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Evolution of dislocation loops in annealed iron pre-irradiated with hydrogen ion in high-voltage electron microscope

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HIGHLIGHTS

- Evolution of dislocation loops was studied in high voltage electron microscope.
- Interstitial and vacancy loops formed simultaneously in hydrogen ion implanted iron.
- The bias factor of two types of loops was compared directly in this work.

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ABSTRACT

The nature of dislocation loops in the annealed pure iron pre-irradiated with hydrogen ion at room temperature was studied by the evolution of loops under electron irradiation in high-voltage electron microscope (HVEM). Only interstitial-type loops were observed when annealed and electron irradiated at 350 °C but only vacancy-type loops formed at temperature higher than 500 °C. When annealed at temperatures from 450 °C to 490 °C, both interstitial-type and vacancy-type loops formed simultaneously in the specimen and vacancy-type loops accounted for an increasing fraction with increasing annealing temperature, from 28.5% at 450 °C to 55% at 490 °C. The bias factor of interstitial-type and vacancy-type loops was compared based on the growth rate or shrinkage rate of the dislocation loops. The bias factor of interstitial-type loops was demonstrated to be higher than that of vacancy-type loops at all three annealing temperatures.

1. Introduction

A well understanding of the accumulation and evolution of point defects in pure iron will allow us to better understand the mechanism of degradation and predict the mechanical properties of more complex materials [1]. In general, interstitial atoms or their clusters are more mobile than their vacancies counterparts and migrate to form dislocation loops while leaving the vacancies behind at relatively low temperature. Thus, these residual vacancies will aggregate to form voids, which lead to the swelling of materials. Simulation experiment in iron has proved the bias effect of dislocations that they absorb more interstitial atoms than vacancies [2,3]. Dislocation loops are also considered as bias sinks and a main reason of irradiation swelling. The nature and Burgers vectors of the loops have an impact on their bias and thus influence the mechanical property. A computer simulation predicted that interstitial loops would contribute a larger effect on radiation hardening than vacancy loops [4], so it's of importance to partition the different types of dislocation loops to investigate the behavior of radiation damage in materials.

Two different types of dislocation loops, i.e., interstitial-type and vacancy-type loops, might form after irradiation in materials. Most of studies focus on the interstitial-type loops in materials under kinds of irradiations [5–8]. High density of interstitial-type loops formed when irradiated at low temperature in pure iron and then their density decreased and size increased during high temperature annealing, via migration and coalescence of dislocation loops [6,9–11]. Effect of alloying elements or impurities on the formation and migration of dislocation loops were also extensively studied [9,10,12–17]. In particularly, hydrogen and helium gas atoms have been proved to be effective for the nucleation of the interstitial-type loops [18,19]. However, few studies were reported on the vacancy-type loops in iron [20,21]. Wan et al. [22,23] reported the

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formation of vacancy-type loops with a size as large as 100 nm in hydrogen ion implanted pure iron after annealing at elevated temperature with higher doses. Same vacancy-type loops were also confirmed in the case of deuterium ion implantation [24]. Vacancy-type loop formation takes place in most cascades in copper and other metals, but much less readily in pure iron. The difficulty of formation of vacancy-type loops in iron might be related to the greater stability of voids than vacancy loops [25]. Recently, Aliaga et al. [26] revealed that vacancy loops tended to form near the free surface of the specimens irradiated with low dose of low energy heavy ions using molecular dynamics simulations.

In our present work hydrogen ions were pre-implanted to pure iron at room temperature. The nature of dislocation loops formed during annealing was investigated by in-situ observation in high-voltage electron microscope (HVEM). Evolution of two different types of dislocation loops under electron irradiation was most of our concern.

2. Materials and methods

Iron samples with 99.98% in purity were cold-rolled to thin plate of 0.15 mm thickness, and then punched into disks of 3 mm in diameter. To eliminate the effect of cold working, the disks were annealed at 700 °C for 60min, followed by quenching in water. The final TEM specimens were made using a twin jet-polishing device, using an electrolyte of 5% (volume) $HClO_4$ and 95% ethanol solution close to −20 °C with the voltage ~25V.

Hydrogen ions of 50 keV were implanted to the TEM specimens to a fluence of 1×10^{17} ions/cm^2 by ion accelerator at room temperature. Irradiation damage was calculated using the software of stopping and range of ions in matter (SRIM-2008) as shown in Fig. 1. The average radiation damage in the typical TEM observation region with depth of 100–150 nm was calculated to 0.3 dpa and the corresponding hydrogen concentration reached up to 7500 appm. After hydrogen ion irradiation, the specimens were annealed at 350 °C, 400 °C, 450 °C, 470 °C and 490 °C and 500 °C respectively in high vacuum for 30 min, followed by in-situ observation at a JEM-ARM 1300 high-voltage electron microscope (HVEM) in Hokkaido University with electron energy of 1250 keV. Images of dislocation loops under in-situ observation were taken with electron beam $\boldsymbol{B} = [\bar{1}11]$ and diffraction vector $\boldsymbol{g} = (110)$.

To identify the nature of dislocation loops, inside-outside method is often used from the contrast of dislocation loops in bright-field images, which is reliable for dislocation loops with size larger than 10–20 nm. Here in our study, in-situ electron irradiation was used for determination of the nature of loops, which was pioneered by Kiritani et al. [27–30]. Electron irradiation produces an interstitial dominant atmosphere during early stage of irradiation simply due to higher mobility of interstitials than vacancies. One can tell the nature of dislocation loops, because interstitial-type dislocation loops are expected to grow and vacancy-type to shrink or disappear. The electron flux should be controlled not too high to avoid ambiguity caused by the formation of defect clusters during the electron irradiation. Electron flux of 3×10^{23} e$^-$/m^{2}s was used to introduce pure Frenkel pairs in this study. The effect of surface in the TEM thin films will be discussed in the discussion section.

3. Results

After hydrogen ion implantation at room temperature, high density of small clusters formed in the iron specimens. These small clusters are supposed to be of interstitial-type because of the low irradiation temperature, though no attempt was made to identify their nature. The density of these clusters depends on the irradiation temperature, beam flux and the final fluence, as well as the types of ion beam. The volume density of dislocation loops after hydrogen ion irradiation at room temperature to 0.1 dpa was in the order of 10^{23} m^{-3} [31]. When annealed at higher temperature, interstitial clusters will migrate and coalesce to form dislocation loops, with low density and large loop size. Meanwhile, vacancy clusters also become mobile and start to migrate, resulting in the annihilation of interstitial clusters and the formation of vacancy loops. The number density of dislocation loops was measured using the same diffraction vector $\boldsymbol{g} = (110)$ and plotted in Fig. 2 with thickness of 200 nm. The thickness of the field was not measured directly but assumed to be in the range of 150–200 nm according to their brightness under high voltage electron microscope. The number density of dislocation loops at 500 °C was 8 times lower compared with that at 350 °C.

Fig. 3 shows the evolution of dislocation loops in annealed iron pre-irradiated with hydrogen ion and in-situ electron irradiation in HVEM at the same temperature. As can be seen, when the TEM specimens were annealed at 350 °C, all of the dislocation loops were observed to grow under electron irradiation; However, when annealed and electron irradiated at 400 °C, a small fraction (2.9%) of dislocation loops were observed to shrink (the circled dislocation loop in the figure) except for the popular growth; When the

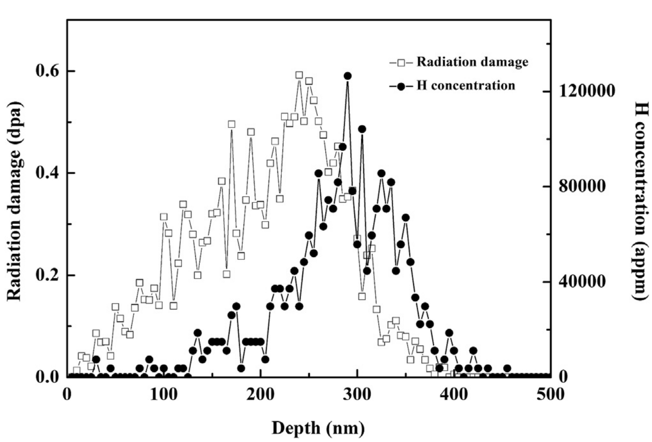

Fig. 1. SRIM calculation of irradiation damage and H concentration profile in hydrogen ion implanted pure iron.

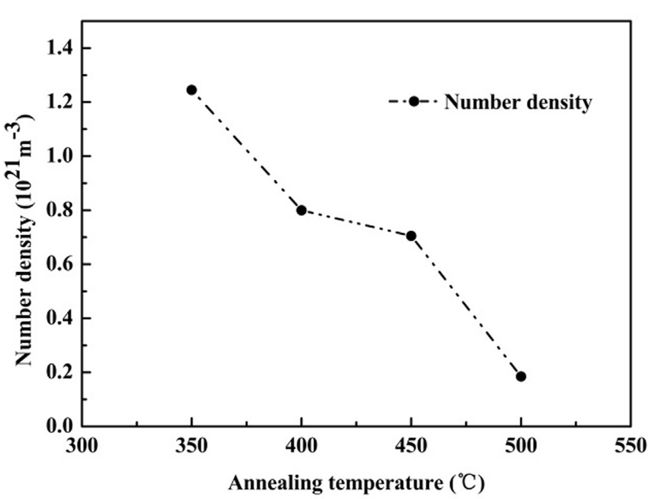

Fig. 2. Number density of dislocation loops as a function of annealing temperature in hydrogen ion implanted iron.