-ODS, respectively. Black dots were also observed.

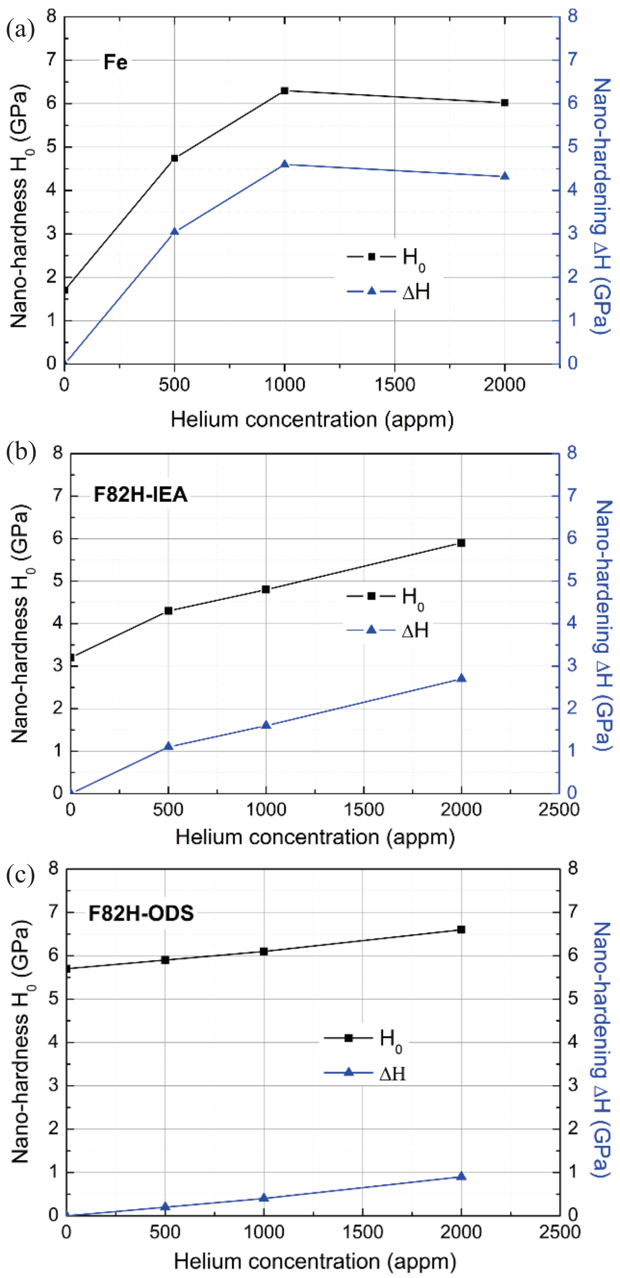

Fig.3 Helium concentration dependence of bulk hardness extracted from Nix-Gao model by using nanoindentation and irradiation hardening.

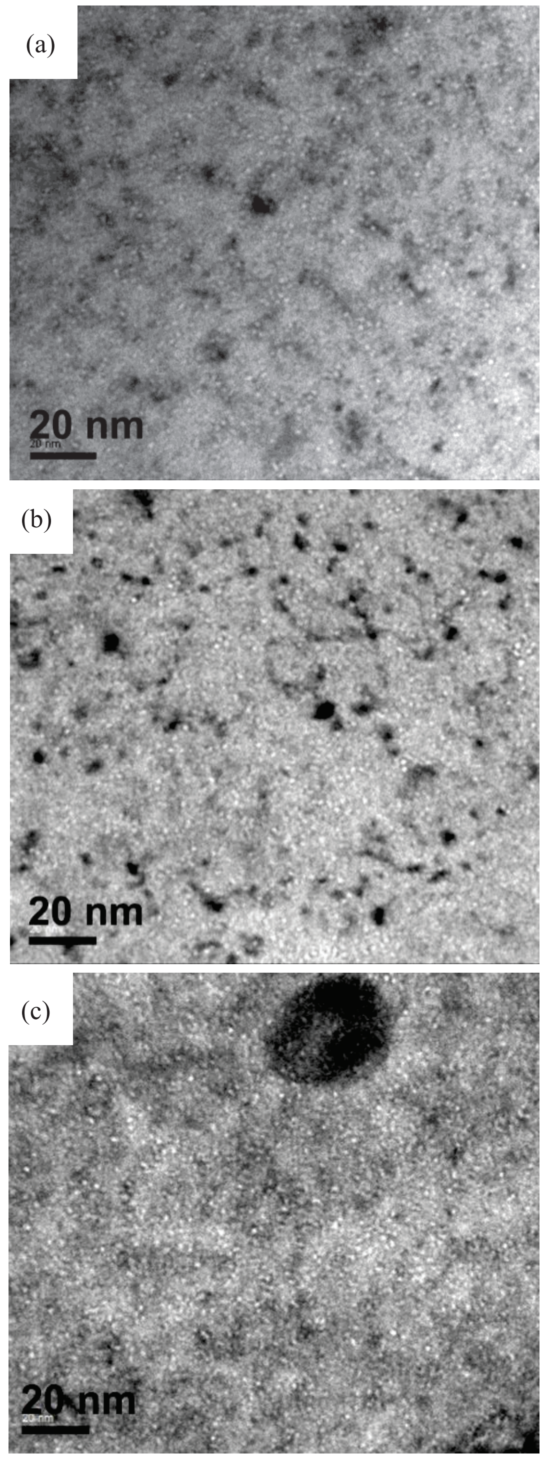

Fig.4 Cavities appearing in Fe (a), F82H-IEA (b), and F82H-ODS (c) after He irradiation with 2000 appm helium.

Although the microstructure of as-received Fe, F82H-IEA and F82H-ODS are quite different, the mean size and distribution of cavities induced by He irradiation at room temperature are not largely different. While, the irradiation hardening differs significantly with materials. Irradiation hardening is produced by helium irradiation. Fe possesses the highest irradiation hardening, followed by F82H-IEA and then F82H-ODS. Irradiation hardening of Fe is saturated when the He concentration is higher than 1000 appm. No saturation tendency was observed for F82H-IEA and F82H-ODS. The results suggest that the microstructure of the as-received materials does not have significant effects on the induced defects by He irradiation at room temperature. However the contribution of the induced defects to the strengthening depends on the microstructure of as-received materials.

4. Conclusions

Helium irradiation induced hardening at room temperature was examined for Fe, F82H-IEA and F82H-ODS. Irradiation hardening occurred by helium implantation with concentration ranging 500-2000 appm, resulting in 3-4, 1-3, and 0.5-1 GPa hardening in Fe, F82H-IEA and F82H-ODS, respectively. The dependence of He irradiation induced hardening with He concentration was investigated. F82H-ODS exhibited a lower irradiation hardening than Fe and F82H-IEA.

The effect of He irradiation on microstructure evolution in Fe, F82H and its ODS steel was examined by TEM observation. Cavities with homogeneous distribution were observed in all materials with He concentration of 2000 appm. The mean size and number density of cavities are not largely different for Fe, F82H-IEA and F82H-ODS.

By He irradiation at room temperature, the mechanical property change is dependent on the microstructure of as-received materials. A material with higher hardness indicates a lower hardening. However, the as-received microstructure does not affect significantly the He irradiation induced defects at room temperature in this experiment.

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