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## W nano-fuzzes: A metastable state formed due to large-flux  $He<sup>+</sup>$ irradiation at an elevated temperature



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## highlights are the control of

 $\bullet$  W nano-fuzzes microscopic evolution during annealing or He $^+$  irradiated have been measured.

- W nano-fuzzes are thermally unstable due to He release during annealing.

 $\bullet$  He are released from the top layer of W fuzzes by annealing.

 $\bullet$  Metastable W nano-fuzzes are formed due to 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<sup>+</sup> irradiation at W surface temperature of 1480 °C. Microscopic evolution of W nano-fuzzes during annealing or low-energy (200 eV)  $He<sup>+</sup>$  bombardments has been observed using scanning electron microscopy and thermal desorption spectroscopy. Our measurements show that both annealing and  $He<sup>+</sup>$  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  $He<sup>+</sup>$  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.

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#### 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  $[1-3]$  $[1-3]$ . 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 [\[3,4\].](#page--1-0) 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 [\[3](#page--1-0)-[5\].](#page--1-0) To form the W nano-fuzzes, He<sup>+</sup> energy and fluence are >20eV and >10<sup>25</sup>/m<sup>2</sup>, respectively. It has been believed that the formation of W nano-fuzzes is related to a He-trapping mechanism which is self driven [\[3\]](#page--1-0). 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 [\[6\]](#page--1-0). 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 [\[7\]](#page--1-0). Coral-like or tree-like nano-fuzzes can be formed due to low-energy  $He<sup>+</sup>$  irradiation, depending on the energy and flux of He ions.

He-induced nano-fuzzes have low thermal conductivity and poor mechanical properties, which cause several concerns such as enhanced erosion and dust formation [\[5\].](#page--1-0) Sputtering yields  $(Y_F)$  of He-induced W nano-fuzzes have been measured as a function of Ar/He energy and W fuzz thickness [\[8,9\].](#page--1-0) 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 [\[10\].](#page--1-0) Because of different W areal density at crystal grains, W surface morphology shows the dependence on their crystal orientation. The deforma-\* Corresponding author. Tel.: +86 41187508902; fax: +86 41187656331. tion of W nano-fuzzes during annealing has been investigated by

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means of in-situ cross-sectional observation using transmission electron microscopy [\[11\]](#page--1-0). 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  $He<sup>+</sup>$  bombardments, and analyze their growth process under large-flux  $He<sup>+</sup>$  irradiation at an elevated temperature. The effects of annealing temperature and  $He<sup>+</sup>$  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  $He<sup>+</sup>$ irradiation is discussed based on the experimental measurements.

#### 2. Experimental procedures

Polycrystalline W (Honglu, Xiamen) with a size of 1 cm  $\times$  1 cm  $\times$  2 mm was used as the specimen. To generate the W nano-fuzzes, the polished W specimen was irradiated with lowenergy (200eV) and large-flux ( $1.3 \times 10^{22}$ /m<sup>2</sup>·s) He<sup>+</sup> plasmas in our LP-MIES system described previously [\[7,12\]](#page--1-0). The radio frequency power of 7.5 kW was injected into the inductively coupled plasma (ICP) source, resulting in W surface temperature of 1480 $^{\circ}$ C. A negative bias of  $-180$  V was applied onto W specimen, which accelerated He<sup>+</sup> in the plasma sheath. The energy of He<sup>+</sup> was 200eV when taking into account the plasma potential of 20 V. He<sup>+</sup> irradiations were performed at the fluence of  $5.0 \times 10^{25}$ ions/m<sup>2</sup>, leading to the growth of W nano-fuzzes.

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

 $He<sup>+</sup>$  bombardments of nano-fuzzes at W surface were carried out at the energy of 200eV and the flux of 1.1  $\times$  10<sup>20</sup>/m<sup>2</sup>·s. The He<sup>+</sup> bombardments were performed at the W temperature of R.T. or 600 °C, and He<sup>+</sup> fluence was changed at  $7.5 \times 10^{23}$ /m<sup>2</sup> increments from 7.5  $\times$  10<sup>23</sup>/m<sup>2</sup> to 6.0  $\times$  10<sup>24</sup>/m<sup>2</sup>. Then, SEM was utilized to observe the microstructural change in W fuzzes due to  $He<sup>+</sup>$  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 $\degree$ C at a heating rate of  $1 \degree C/s$ .

#### 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. Fig. 1 shows the effect of annealing temperature on the microstructure of W fuzzes. High-density W nano-fuzzes are formed due to lowenergy (200eV) and large-flux (1.3  $\times$  10<sup>22</sup>/m<sup>2</sup>·s) He<sup>+</sup> irradiations, as shown in Fig. 1(a). Prior to  $He<sup>+</sup>$  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 $\degree$ C, as shown in Fig. 1(f). However, the diameter of W nano-fuzzes does not strongly depend on the annealing temperature (Table 1), and it remains almost constant when the annealing temperature varies from 200 $\degree$ C to 1000 $\degree$ C. The cross-sectional views of W nano-fuzzes

Table 1 The diameter of W nano-fuzzes under different annealing temperature.

Specimen	Annealing temperature $(T)$ ( $\degree$ C)	W nano-fuzz diameter (nm)
11	unannealed	$25 \pm 5$
12	200	$24 \pm 4$
13	400	$27 \pm 5$
14	600	$26 \pm 4$
15	800	$26 \pm 5$
16	1000	$24 + 4$



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

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