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Journal of Nuclear Materials 283–287 (2000) 1134–1138

journal of nuclear materials

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Microstructure evolution in tungsten during low-energy helium ion irradiation

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Abstract

In situ transmission electron microscopy (TEM) study was performed to investigate the microstructural changes in tungsten during low-energy He^+ ion irradiations in an electron microscope linked with an ion accelerator. The irradiations were carried out with 8 and 0.25 keV He^+ ions at 293, 873 and 1073 K. In the case of the 8 keV irradiation, irradiation-induced vacancies act as nucleation sites for dislocation loops and helium (He) bubbles. Accordingly, such defects were formed even at the higher temperatures. With increasing irradiation temperature, the growth rates of dislocation loops and He bubbles rise remarkably. Although no vacancies are produced during 0.25 keV irradiation, He platelets, interstitial loops and He bubbles were formed. Impurity atoms may act as trapping centers for He atoms, which form bubbles by ejecting W atoms from their lattice site.

1. Introduction

Plasma facing materials suffer strong bombardment from the plasma by particles such as hydrogen (H) isotopes and helium (He) with the energies ranging from 10 eV to several keV in addition to D–T neutrons. These energetic particles along with neutrons, induce irradiation damage in the plasma facing materials.

It was shown that in situ transmission electron microscope (TEM) observation under ion irradiation is a very useful technique to study mechanisms of damage accumulation. Microstructural evolution in Mo and W under H ion irradiation with energies comparable to the boundary plasma has been investigated by the authors [1,2]. It was shown that the irradiation effects of He are much stronger than H [3,4], but the mechanism of defect accumulation under He ion irradiation has not been seriously studied. In the present work, in situ TEM observation of W under irradiation of He ions with fusion relevant energies was carried out to study defect accumulation processes.

2. Experimental procedures

Material used in the present work was high purity (99.95%) powder metallurgy W containing 40 wppm Mo, 20 wppm Fe, 15 wppm C and 5 wppm O and N. After rolling to 0.1 mm thickness, and cutting into disks of 3 mm in diameter, they were annealed at 2273 K for 600 s in a vacuum of about 5×10^{-4} Pa. Pre-thinned samples for TEM observation were obtained by twin-jet electro-polishing the disks.

The in situ observation under He ion irradiation was conducted using a 200 kV transmission electron microscope equipped with a low energy ion accelerator. Details of the facility are described elsewhere [5]. Helium ions at 8 or 0.25 keV were irradiated with the specimens at 293, 873 and 1073 K. Microstructural evolution was recorded either on films or video.

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PII: S0022-3115(00)00289-0

3. Results

3.1. Microstructural evolution under 8 keV He ion irradiation

Typical microstructural evolution in W at 293 K under irradiation with 8 keV He ions is shown in Fig. 1. Interstitial type dislocation loops appeared at first and increased in density with increasing dose. The density saturated at a dose level of around 1.3×10^{19} ions/m^2, while the size of each loop continued to increase. At 4.3×10^{19} ions/m^2, the average size of the loops becomes about 5 nm, and the loops piled up as tangled dislocations. The saturation density is about six times higher than that for H ion irradiation at a comparable energy [2]. Fig. 2 shows the temperature dependence of loop formation. With increasing irradiation temperature the density of the loops dropped drastically, while the loop size increased. At 873 and 1073 K they grew rapidly and tangled with each other.

Fig. 3 shows micrographs taken at rather high dose with large s diffraction condition. Helium bubbles are observed at all temperatures in addition to dislocation loops. In the case of the 873 K irradiation, the bubbles are aligned along the traces of {1 1 0} matrix planes, this has been observed in bcc metals following high-dose irradiation with He at around $0.2T_m$ [6], where T_m is the melting temperature. In the case of irradiation at 1073 K, large bubbles above 20 nm in diameter are formed together with rather small bubbles of about 5 nm.

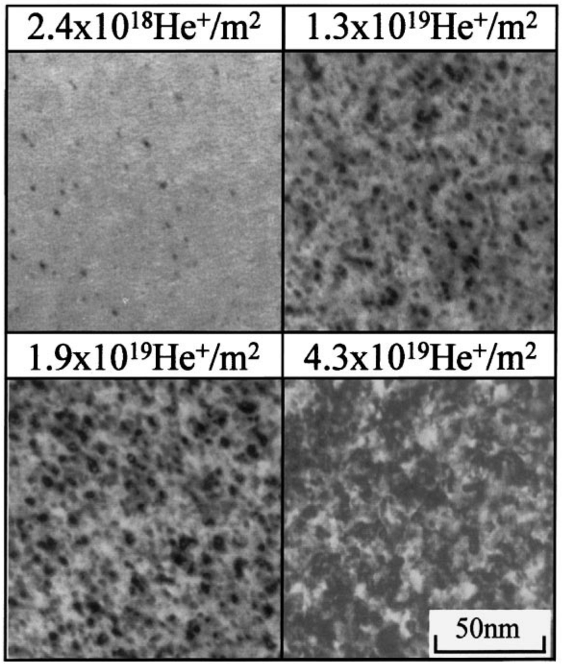

Fig. 1. Microstructural evolution of W at room temperature during irradiation with 8 keV He$^+$ ions.

3.2. Microstructural evolution under 0.25 keV He ion irradiation

Fig. 4 shows microstructural evolution at 293 K under irradiation with 0.25 keV He ions. Despite insufficient energy for knock-on damage, dense defects appeared suddenly around 1.4×10^{19} He$^+$/m^2. According to stereoscopic observation, the defects distributed in a region about 20 nm from the beam incident surface. The contrast of the defects is very weak at low dose in comparison with a dislocation loop, but it becomes stronger gradually above 3.0×10^{19} He$^+$/m^2. Defects at low dose could be clearly observed only under the Bragg condition. This fact indicates that they are not dislocation loops as observed in 8 keV He irradiation, but some other defect whose strain field is weak at the beginning but becomes stronger with dose. As discussed later, they are probably plane agglomerates of injected He (called

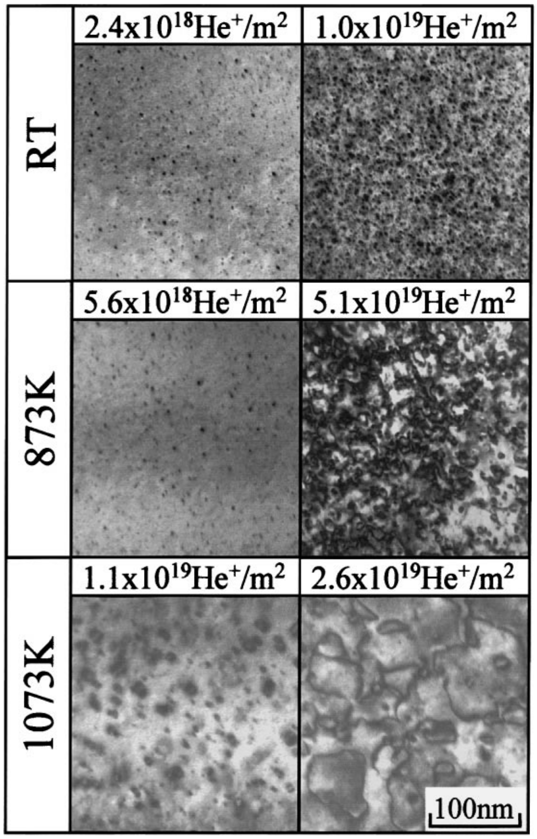

Fig. 2. Temperature dependence of dislocation loop formation during irradiation with 8 keV He$^+$ ions.

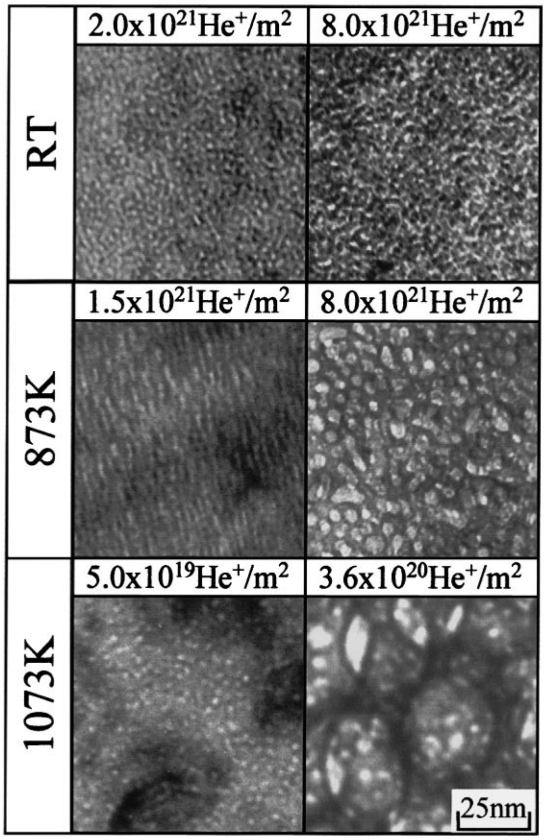

Fig. 3. Temperature dependence of bubble formation during irradiation with 8 keV He^+ ions.

here He platelets). As shown in Fig. 4(d) and demonstrated in serial video images in Fig. 5, a new defect cluster with strong contrast appears suddenly beside the He platelet above 3.0×10^{19} ions/m^2, and one or two more defect clusters are formed for each He platelet by prolonged irradiation. Image contrast of the He platelet becomes weaker after formation of a new cluster. The size of the new cluster is comparable to the original platelet. This phenomenon suggests the punching out of an interstitial loop from a platelet precipitation [7]. The punched out dislocation loops subsequently grow under irradiation. Hence, dislocation loops, which are usually formed as agglomerates of interstitials formed by knock-on damage, are formed even under non-displacement damage conditions. The formation of He platelets and dislocation loops occurs even at temperatures as high as 1073 K (see Fig. 6).

At higher dose, He bubbles were also formed at all examined temperatures as shown in Fig. 7. Formation of bubbles at the 0.25 keV irradiation is not much different from that at 8 keV, in spite of the large difference in ion energy. This indicates that the major factor controlling He bubble formation is not knock-on damage but injection of He.

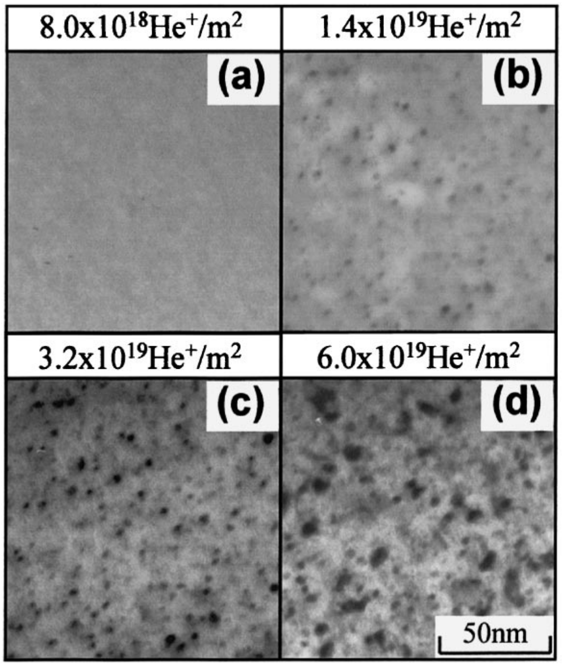

Fig. 4. Microstructural evolution of W at room temperature during irradiation with 0.25 keV He^+ ions.

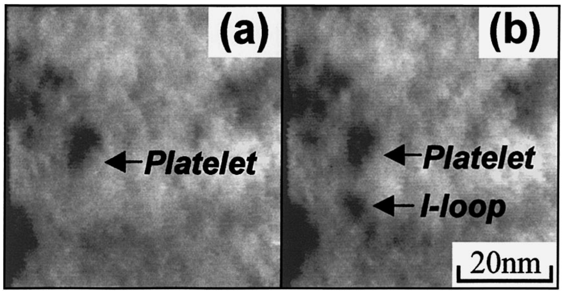

Fig. 5. Sequential micrographs of loop punching from He platelet taken from a video camera.

4. Discussion

4.1. Formation of interstitial loops under He irradiation

It is known that formation of interstitial loops under He ion irradiation is enhanced by the trapping of interstitials around He–vacancy complexes [3,8,9]. Details of the loop nucleation mechanism under He ion irradiation are discussed here within the content of this basic idea. Thermal He desorption spectrometry (TDS)

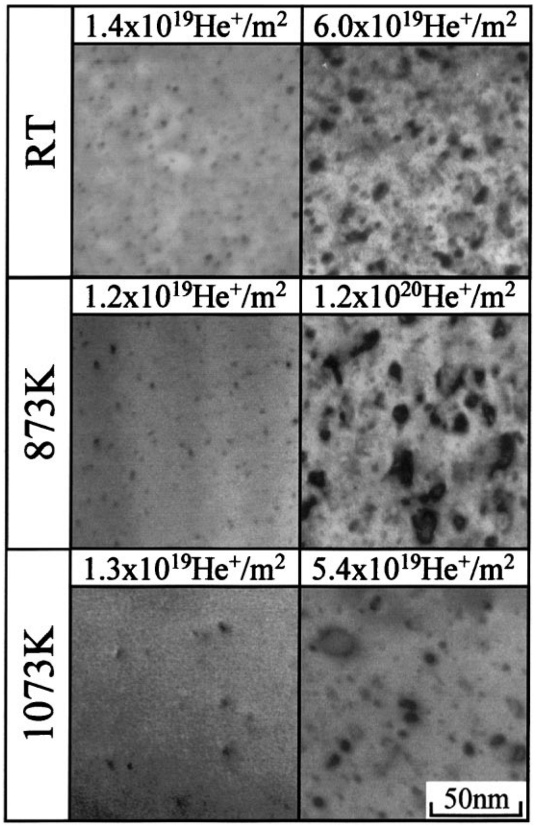

Fig. 6. Temperature dependence of platelet and dislocation loop formation during irradiation with 0.25 keV He^+ ions.

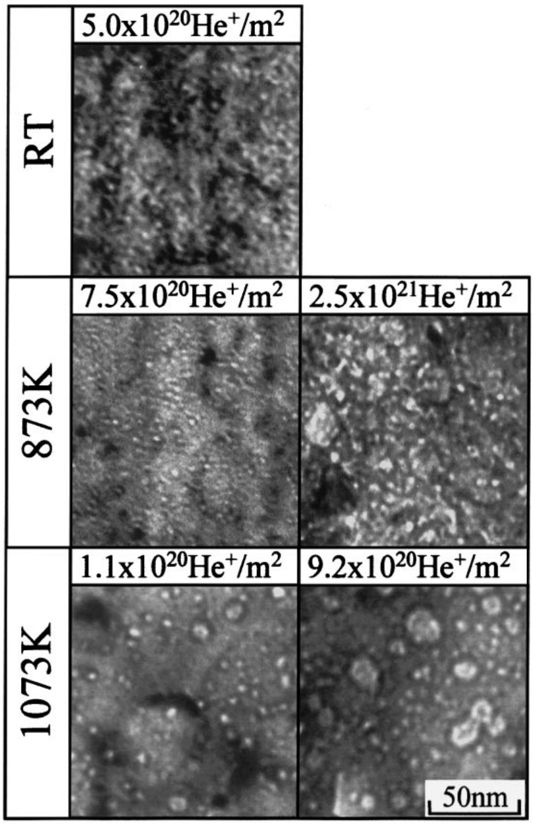

Fig. 7. Temperature dependence of bubble formation during irradiation with 0.25 keV He^+ ions.

experiments showed that injected He atoms with keV-range energies are trapped in radiation-induced vacancies and form He–vacancy complexes of various size, i.e., He_iV_1 $(i \leqq 6)$ due to very strong He–vacancy binding energy [10]. Here, the suffixes denote the number of He atoms or vacancies (V). He_iV_1 complexes with small i, however, may disappear by absorbing a mobile interstitial [11,12]. Some fraction of the He_iV_1 complexes, which reach a critical size ($i = 5$ or 6) mutate into a complex with two vacancies (He_iV_2) by ejecting an interstitial into the matrix [13]. By absorbing He atoms and further ejecting interstitials, large complexes He_iV_j $(i > 6, j \geqq 2)$ are formed. If the number of He (i) is large enough, the complex cannot absorb interstitials but may trap them around it. This trapping effect for interstitials and the formation of excess interstitials by the ejection may result in the enhancement of interstitial loop formation. Because of the strong stability of large He_iV_j complexes [11,14], interstitial loops are formed even at 1073 K. Formation of interstitial loops at such high temperature is a peculiar feature of He ion irradiation; in the case of H ion irradiation, for example, no loops are formed above 873 K [2].

4.2. He bubble formation under 8 keV He ion irradiation

In the case of the 8 keV He ion irradiation, where vacancies and interstitials are formed by knock-on damage, He bubbles are formed at high dose depending on irradiation temperature. At low temperatures, where vacancies have no thermal mobility, He bubbles can grow by ejecting interstitials due to a high He gas pressure in the bubbles (gas-driven growth).

At elevated temperatures, where vacancies are thermally mobile, radiation-induced vacancies enhance bubble formation. For example, the critical dose for visible bubble formation at 1073 K is about 1/50 of that at 293 K. By absorbing mobile vacancies, nucleation and growth of bubbles occurs (see Fig. 3).

4.3. Defect formation under 0.25 keV He irradiation

In situ TEM observation under 0.25 keV He ion irradiation showed that the defects that appear at low dose must not be dislocation loops because of their weak contrast. This feature of the image indicates that the strain field around the defects is weak. A possible structure of the defects is platelets of He atoms formed between W lattice planes, which were observed in Mo irradiated with low energy 0.15 keV He ions [15]. Image contrast of the defects becomes stronger when their size exceeds about 5 nm. This indicates that platelets become thicker and thicker and result in a stronger strain field around them. Finally, an interstitial type dislocation loop is punched out to reduce the increased strain field as shown in Fig. 5. This loop punching process is repeated two or three times, but stops at high dose where bubbles are formed. This indicates that absorption of He by bubbles is more favorable than forming He platelets.

Since no vacancies are produced by knock-on process under 0.25 keV He ion irradiation, an alternative mechanism for bubble formation is required. One possible mechanism is trapping of He by impurities. TDS experiments showed that substitutional impurities such as Ag and Cr strongly trap He atoms as well as vacancies [16]. If the number of trapped He atoms exceeds a critical value, the impurity atom or the nearby W atom is pushed out in a interstitial site and a $\mathrm{He}_i\mathrm{V}_1$ complex is formed. With increasing irradiation temperature, the probability of trapping He around impurity atoms decreases and this results in the reduction of bubble density at elevated temperatures as shown in Fig. 7. Impurities may also act as nucleation sites of He platelets.

Once $\mathrm{He}_i\mathrm{V}_1$ complexes are formed they can easily grow as He bubbles by continous absorption of He and ejection of interstitials as discussed before. According to a molecular dynamics calculation, interstitials ejected from a high pressure $\mathrm{He}_i\mathrm{V}_j$ complex, are bonded to the complex at room temperature [17]. At elevated temperature, however, they are released thermally from the complex and contribute to the growth of punched out loops (see Fig. 7).

5. Conclusions

Microstructural evolution in W under irradiation of 0.25 keV or 8 keV He ion at 293, 873 and 1073 K was observed in situ by TEM. In the case of the 8 keV He ion irradiation, complexes of injected He and radiation-induced vacancies act as nuclei for He bubbles and they enhance nucleation of interstitial loops. These phenomena were widely observed at all temperatures examined, though the density and size of the defects strongly depend on temperature.

In the case of the 0.25 keV He ion irradiation, where knock-on damage does not occur, He platelets, interstitial loops and bubbles were formed. Impurity atoms may act as trapping centers for He atoms, which form bubbles by ejecting W atoms from their lattice sites. Formation of He platelets leads to nucleation of interstitial loops by punching-out process and the loops grow by absorbing interstitials ejected from bubbles.

The present results indicate that the plasma facing materials in D–T burning devices may suffer serious radiation damage by the bombardment of He ions from the plasma even at elevated temperatures and also even when the particle energy is below the threshold for knock-on damage.

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