W nano-fuzzes: A metastable state formed due to large-flux He^+ irradiation at an elevated temperature

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Abstract

W nano-fuzzes have been formed due to the large-flux and low-energy (200eV) He^+ irradiation at W surface temperature of 1480°C. Microscopic evolution of W nano-fuzzes during annealing or low-energy (200eV) He^+ bombardments has been observed using scanning electron microscopy and thermal desorption spectroscopy. Our measurements show that both annealing and He^+ bombardments can significantly alter the structure of W nano-fuzzes. W nano-fuzzes are thermally unstable due to the He release during annealing, and they are easily sputtered during He^+ bombardments. The current study shows that W nano-fuzzes act as a metastable state during low-energy and large-flux He^+ irradiation at an elevated temperature.

Keywords: W nano-fuzzes, scanning electron microscopy, metastable state, He^+ irradiation

1. Introduction

He-induced W nano-fuzzes have been widely recognized as a potential drawback for the plasma facing W material in a D+T fusion reactor [1-3]. The growth of W nano-fuzzes does not strongly depend on the W grades, and He-induced nano-fuzzes have been observed under a wide range of plasma conditions [3,4]. Up to now, it has been widely accepted that W nano-fuzzes can be formed due to He^+ irradiation of polycrystalline W at the surface temperature of 900-1800°C [3-5]. To form the W nano-fuzzes, He^+ energy and fluence are $>20\text{eV}$ and $>10^{25}/\text{m}^2$, respectively. It has been believed that the formation of W nano-fuzzes is related to a He-trapping mechanism which is self driven [3]. Kajita proposed that the formation and growth of W nano-fuzzes can be attributed to the diffusion and coalescence of He nano-bubbles at an elevated temperature [6]. Our previous study indicates that the diffusion and coalescence of He atoms in W surface layer can control the growth and structure of nano-fuzzes [7]. Coral-like or tree-like nano-fuzzes can be formed due to low-energy He^+ irradiation, depending on the energy and flux of He ions.

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He-induced nano-fuzzes have low thermal conductivity and poor mechanical properties, which cause several concerns such as enhanced erosion and dust formation [5]. Sputtering yields (Y_F) of He-induced W nano-fuzzes have been measured as a function of Ar/He energy and W fuzz thickness [8,9]. A large fraction of sputtered W atoms can be deposited onto neighboring fuzz nanostructures, resulting in the significant reduction in Y_F . The thickness of W nano-fuzzes decreases with increasing He energy from 200 to 500eV. The effect of crystallographic orientation on W surface morphology was investigated in the linear plasma device by exposing the high-density He plasma to ITER grade W [10]. Because of different W areal density at crystal grains, W surface morphology shows the dependence on their crystal orientation. The deformation of W nano-fuzzes during annealing has been investigated by means of in-situ cross-sectional observation using transmission electron microscopy [11]. W nano-fuzzes were found to be unstable, and their surface shrinkage and coalescence occurred around 800-900°C.

The purpose of this current study is to explore the stability of W nano-fuzzes during annealing or He^+ bombardments, and analyze their growth process under large-flux He^+ irradiation at an elevated temperature. The effects of annealing temperature and He^+ bombardments on W nano-fuzzes are investigated by scanning electron microscopy (SEM), and the thermal stability is analyzed by thermal desorption spectroscopy (TDS). The growth process of W nano-fuzzes as a metastable state formed due to large-flux He^+ irradiation is discussed based on the experimental measurements.

2. Experimental procedures

Polycrystalline W (Honglu, Xiamen) with a size of 1cm×1cm×2mm was used as the specimen. To generate the W nano-fuzzes, the polished W specimen was irradiated with low-energy (200eV) and large-flux ($1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$) He^+ plasmas in our LP-MIES system described previously [7,12]. The radio frequency power of 7.5kW was injected into the inductively coupled plasma (ICP) source, resulting in W surface temperature of 1480°C. A negative bias of -180V was applied onto W specimen, which accelerated He^+ in the plasma sheath. The energy of He^+ was 200eV when taking into account the plasma potential of 20V. He^+ irradiations were performed at the fluence of 5.0×10^{25} ions/ m^2 , leading to the growth of W nano-fuzzes.

After the growth of W nano-fuzzes, W specimen was annealed or bombarded with He^+ plasma in our MIES system [13]. The chamber background pressure was $<3.0 \times 10^{-4}$ Pa. A variable-power semiconductor laser was used to heat the backside of W specimen, and W surface temperature is adjustable in the range of 25 to 1000°C. The temperature was measured with an infrared STL-150B pyrometer. Annealing temperature was changed at 200°C increments from room temperature (R.T.) to 1000°C. The specimen was heated to each temperature at a heating rate of 3.3°C/s. After the annealing for 60 minutes, the temperature was decreased to R.T., then SEM was utilized to observe the microstructural change in W fuzzes.

He^+ bombardments of nano-fuzzes at W surface were carried out at the energy of

200eV and the flux of $1.1 \times 10^{20}/\text{m}^2 \cdot \text{s}$. The He^+ bombardments were performed at the W temperature of R.T. or 600°C , and He^+ fluence was changed at $7.5 \times 10^{23}/\text{m}^2$ increments from $7.5 \times 10^{23}/\text{m}^2$ to $6.0 \times 10^{24}/\text{m}^2$. Then, SEM was utilized to observe the microstructural change in W fuzzes due to He^+ bombardments. Thermal desorption spectroscopy (TDS) was used to investigate the thermal desorption property of the fuzz W. In TDS analysis, W specimen was heated continuously up to 1000°C at a heating rate of $1^\circ\text{C}/\text{s}$.

3. Results

3.1 Effect of annealing temperature on W nano-fuzzes

To analyze the microstructural evolution of W nano-fuzzes during annealing, SEM observations were performed for the same locations of W specimen annealed at different temperatures. Figure 1 shows the effect of annealing temperature on the microstructure of W fuzzes. High-density W nano-fuzzes are formed due to low-energy (200eV) and large-flux ($1.3 \times 10^{22}/\text{m}^2 \cdot \text{s}$) He^+ irradiations, as shown in Fig. 1(a). Prior to He^+ bombardments, W nano-fuzzes are 15-30 nm in diameter. When the annealing temperature increases from 200°C to 800°C , the density of W nano-fuzzes slowly decreases, as shown in Fig. 1(b)-(e). Plenty of nanometer-sized W grains can be observed at the annealing temperature of 1000°C , as shown in Fig. (f). However, the diameter of W nano-fuzzes does not strongly depend on the annealing temperature (Fig. 2), and it remains almost constant when the annealing temperature varies from 200°C to 1000°C . The cross-sectional views of W nano-fuzzes at the same locations are also observed by SEM, as shown in Fig. 3. Our cross-sectional observation by SEM shows that the thickness of W fuzz layer is about $1\mu\text{m}$. Fine W fuzzes are formed inside the top layer of W fuzz layer. The thickness of W fuzz layer is greatly reduced due to the annealing at 200°C , and the thickness slowly decreases at an increasing annealing temperature. This indicates that W nano-fuzzes are thermally unstable, and they may develop into W grains at an increasing surface temperature. In-situ TEM observation of W nano-fuzzes formed using NAGDIS-II [11] shows that the thickness of fuzz layer decreased as the annealing temperature increases from 500°C to 1000°C . In particular, the structure of the fuzz changed drastically at 900°C .

3.2 Effect of He^+ bombardments on W nano-fuzzes

SEM observations were performed for the same locations of W specimen when the nano-fuzzes are bombarded at R.T. with He^+ fluences of 7.5×10^{23} - $6.0 \times 10^{24}/\text{m}^2$ (Fig. 4). Both the diameter and density of W nano-fuzzes do not show strong dependence on He^+ fluence ranging from 7.5×10^{23} to $3.0 \times 10^{24}/\text{m}^2$. When He^+ fluence is larger than $3.0 \times 10^{24}/\text{m}^2$, the diameter of W nano-fuzzes significantly increases, accompanied by a decrease in their density. This indicates that etching and re-deposition processes may result in the serious surface change.

Figure 5 shows the microscopic evolution of W nano-fuzzes bombarded at 600°C with the He⁺ fluences of 7.5×10^{23} - $6.0 \times 10^{24}/\text{m}^2$. Both the diameter and density of W nano-fuzzes are clearly dependent on the He⁺ fluence ranging from 7.5×10^{23} to $6.0 \times 10^{24}/\text{m}^2$. The density and length of W nano-fuzzes are reduced due to the etching process. Nanometer-sized holes are formed at the W surface. The formation of these holes can result from He diffusion and coalescence along the W surface layer. During He⁺ bombardments at 600°C, W re-deposition and He diffusion processes can lead to a slow increase in the diameter of W nano-fuzzes (Fig. 6). However, at a relatively low irradiation temperature, the diameter of W nano-fuzzes significantly increases from 50 to 100 nm when He⁺ fluence is larger than $3.75 \times 10^{24}/\text{m}^2$ (Fig. 6). This indicates that the irradiation temperature plays a crucial role in controlling the growth of W nano-fuzzes. Figure 7 exhibits the high-magnification SEM images of W nano-fuzzes bombarded with He⁺ at (a) R.T. and (b) 600°C. The fluence of He⁺ is $6.0 \times 10^{24}/\text{m}^2$. The structure of W nano-fuzzes is strongly dependent on W surface temperature. The W nano-fuzzes bombarded at R.T. are relatively smooth, compared to those at 600°C. Plenty of nanometer-sized holes are formed inside W nano-fuzzes, which is presumably due to the rapid He diffusion at a relatively high temperature.

3.3 TDS analysis

Figure 8 shows the thermal desorption spectrum of fuzz W obtained at a heating rate of 1°C/s. One strong desorption peak is formed at 815°C (Fig. 8(a)). The TDS spectrum has no clear peak in the low temperature range. Fig. 8(b) is the enlarged plot of Fig. 8(a) at low temperature of <400°C. Fig. 8(b) shows that He atoms are released from the W specimen at the surface temperature of <200°C. This shows the consistency with the annealing measurements, where fine W fuzzes inside the top layer disappear at the annealing temperature of 200°C. Release of He atoms from W nano-fuzzes results in the surface shrinkage of W nano-fuzzes. This indicates that the retention of He atoms in W surface layer can play a crucial role in forming W nano-fuzzes. The release of He atoms from W nano-fuzzes at <200°C can be due to the low activation energy of desorption reaction [11]. He atoms can get weakly trapped into trapping sites inside fine W fuzzes. W fuzzes on the bottom of W fuzz layer get thicker, and the activation energy can be greatly improved. The strong TDS peak at 815°C indicates that W fuzzes at the surface are completely destroyed.

4. Discussion

The growth process of W nano-fuzzes is well described by Kajita et al. [6, 11], where He bubbles migrate, coalesce, and grow at a relatively high surface temperature (>1100°C). The large bubbles push up the surface, leading to the formation of W nano-fuzzes. Our measurements show that W nano-fuzzes are thermally unstable, and the annealing at 200°C leads to the shrinkage of fine fuzzes at the surface. This indicates that the He pressure inside W fuzzes is extremely high due to He⁺ irradiation.

A further increase in the surface temperature ($>800-1000^{\circ}\text{C}$) results in their coalescence into W grains, accompanied by a strong He release from W nano-fuzzes. The He pressure inside W surface layer can be mainly determined by He^+ flux and W surface temperature. W surface temperature can control the He diffusion and their coalescence into He bubbles. He^+ irradiation at a high surface temperature results in the dynamic balance of He diffusion into W surface layer and their release from the surface layer. The fine fuzzes on the top of W fuzz layer are exposed to the stronger He^+ irradiation, compared to the W fuzzes on the bottom of W fuzz layer. The relatively high He pressure inside the fine fuzzes can cause them to be more unstable, as observed from our annealing measurements.

Two necessary conditions have to be fulfilled for forming W nano-fuzzes at the surface of polycrystalline W: (1) Large-flux and low-energy He^+ irradiation, and (2) a high W surface temperature ($>1100^{\circ}\text{C}$) [11]. Without He^+ irradiation, the dynamic balance can be destroyed, and W nano-fuzzes disappear at a high W surface temperature. The strong etching and re-deposition processes occur during He^+ bombardments, resulting in the surface shrinkage of W nano-fuzzes at a low surface temperature. Due to their weakly bonded sites, W nano-fuzzes containing He vacancies are easily sputtered during He^+ bombardments. Our measurements show that He^+ bombardments at a relatively high W surface temperature (600°C) do not significantly change the structure of W nano-fuzzes. This indicates that He diffusion into W fuzzes or surface layer plays a crucial role in forming the W nano-fuzzes. The measurements confirm that W nano-fuzzes as a metastable state are formed due to large-flux He^+ irradiation at a high surface temperature. However, the threshold of He^+ flux for forming W nano-fuzzes and the effect of surface temperature on the threshold still need to be investigated.

5. Conclusions

The effects of annealing temperature and He^+ bombardments on W nano-fuzzes have been investigated by SEM and TDS measurements. Both annealing temperature and He^+ bombardments at a relatively low temperature can significantly alter the structure of W nano-fuzzes, and finally result in their surface shrinkage. He^+ irradiation at a high surface temperature results in the dynamic balance of He diffusion into W surface layer and their release from the surface layer. W nano-fuzzes as a metastable state are formed due to large-flux He^+ irradiation at a high surface temperature.

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Figure caption

Figure 1. The effect of annealing temperature on the microstructure of W fuzzes. SEM observations were performed for the same location of W specimen.

Figure 2. The diameter of W nano-fuzzes as a function of the annealing temperature.

Figure 3. The cross-sectional views of W nano-fuzzes annealed at different temperature. The cross-sectional observations were performed for the same location of W specimen.

Figure 4. SEM images of W nano-fuzzes bombarded at room temperature (R.T.) with He^+ fluences of 7.5×10^{23} - $6.0 \times 10^{24}/\text{m}^2$. SEM observations were performed for the same location of W specimen.

Figure 5. SEM images of W nano-fuzzes bombarded at 600°C with He^+ fluences of 7.5×10^{23} - $6.0 \times 10^{24}/\text{m}^2$. SEM observations were performed for the same location of W specimen.

Figure 6. The diameter of W nano-fuzzes bombarded with He^+ at room temperature (R.T) and 600°C as a function of He^+ fluence.

Figure 7. High-magnification SEM images of W nano-fuzzes bombarded with He^+ at (a) room temperature (R.T.) and (b) 600°C . The fluence of He^+ is $6.0 \times 10^{24}/\text{m}^2$.

Figure 8. Thermal desorption spectrum of fuzz W obtained at a heating rate of $1^\circ\text{C}/\text{s}$. Fig. 8(b) is the enlarged plot of Fig. 8(a) at the low temperature of $<400^\circ\text{C}$.

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