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The behavior of vacancy-type dislocation loops under electron irradiation in iron

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ABSTRACT

Two different types of dislocation loops were found in hydrogen ion implanted pure iron after annealing. The natures of these dislocation loops have been characterized by the inside–outside method of transmission electron microscope (TEM). It is found that the dislocation loops in specimens annealed at 670 K are interstitial type with $b = 1/2\langle 1\,1\,1\rangle$, $n \approx \langle 1\,1\,2\rangle$, and the dislocation loops in the specimens annealed at 770 K are vacancy type with $b = 1/2\langle 1\,1\,1\rangle$, $n \approx \langle 0\,1\,1\rangle$ or $b = \langle 1\,0\,0\rangle$ and, $n \approx \langle 1\,1\,2\rangle$. Adding nickle element in iron could decrease the formation temperature of the vacancy loops.

1. Introduction

Interstitial atoms and vacancies, known as Frenkel pairs, are produced during irradiation process in materials. It is not so easy to check the existence of vacancies by TEM until they effect the diffusion of alloy elements, or form into voids which result in irradiation swelling of materials. In contrast, interstitial atoms would accumulate as dislocations at very early stage of irradiation, and such dislocations could appear clearly under TEM observation. The behavior of irradiation defects, including interstitial atoms and vacancies, may be investigated from dislocations evolution.

The neutrons with energy of 14 MeV released by D–T fusion reaction will produce a large number of crystal defects and in turn cause serious irradiation damages in materials. At the same time, the neutron irradiation will also produce transmutation reactions of (n,p) and (n,α) and results in the formation of helium or hydrogen in materials. Such kinds of gas atoms may play a important role in the damage behavior during irradiation.

There are many reports about the effect of hydrogen on irradiation damage in fcc metals [1–5] than that in bcc metals in the literature. It was usually thought before that hydrogen would escape from bcc metals very easily, thus having little effect on radiation damage at elevated temperatures. However, our prior work has proven the existence of the hydrogen effect on irradiation damage in bcc metals [6,7].

In general, almost all dislocation loops formed in pure iron after irradiation were of interstitial type. However, vacancy-type dislocation loops with a size larger than 100 nm in diameter were found in pure iron implanted by hydrogen [6]. Our research showed that under electron irradiation, these vacancy-type dislocation loops would shrink and eventually disappear. Vacancy-type loops were also found to form in deuteron ion implanted iron, which allowed us to demonstrate the difference in sink strength between hydrogen and deuteron implantation [7].

In our present work hydrogen or helium ions were implanted in TEM specimens of pure iron at room temperature (R.T.). After ion implantation, the specimens were aged in vacuum at higher temperature, allowing the implantation defects to develop into large clusters. Electron irradiation was carried out using high voltage electron microscopy to observe the change of the clusters.

Much detailed work was needed to analyze these vacancy loops, such as their Burgers vector and the effect of alloy element on the formation of vacancy loops. To compare with hydrogen, helium ions were also introduced to bcc iron to study its effect on dislocation evolution during irradiation.

2. Experimental method

Iron sample with 99.98% in purity was cold-rolled into a plate of 0.15 mm thickness, then punched into disks of 3 mm diameter. To eliminate the defects formed by cold working, the disks were annealed in vacuum of 10^3 Pa at 970 K for 60 min, followed by quenching in cold water. A dual jet-polish device was used to thin the disks into specimens for TEM experiment.

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Hydrogen ions or helium ions were implanted to the specimens at 50 KV at room temperature to 1×10^{21} ions/m^2 by ion accelerator. The energy of these ions could keep the gas atoms and crystal defects staying in a depth appropriate for TEM experiment, shown by TRIM computer code. After ion implantation, the specimens were aged in vacuum at high temperature, allowing implanted defect clusters to develop into large dislocation loops.

Electrons are needed to have a energy more than about 500 KV to produce irradiation defects in materials. So the observation by a TEM with a accelerating voltage as low as 200 KV would never introduce any irradiation defects in metal samples. The defects formed by electron irradiation are pure Frenkel pairs, i.e. vacancy-interstitial pairs. The higher mobility of interstitial atoms results in more interstitial atoms than vacancies interacting with a dislocation loop during electron irradiation. This difference of mobility between interstitial atoms and vacancies was used to identify the type of a dislocation loop.

Electron irradiation was carried out using a high voltage electron microscope (HVEM) with a electron flux of 3×10^{23} m^2 s at 1 MV. The changes of dislocation loops under electron irradiation were observed in-situ using HVEM. The nature of dislocation loops was determined by the inside- outside method [8] of TEM experiment with accelerate voltage of 200 KV at R.T.

3. Results and discussion

Many tiny defect clusters formed in bcc iron after hydrogen ion implantation at R.T. These tiny defect clusters may be of interstitial type, or vacancy type, but are usually too small to be easily identified as a particular type of cluster. When aging at high temperature, the tiny clusters develop into larger dislocation loops with the number density of defect clusters decreasing.

Fig. 1a shows the dislocation loops which formed after aging at 670 K. All of these loops are of interstitial type and would grow continuously upon electron irradiation at same 670 K. Fig. 1b and c are the loops after irradiated to 84 s and 140 s respectively.

Such kind of loop growth could always be observed if aging temperature was lower than 700 K. That means that when aging at lower than 700 K, the interstitial type clusters would move to form in loops, but the vacancy type clusters would not move.

If aging temperature was lifted to as high as 770 K, the vacancy type clusters start to move to clear the interstitial loops which were already formed at lower temperature aging. At last, an excess of vacancy clusters may result in the formation of vacancy type dislocation loops, shown as in Fig. 2a, a different field from Fig. 1a in the same sample. The vacancy type loops would shrink to disappear when absorb the interstitial atoms which were produced under electron irradiation at same 770 K, shown as in Fig. 2b and c.

As shown before, the formation temperatures of these two types of loops are different, the former is lower (around 670 K), and the later is higher (around 770 K). It is important to note that both of the two types of loops can be observed at same time if aging at 720 K [6].

Usually only interstitial type loops could be observed in irradiated pure iron without hydrogen, and all of the interstitial loops will grow under electron irradiation [9]. So it is reasonable to consider that the shrinking loops under electron irradiation are of vacancy type.

The formation of such kind of vacancy loops could not be observed in the case of helium ion implantation, where only the growing of interstitial type loops could be found by electron irradiation even at 770 K (Fig. 3). The difference between hydrogen and helium may be explained by their different trapping site near vacancy (Fig. 4).

To explain the formation mechanism of such vacancy type loops, it is assumed that hydrogen was trapped at an off-site of the vacancy, for example at a octahedral or tetrahedral interstitial site rather than vacancy center. The complex composed of a hydrogen atom and a vacancy looks like a dumb-bell, and would aggregate together along a plane to form a vacancy type loop due to the anisotropy of the dumb- bell. It was reported that the hydrogen in iron is trapped at the site with a distance of 0.54 au from the vacancy center along $\langle 100\rangle$ direction [10]. On the other hand, a helium atom may be trapped just at the center of a vacancy. The complex of helium and vacancy would aggregate into a void instead of a loop.

The Burgess vector and habit planes of 10 vacancy-type loops (labeled by letter of A–J) were determined using inside–outside method of TEM experiment. Two kinds of vacancy loops $b = 1/2\langle 111\rangle$ and $b = \langle 100\rangle$ were found. The effect of alloy elements on the formation temperature of vacancy loops will be discussed.

From analysis of inside–outside image of loops by TEM (Fig. 5 and Table 1), it is known that there are two different vacancy type loops, one having Burgers vector $\mathbf{b} = 1/2\langle 111\rangle$ and loop plane $\mathbf{n} \approx \langle 011\rangle$, and the other $\mathbf{b} = \langle 100\rangle$, and $\mathbf{n} \approx \langle 112\rangle$, as shown in Table 2. The interstitial loops shown in Fig. 1a were proven to have Burgers vector $\mathbf{b} = 1/2\langle 111\rangle$ and loop plane $\mathbf{n} \approx \langle 211\rangle$.

It should be noted that among the 10 analyzed loops, only one has a Burgers vector $1/2\langle 111\rangle$, but the 9 loops have Burgers vector $\langle 100\rangle$. However, in the case of interstitial-type loops in bcc iron, many loops have $1/2\langle 111\rangle$, but fewer with $\langle 100\rangle$ [11].

The formation temperature of vacancy loops would change by adding alloying element in iron. For example, vacancy type loops

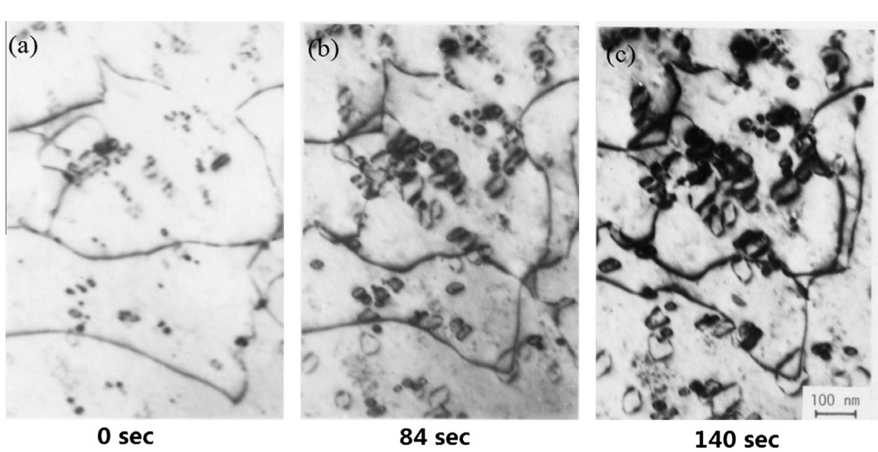

Fig. 1. Change of loops during electron irradiation at 670 K in pure iron which was pre-implanted by hydrogen ions at R.T.