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Observation of interstitial loops in He^{+} irradiated W by conductive atomic force microscopy

Qi Yang,[a,b] Hongyu Fan,[b] Weiyuan Ni,[b] Lu Liu,[b] Tobias Berthold,[c] Günther Benstetter,[c] Dongping Liu[a,b,d,*] and Younian Wang[a,*]

[a] *School of Physics and Optoelectronic Technology, Dalian University of Technology, Dalian 116024, People's Republic of China*
[b] *School of Physics and Materials Engineering, Dalian Nationalities University, Dalian 116600, People's Republic of China*
[c] *Faculty of Electrical Engineering and Media Technology, University of Applied Sciences Deggendorf, Deggendorf 94469, Germany*
[d] *Fujian Key Laboratory for Plasma and Magnetic Resonance, Department of Electronic Science, Aeronautics, School of Physics and Mechanical & Electrical Engineering, Xiamen University, Xiamen 361005, People's Republic of China*

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Abstract—Polycrystalline tungsten (W) has been irradiated with a low-energy (30–830 eV) He^{+} beam at the W surface temperature of 523–1073 K up to a fluence of $1.0 \times 10^{25}/m^2$. Measurements by non-destructive conductive atomic force microscopy show the existence of nanometer-sized interstitial loops in He^{+}-implanted layer. The size and distribution of interstitial loops are significantly affected by He^{+} energy and fluence, and W surface temperature. The distribution of interstitial loops becomes orientated in one certain direction after being irradiated at a relatively high fluence or W surface temperature. The cascading slipping of W atoms along one certain dense-packed face has been proposed to explain the ordered arrangement of interstitial loops at elevated temperature. Analysis indicates that the continuous growth of unstable nanometer-sized interstitial loops can result in the surface exfoliation of W materials.
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1. Introduction

Due to its low sputtering yield, high thermal conductivity, and low hydrogen solubility, tungsten (W) has been proposed to be one of the best candidates for plasma facing materials in fusion reactors (PFMs) [1–3]. PFMs are subject to the high heat load and the bombardments by low-energy H^{+}/He^{+} ions and 14 MeV neutrons. It has been reported that He^{+} bombardments may lead to the formation of nanometer-sized defects including vacancies, interstitials, and dislocation loops [4–10]. Radiation induced defects in W materials are of great interest since they can act as the nucleus for He bubble formation and develop into nanometer-sized bubbles in the implanted layers of W materials. The radiation induced defects can also result in an increase in yield strength and surface embrittlement, and determine the performance of irradiated W materials in nuclear reactor environment [11]. The radiation induced defects and microstructural evolution of W materials exposed to low-energy and high-flux He ions still remain unclear. It has been proposed that He-vacancy complexes should be formed by continuous absorption of He and ejection of interstitials [12]. Impurity atoms could act as trapping centers for He atoms, which formed bubbles by ejecting W atoms from their lattice sites. Nishijima et al. proposed that He atoms diffusing in W can be easily trapped in the vacancy and become nano-scale defects or bubbles [13]. An increase in the internal pressure of these trapping sites resulted in the mutation of crystal lattices. Yoshida et al. proposed that the nucleation of interstitial loops in the stress field of a He-vacancy complex can greatly contribute to the formation of defects or He bubbles, finally resulting in the nanostructured W surfaces [14].

Recently, our conductive atomic force microscope (CAFM) measurements have shown that ordered and nanometer-sized defects are formed after the W specimens are irradiated with low-energy (220 eV) He^{+} at $>3.0 \times 10^{24}/m^2$ [7]. The CAFM measurement can provide a direct comparison between the sizes and distribution of nanostructured defects, and conductivity and surface features of irradiated W materials. In this study, CAFM has been utilized to detect the nanometer-sized interstitial loops in He^{+}-irradiated polycrystalline W materials. The effect of

* Corresponding authors at: School of Physics and Optoelectronic Technology, Dalian University of Technology, Dalian 116024, People's Republic of China (D. Liu). Tel.: +86 411 87508902; fax: +86 411 87656331; e-mail addresses: dongping.liu@dlnu.edu.cn; ynwang@dlut.edu.cn

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He^+ fluence and energy, and W surface temperature on the size, density, and distribution of interstitial loops are presented, and their microscopic evolution is discussed based on these experimental measurements.

2. Experimental procedures

Polycrystalline W (Honglu Corporation, China) with a purity of 99.99 at.% was used as the specimen. The specimens were cut into pieces with dimension of $10 \times 10 \times 2$ mm. The surfaces of specimens were mechanically mirror-polished to a surface RMS roughness of <0.1 µm. W specimens were annealed at 1273 ± 20 K for 2 h in vacuum with a background pressure of 10^{-5} Pa to relieve internal stresses and reduce the large concentration of nanometer-sized defects. Then, specimens were irradiated with He^+ beam in MIES [7]. The incident He^+ energy was controlled by negatively biasing the specimen up to −800 V. He^+ energy was typically varied from 30 to 830 V when considering the plasma potential of 30 V at a RF power of 350 W. The ion beam at a constant He^+ flux of $\sim 10^{21}/m^2$ s bombarded the specimen at a normal incidence. A variable-power semiconductor laser was used to heat the backside of one W specimen, and the surface temperature of the W specimen was monitored with an infrared STL-150B pyrometer fixed outside of the vacuum chamber. The surface temperature of W specimen was adjustable typically in the range of 523–1073 K. The detailed irradiation conditions for W specimens are listed in Table 1, where He^+ fluence was varied from 3.0×10^{22} to $1.0 \times 10^{25}/m^2$. He concentration and implantation depth can be estimated using the program TRIM 2008.04 [15] with a displacement threshold energy of $E_d = 90$ eV [16]. After He^+ irradiation, scanning electron microscope (SEM, Hitachi S-4800) was utilized to observe the surface microstructures of W specimens.

Previously, CAFM (Veeco DI 3100) has been used to detect the nanometer-sized defects of He^+-irradiated hydrocarbon films [17], single-crystalline 6H-SiC [18] and polycrystalline W materials [7]. Compared to TEM analysis, the CAFM method is relatively simple, and it does not make any damage to the irradiated materials. From the CAFM measurement, one can simultaneously obtain the surface topography and current emission images of irradiated materials, which plays a crucial role in understanding the microstructural evolution of irradiated fusion materials. In the CAFM method, one laser system is used to keep the constant deflection of the PtIr-coated tip in contact with the measured specimen. When the tip is scanned across the specimen surface in the contact mode, the surface topography is obtained. To simultaneously obtain the current image of irradiated W materials, a constant voltage (V_{tip}) is applied between the PtIr-coated tip and the W specimen, as shown in Fig. 1. The tip electron emission through the W specimen is strongly dependent on the V_{tip} and the total resistance of detection circuit (R_{total}). R_{total} mainly consists of the resistance from the nanometer-sized tip (R_{tip}), the contact resistance from the nanotip-W interface (R_c), and the W resistance due to the electron emission through the nanometer-sized interface (R_M). R_{total} can be expressed as

$$R_{total} = R_{tip} + R_c + R_M \tag{1}$$

When considering the charges emit from nanometer-sized interface through W in a hemispherical shape (Fig. 1(a)), R_M can be estimated as

$$R_M = \int_{r_0}^{\infty} \frac{\rho_0 dr}{2\pi r^2} = \frac{\rho_0}{2\pi r_0} \tag{2}$$

where r_0 is the radius of the nanometer-sized interface, and ρ_0 the resistivity of W materials. r is the distance between any location in W and the center of nanometer-sized interface. When the nanometer-sized tip is scanned at the location of defects existing in the implantation layer of W materials, R_M can be expressed as

$$R_M = \frac{\rho_1}{2\pi r_0} - \frac{\rho_1}{2\pi r_1} + \frac{\rho_0}{2\pi r_1} \tag{3}$$

where r_1 and ρ_1 are the radius and resistivity of nanometer-sized defects, respectively, and $r_1 > r_0$. Combining Eqs. (1) and (3), one can obtain

$$R_{total} = R_{tip} + R_C + \frac{\rho_1}{2\pi r_0} - \frac{\rho_1}{2\pi r_1} + \frac{\rho_0}{2\pi r_1} \tag{4}$$

At a given V_{tip}, the determined current through the tip is inversely proportional to R_{total}. Thus, if one defect contains a high density of W atoms, i.e. $\rho_1 < \rho_0$, the determined current is improved when the tip is scanned at the location of the defect. Since the interstitial loops contain a high density of W atoms, CAFM can be used to detect the electron emission from the interstitial loops formed in W. Closed loops can be observed from the current images of irradiated W specimens.

Nanoindentation testing system (Hysitron, TS75) combined with the CAFM system is applied to observe the physical properties of He^+-implanted layer of the W specimen. A pyramidal diamond tip with a tip curvature radius of 50 nm is used to generate the nanometer-sized impressions. The loading force of 5000 µN applied to the diamond tip is normal to the surface of the irradiated W specimen. After indentation, CAFM is used to image the nanostructured impressions by using a PtIr tip.

3. Results

Fig. 2 shows the SEM images of W specimens irradiated at the He^+ fluences of (a) $3.0 \times 10^{22}/m^2$, (b) $3.0 \times 10^{23}/m^2$, (c) $3.0 \times 10^{24}/m^2$, and (d) $1.0 \times 10^{25}/m^2$. The He^+ energy and the irradiation temperature are 130 eV and 873 K, respectively. No obvious change in surface microstructure can be observed for the W specimens irradiated at the He^+ fluences of $3.0 \times 10^{22}/m^2$ and $3.0 \times 10^{23}/m^2$. The W specimen irradiated at the He^+ fluence of $3.0 \times 10^{24}/m^2$ exhibits wavy-like surface structures due to the He^+ irradiation at a higher fluence [7,19]. The wavy-like structure appears to be thin strips of material orientated in a certain direction. When He^+ fluence is further increased to $1.0 \times 10^{25}/m^2$, surface swelling or He bubbles with their average sizes of about 2–5 µm are formed. Local surface exfoliation can be observed when the He bubble collapses presumably due to the over-high He pressure. Previously, the surface exfoliation of W materials has been observed above $2 \times 10^{22}/m^2$ after being bombarded with 3-MeV He^+ at 823 K [20]. However, there have been few reports about the surface exfoliation occurring due to low-energy He^+ irradiation. Our SEM measurements indicate that

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