High-flux He⁺ irradiation effects on surface damages of tungsten under ITER relevant conditions

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Abstract

A large-power inductively coupled plasma source was designed to perform the continuous helium ions (He⁺) irradiations of polycrystalline tungsten (W) under International Thermonuclear Experimental Reactor (ITER) relevant conditions. He⁺ irradiations were performed at He⁺ fluxes of 2.3×10^{21} - $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$ and He⁺ energies of 12 – 220 eV. Surface damages and microstructures of irradiated W were observed by scanning electron microscopy. This study showed the growth of nano-fuzzes with their lengths of 1.3 - 2.0 μm at He⁺ energies of >70 eV or He⁺ fluxes of $>1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$. Nanometer-sized defects or columnar microstructures were formed in W surface layer due to low-energy He⁺ irradiations at an elevated temperature (>1300 K). The diffusion and coalescence of He atoms in W surface layers led to the growth and structures of nano-fuzzes. This study indicated that a reduction of He⁺ energy below 12-30 eV may greatly decrease the surface damage of tungsten diverter in the fusion reactor.

1. Introduction

Currently, W has been selected as one of the best plasma-facing materials for ITER due to its low hydrogen solubility, low sputtering yield, and good thermal properties. The W diverter of fusion reactor will suffer from a large flux ($>10^{22}$ - $10^{24}/\text{m}^2 \cdot \text{s}$) of particle bombardments by low-energy (tens of eV to hundreds of eV) helium (He) and hydrogen ions [1, 2]. However, the bombardments of W by the high-flux and low-energy He ions lead to the serious irradiation damages, such as the formation of voids, wave-like nanostructures, or nano-fuzzes on the surface [3-6]. The enhanced erosion and dust formation of W nano-fuzzes can have fatal influence on the stability of fusion plasmas in the fusion reactor [7].

The structural changes of W surfaces are obviously dependent on the incident He⁺ energy, W surface temperature and He⁺ fluence [3-6]. Nanostructured surfaces are often formed when W materials are irradiated at the surface temperature (T) of >1000 K and the incident He⁺ energy of >20 eV [8]. Nanometer-sized defects, including vacancies, interstitials, and dislocation loops can be formed at this early stage during He⁺ irradiation of W materials [9-11]. Radiation induced defects in W materials are of great interest since they can act as the nucleus for He bubble formation, and finally develop into nano-fuzzes in the implanted layers of W materials. Although the surface change of W materials exposed to the low-energy He ions has been widely studied, the influence of He⁺ flux and energy on the

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micro-structural evolution of W materials still needs to be studied. Up to now, there have been few reports about the low-energy He⁺ irradiation of W materials performed at various He⁺ fluxes and energies [8]. It is relatively difficult to control the He⁺ flux and energy among the previous irradiation techniques. A change in the He⁺ flux and energy is of great interest since they play a crucial role in controlling the W surface microstructure during He⁺ irradiation.

In this study, a high-density ($\sim 10^{18}$ /m³) plasma irradiation system has been used to efficiently control He⁺ flux and energy, and perform He⁺ irradiation of polycrystalline W materials under ITER relevant conditions. The surface damages of irradiated W materials have been observed by scanning electron microscopy, and the influences of He⁺ flux and energy on the surface damages of W materials have been reported.

2. Experimental procedure

Polycrystalline W specimens (purity: 99.99%, Honglu Corporation, China) with a dimension of 10×10×2 mm were used for large-flux He⁺ irradiations. W surfaces were mechanically mirror-polished to a surface roughness of <0.1 μm. After polishing, W specimens were annealed at 1373 K for 2 h in vacuum with a background pressure of 10⁻⁵ Pa to relieve internal stress and reduce the large concentration of nanometer-sized defects.

The large-power inductively coupled plasma (ICP) irradiation system built in our lab has been used to irradiate W specimens [12], as schematically illustrated in Fig. 1. In this system, the stable and large-power ($P_{\max}=10$ kW) ICP plasmas were generated inside the quartz tube (10 cm in diameter, 18 cm in length) by inductively coupling of 2 megahertz (MHz) radio frequency (RF) to the water-cooled RF coil. The quartz tube was subject to very high thermal loads during the operation of large-power RF plasmas. A water-cooled stainless-steel Faraday shield was utilized to protect the quartz tube from the heat load of the plasma. The permanent magnets were installed on the bottom of the quartz tube to create a magnetic field for the enhancement of the plasma confinement. The molecular pump was utilized to pump the main vacuum chamber, and the base pressure was 1.0×10^{-4} Pa. The W specimen was placed on a specially designed holder into the high-density RF plasma. Specimen temperature was measured with an infrared STL200-A620 pyrometer. The infrared pyrometer was fixed outside of the plasma source in the ambient air. The infrared pyrometer collected a portion of the thermal radiation emitted by the W sample.

During He⁺ irradiation, He pressure in the plasma source remained constant at 30 Pa. The detailed irradiation conditions were listed in Table 1. Increasing RF power from 1.5 to 8.5 kW led to an increase in the electron density from 3.0×10^{17} to 2.8×10^{18} /m³ (Table I). The plasma potential did not show the dependence on the RF power ranging from 1.5 to 8.5 kW. The negative bias voltage ranging from -10 to -200 V was applied to the W specimen. This results in an incident He⁺ energy of 30-220 eV when taking account the plasma potential of 20 V measured by a Langmuir probe in this lab. When W specimen was floating, the energy of He⁺ ions may be expressed as [13]

$$E = kT_e \ln \left(\frac{m_i}{2\pi m_e} \right)^{1/2}$$

where E was He⁺ energy, k Boltzmann constant, and T_e electron temperature. m_i and m_e were the mass of ions and electrons, respectively. This calculation led

to $E=12$ eV. Table I shows that W surface temperature is significantly affected by the RF power and the negative bias. When RF power varied from 1.5 to 8.5 kW, He^+ flux increased from 2.3×10^{21} to $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$, leading to an obvious increase in W surface temperature from 960 to 1560 K. When the bias varied from -10 to -200 V, W surface temperature significantly increased from 1350 to 1560 K. In this study, all He^+ irradiations of polycrystalline W were performed at the He^+ fluence of $1.0 \times 10^{26}/\text{m}^2$.

After He^+ irradiation, scanning electron microscopy (SEM) (Hitachi S-4800) was utilized to observe the surface microstructures of W specimens. The SEM measurements were also carried out on specimen fracture sections that were produced by flexing to fracture specimens. The resolution limit of SEM is about 10 nm.

3. Results

3.1 Effect of He^+ flux on microstructures

Figure 2 shows the SEM images of W specimens irradiated at He^+ fluxes of (a) $2.3 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (b) $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (c) $7.7 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (d) $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$, (e) $1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$ and (f) $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. Their surface temperatures are (a) 960 K, (b) 1130 K, (c) 1210 K, (d) 1350 K, (e) 1480 K and (f) 1560 K, respectively. The surface microstructures are strongly dependent on the He^+ flux ranging from 2.3×10^{21} to $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. After W specimens are irradiated at the fluxes of 2.3×10^{21} and $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$, no obvious change in their surface microstructures (SEM micrographs) is formed, as shown in Fig. 2(a) and (b). However, M. Miyamoto et al. have proved the existence of nano-scale helium bubbles in W irradiated under the similar conditions through TEM observation [14]. This could be attributed to the difference in the detection methods. He^+ irradiation at the fluxes of 7.7×10^{21} and $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$ results in nanometer-sized protrusions at W surfaces (Fig. 2(c) and (d)). The formation of nanometer-sized structures due to He^+ irradiation has been reported previously [15-17]. It has been widely accepted that nanometer-sized defects or He bubbles are formed due to the diffusion and coalescence of He atoms into vacancies, dislocations, or defects at an elevated temperature [15, 16]. Such surface changes have been proposed to result from the growth of nanometer-sized He bubbles [15-17, 12]. When He^+ flux increases to $>1.3 \times 10^{22}$, plenty of fiberform nano-fuzzes are formed at W surfaces, as shown in Figs. 2(e) and (f). The dependence of surface microstructures on He^+ flux ranging from 2.3×10^{21} to $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$ can be attributed to an increase in W surface temperature. The diffusion and coalescence processes of He atoms in the surface layer can be enhanced at an increasing temperature. The formation of fiberform nano-fuzzes can be ascribed to the surface growth and swelling of nanometer-sized He bubbles [18].

Figure 3 shows the cross-sectional SEM images of W specimens irradiated at He^+ fluxes of (a) $2.3 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (b) $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (c) $7.7 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (d) $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$, (e) $1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$, and (f) $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. The cross-sectional measurements of W specimens show that surface microstructures are significantly affected by He^+ flux. No He^+ irradiation damage is observed for W specimens irradiated at He^+ fluxes of 2.3×10^{21} and $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$ (Fig. 3(a) and (b)). Figure 3 (c) and (d) show that columnar nanostructures are formed due to He^+ irradiations at He^+ fluxes of 7.7×10^{21} and $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$. These columnar nanostructures are almost perpendicular to W surfaces. Diffusion and coalescence of trapped He atoms prefer to occur along one certain face of W surface layer [12], and the diffusion depth of

trapped He atoms is affected by He^+ energy and surface temperature. The coalescence of trapped He atoms results in an over-high He pressure in the surface layer, which is responsible for the formation of columnar microstructures after the fractures of irradiated specimens. Our cross-sectional SEM measurements of irradiated W specimens indicate that the diffusion depth of trapped He atoms can be larger than 1 – 2 μm . Figure 3(e) and (f) show that the length of fiberform nano-fuzzes is 1.7 – 2.0 μm long after W specimens are irradiated at He^+ fluxes of 1.3×10^{22} and $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. However, plenty of nanometer-sized defects or bubbles are formed at the bottom of nano-fuzzes. This indicates that the formation of nano-fuzzes can result from the swelling of nanometer-sized bubbles in the surface layer.

3.2 Effect of He^+ energy on microstructures

The surface microstructures of W specimens irradiated at various He^+ energies are shown in Figs. 4. The surface microstructures are strongly dependent on the He^+ energy ranging from 12 to 220 eV. Figures 4(a) and (b) show that nanometer-sized structures are formed due to He^+ irradiation at the energies of 12 and 30 eV. The formation of nanometer-sized structures can result from the surface swelling of He bubbles, as described previously. Plenty of nano-fuzzes are formed when He^+ energy varies from 70 to 220 eV (Figs. 4(c), 4(d) and 2(f)). The growth of nano-fuzzes can be ascribed to an increase in He^+ energy and surface temperature. Increasing He^+ energy can lead to an increase in the depth of He implantation into W surface layer.

Figure 5 shows the cross-sectional SEM views of W specimens irradiated at He^+ energies of (a) 12 eV, (b) 30 eV, (c) 70 eV, and (d) 120 eV. Their surface temperatures are (a) 1350 K, (b) 1360 K, (c) 1430 K, and (d) 1490 K, respectively. The cross-sectional SEM observations show that the microstructures and thickness of W surface layer damaged are significantly affected by He^+ energy. After W specimens are irradiated at He^+ energies of 12 and 30 eV, the thickness of damaged surface layer is typically lower than 200 nm (Fig. 5(a) and (b)). Figures 5(c), 5(d), and 3(f) show that plenty of nano-fuzzes are formed at He^+ energies of >70 eV, which shows the consistence with SEM measurements in Fig. 4. The thickness of nano-fuzzes obviously increases from 1.3 to 2.0 μm when He^+ energy varies from 70 to 220 eV. He^+ irradiation at the energy of 70 eV results in the cross-linking nano-fuzzes at W surface, as shown in Fig. 5(c). When He^+ energy is larger than 120 eV, nano-fuzzes prefer to grow vertically upward, resulting in tree-like nano-fuzzes (Figs. 3(f) and 5(d)). Columnar nanostructures are clearly visible below tree-like nano-fuzzes (Fig. 5(d)), indicating the diffusion and coalescence along one certain crystal face lead to the distortion of crystal lattice. The growth of tree-like nano-fuzzes can be related to the formation of columnar nanostructures in the surface layer.

4. Discussion

The changes in W surface microstructures under high-flux He^+ irradiation have been described previously [12,18,19]. The surface damage processes are diffusion-controlled, and strongly influenced by surface temperature [19]. Under a rather high surface temperature (>1000 K) condition, He nanobubbles rapidly grow by thermally migrating and coalescing with each other, and push up the surface, leading to the formation of nano-fuzzes [18]. In our previous study, it was found that He atoms preferred to diffuse and cluster along one certain crystal face at an elevated temperature, leading to surface swelling of He-enriched strips [12]. Helium atoms are energetically favorable to cluster together in a close-packed arrangement between

(110) planes forming helium monolayer structure [20]. Charlotte S. Becquart and Christophe Domain have proved that He atoms can be trapped by other He atoms and can explain the formation of He blisters close to the surface of He implanted tungsten [21]. Nano-fuzzes were formed during the swelling, accompanied by the generation of nanostructured trenches. Nanostructured fuzzes at W surfaces can exhibit an orientation dependence [12, 19, 22].

Our current study indicates that surface microstructures of polycrystalline W specimens irradiated under ITER relevant conditions are greatly dependent on W surface temperature and He^+ energy. The effects of temperature and He^+ energy on microstructural evolution can be schematically illustrated in Figs. 6 and 7. At a relatively low irradiation temperature (<1000-1300 K), implanted He atoms diffuse in the shallow surface layer, and coalesce into He nanobubbles, as shown in Fig. 6. When the energy of He^+ ions is lower than the threshold (E_d) for atomic displacements, the formation process of surface microstructures (Fig. 4(a) and (b)) is due to the diffusion and coalescence of He atoms in the W sub-surface layer. Nanometer-sized structures are formed due to surface burst and swelling of He bubbles. Our previous measurements by conductive atomic force microscopy show that He-enriched strips are formed due to low-energy He^+ irradiation [12]. He nanobubbles tend to be formed along one certain face (Fig. 6(a)). Our calculation predicts that the formation of He-enriched strips is due to He clustering between close-packed {101} planes [12]. In this case, the growth of He-enriched strips at W surface shows the dependence on the W surface orientation. With He accumulation, He nanobubbles grow and coalesce into large-sized bubbles, as shown in Fig. 6(a). The surface swelling of He bubbles leads to the formation of nano-fuzzes at W surface (Fig. 6(b)) [18, 12], and their growth is dependent on the W surface orientation. The formation of cross-linking (coral-like) nano-fuzzes is due to the continuous growth of nano-fuzzes, as shown in Fig. 6(c).

Figure 7 shows the growth process of tree-like nano-fuzzes at a relatively high surface temperature (>1300-1600 K). Due to thermal diffusion, implanted He atoms penetrate deeply into the surface layer. Nanometer-sized He bubbles grow vertically along one certain face, as shown in Fig. 7(a). Due to an increase in the inner pressure of He bubbles, the formation of large-sized He bubbles at the W near-surface layer seriously leads to the distortion of W crystal between He bubbles. The columnar microstructures are formed during the growth of He bubbles in the near-surface layer. They develop into the tree-like nano-fuzzes when He bubbles push up the surface and collapse during the growth of He bubbles (Fig. 7(b)). An increase in He^+ energy (70-220 eV) can lead to the atomic displacements so that He atoms can diffuse more deeply along the W surface layer. The microstructure evolutions of W surface layer damaged are clearly observed from the cross-sectional SEM measurements (Fig. 5(c) and (d)). Meanwhile, the thickness of nano-fuzzes increases from 1.3 to 2.0 μm . The tree-like nano-fuzzes are perpendicular to W surface. With He accumulation, the tree-like nano-fuzzes grow vertically at W surface (Fig. 7(c)). The growth of nano-fuzzes decreases the energy and the flux of He^+ ions bombarding the surface. He diffusion and coalescence along the W surface layer are reduced, which slows the growth of He bubbles in W surface layer. A reduction in the size of He bubbles stunts the growth of tree-like nano-fuzzes at the W surface.

5. Conclusions

High-density ICP plasmas have been successfully used to generate large-flux

($2.3 \times 10^{21} - 1.6 \times 10^{22} / \text{m}^2 \cdot \text{s}$) and low-energy (12-220 eV) He^+ ions for the continuous irradiation of W specimens under ITER relevant conditions. The surface damages of polycrystalline W are greatly affected by He^+ energy and fluxes. Nano-fuzzes with their lengths of 1.3-2.0 μm are formed at He^+ energies of >70 eV or He^+ fluxes of $>1.3 \times 10^{22} / \text{m}^2 \cdot \text{s}$. The cross-sectional SEM observations of irradiated W indicate that the diffusion of implanted He atoms in W surface layer at an elevated temperature seriously leads to the distortion of W crystal, thus the formation of nanometer-sized defects or bubbles. Coral-like and tree-like nano-fuzzes can be formed due to the diffusion and coalescence of He atoms at an elevated temperature.

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Figure Captions

Figure 1. The schematic of large-power RF plasma irradiation system used to irradiate W specimens.

Figure 2. SEM images of W specimens irradiated at He⁺ fluxes of (a) $2.3 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (b) $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (c) $7.7 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (d) $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$, (e) $1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$, and (f) $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. He⁺ irradiations were performed at He⁺ energy of 220 eV and He⁺ fluence of $1.0 \times 10^{26}/\text{m}^2$. The specimen temperatures are (a) 960 K, (b) 1130 K, (c) 1210 K, (d) 1350 K, (e) 1480 K, and (f) 1560 K.

Figure 3. Cross-sectional SEM images of W specimens irradiated at He⁺ fluxes of (a) $2.3 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (b) $5.4 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (c) $7.7 \times 10^{21}/\text{m}^2 \cdot \text{s}$, (d) $1.0 \times 10^{22}/\text{m}^2 \cdot \text{s}$, (e) $1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$, and (f) $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$. He⁺ irradiations were performed at He⁺ energy of 220 eV and He⁺ fluence of $1.0 \times 10^{26}/\text{m}^2$. The specimen temperatures are (a) 960 K, (b) 1130 K, (c) 1210 K, (d) 1350 K, (e) 1480 K, and (f) 1560 K.

Figure 4. SEM images of W specimens irradiated at He⁺ energies of (a) 12 eV, (b) 30 eV, (c) 70 eV, and (d) 120 eV. He⁺ irradiations were performed at He⁺ flux of $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$ and He⁺ fluence of $1.0 \times 10^{26}/\text{m}^2$. Specimen temperatures are (a) 1350 K, (b) 1360 K, (c) 1430 K, and (d) 1490 K.

Figure 5. Cross-sectional SEM images of W specimens irradiated at He⁺ energies of (a) 12 eV, (b) 30 eV, (c) 70 eV, and (d) 120 eV. He⁺ irradiations were performed at He⁺ flux of $1.6 \times 10^{22}/\text{m}^2 \cdot \text{s}$ and He⁺ fluence of $1.0 \times 10^{26}/\text{m}^2$. Specimen temperatures are (a) 1350 K, (b) 1360 K, (c) 1430 K, and (d) 1490 K.

Figure 6. The formation process of coral-like nano-fuzzes due to the diffusion and coalescence of He atoms in the W sub-surface layer. At a relatively low irradiation temperature (<1100-1300 K), implanted He atoms diffuse in the shallow surface layer, and coalesce into He nanobubbles. (a) He nanobubbles tend to be formed along one certain face. With He accumulation, He nanobubbles grow and coalesce into large-sized bubbles. (b) The surface swelling of He bubbles leads to the formation of nano-fuzzes at W surface. (c) The continuous growth of nano-fuzzes results in the formation of cross-linking (coral-like) nano-fuzzes.

Figure 7. The formation process of tree-like nano-fuzzes due to the diffusion and coalescence of He atoms in W surface layer at a relatively high temperature (>1300-1500 K). Due to thermal diffusion, implanted He atoms penetrate deeply into the surface layer. (a) Nanometer-sized He bubbles grow vertically along one certain face. The columnar microstructures are formed during the growth of He bubbles in the near-surface layer. (b) The columnar microstructures develop into the tree-like nano-fuzzes when He bubbles push up the surface and collapse during the growth of He bubbles. (c) With He accumulation, the tree-like nano-fuzzes grow vertically at W surface. The growth of nano-fuzzes decreases the energy and the flux of He⁺ ions bombarding the surface. He diffusion and coalescence along the W surface layer are

reduced.

Table Captions

Table 1. Detailed irradiation conditions for polycrystalline W specimens irradiated in large-power RF irradiation system.

Table 1

Specimen	RF power (kW)	Plasma density (/m ³)	Bias (V)	He ⁺ energy (eV)	He ⁺ flux (ions/m ² • s)	Surface temperature (K)
11	1.5	3.0×10^{17}	-200	220	2.3×10^{21}	960
12	3.0	7.2×10^{17}	-200	220	5.4×10^{21}	1130
13	4.5	1.0×10^{18}	-200	220	7.7×10^{21}	1210
14	6.0	1.2×10^{18}	-200	220	1.0×10^{22}	1350
15	7.5	1.7×10^{18}	-200	220	1.3×10^{22}	1480
16 (25)	8.5	2.8×10^{18}	-200	220	1.6×10^{22}	1560
21	8.5	2.8×10^{18}	Floating	12	1.2×10^{22}	1350
22	8.5	2.8×10^{18}	-10	30	1.4×10^{22}	1360
23	8.5	2.8×10^{18}	-50	70	1.5×10^{22}	1430
24	8.5	2.8×10^{18}	-100	120	1.5×10^{22}	1490

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

(a)

(b)

(c)

Figure7

(a)

(b)

(c)