Effect of Biasing Voltage on Growth of Fiber-Form Nanostructures on Tungsten Surfaces by Collisional Helium Arc Plasma Irradiation

M. Tajima¹, Y. Kikuchi¹, T. Aota², S. Maenaka², K. Fujita², S. Takamura²

¹Graduate School of Engineering, University of Hyogo, Hyogo, Japan ²Yumex Inc., Hyogo, Japan

Abstract: Fiber-form nanostructures were formed on tungsten (W) surfaces irradiated with helium (He) arc discharge plasma under a gas pressure of 5 kPa. The biasing voltage (V_b) applied between the W substrate and the vacuum vessel was varied from -52 to -242 V to control the incident energy E_i of He ions to the W substrate. The thickness of the fiber-form nanostructured layer increased with increasing V_b , reaching a maximum at $V_b = -162 \sim -202$ V and then decreasing towards $V_b = -242$ V. This roll-over phenomenon is thought to be caused by the competition between the growth of fiber-form nanostructures and sputtering erosion.

Keywords: Fiber-form nanostructure, Helium arc irradiation

1. Introduction

Recently, it has been found that fiber-form nanostructures are formed on metal surfaces such as tungsten (W) by irradiation with helium (He) plasma [1]. We are conducting experiments to produce fiber-form nanostructures on W surfaces by He arc discharge plasma irradiation under high neutral gas pressure. To control the He ion incident energy $E_{\rm i}$ to the W sample surface, a negative biasing voltage $V_{\rm b}$ was applied between the sample and the vacuum vessel wall. Under such high neutral gas pressure (5 kPa), collisions between ions and neutral particles in the sheath cannot be neglected and E_i has a distribution. Our previous experimental results have shown that there was no significant change on the W surface at a shallow biasing $V_{\rm b}$ = -22 V, and on the other hand fiber-form nanostructures were successfully formed at $V_{\rm b} = -112$ V [2]. On the other hand, experiments on low gas pressure plasma irradiation $(\sim 1 \text{ Pa})$ have shown that at E_i below the sputtering threshold energy of W by He ions ($E_s \sim 150 \text{ eV}$), the growth rate of fiber-form nanostructures is faster at higher E_i . In the condition of $E_i > E_s$, it has been found that as E_i is increased, the growth of fiber-form nanostructured layers saturates at lower He fluence, resulting in thinner W nanostructured layer [3]. In the present study we investigate the formation of fiber-form nanostructures on W surfaces when V_b is applied deeper than those in our previous experiments.

2. Experimental setup

Details of the experimental apparatus are described in the previous paper [2]. In this experiment, a DC He arc discharge (discharge voltage: 39 V, discharge current: 34 A) was generated between W electrodes installed in a vacuum vessel at a He gas flow rate and gas pressure of 1 L min⁻¹ and 5 kPa, respectively. In the experiments, powder metallurgy W plates ($10 \times 15 \times 2 \text{ mm}^3$) were employed. The W substrate is connected to a water-cooled holder made of copper, and the substrate temperature during plasma irradiation is determined by three mechanisms: heating by plasma irradiation, water cooling, and radiation cooling of the W surface. The surface temperature was measured using a radiation pyrometer with a radiation wavelength of 0.96 μ m during the plasma irradiation. The radiation pyrometer's emissivity was set to a fixed value of 0.43. As the emissivity of the W substrate surface may be approaching to a black body due to the formation of surface nanostructures, the measurement was performed just after the surface temperature reached a steady-state value. The sample temperature was about 1000°C and the ion flux was 6.4 × 10^{22} m⁻²s⁻¹. The ion fluence was 3.5×10^{26} m⁻². The value of $V_{\rm b}$ was varied from -52 to -242 V. The thickness *d* of the fiber-form nanostructured W layer was measured with a



Fig. 1. SEM image of indentation marking on fiber-form nanostructured W surface (a) and cross-sectional schematic of W substrate (b)

nanoindenter. As shown Fig. 1, an indentation mark was observed by a scanning electron microscope (SEM), and d was calculated based on the Berkovich indenter tip shape.

3. Experimental results

Figure 2 shows FE-SEM images of the W sample surface at different V_b conditions, showing the formation of fiber-form nanostructures at all biasing voltages, including $V_b = -52$ V. Here, the plasma potential is about -12 V and the sheath voltage is about 40 V. In the previous study [2], the value of E_i determined using the sputtering yield of carbon substrates by He arc discharge plasma irradiation was obtained as 40 - 50 eV at $V_b = -112$ V. Figure 3 shows the W fiber diameter (X_d) on the top surface of the fiber-form nanostructured layer as a function of V_b . This result indicates that the diameter of the W fiber on the top surface becomes smaller as V_b increases. In other words, the top surface W fibers become thinner as the thickness of the fiber-form nanostructured layer increases [5].

Figure 4 shows the relationship between $V_{\rm b}$ and d. Here, the value of d at $V_b = -112$ V was found to be approximately 2.9 µm obtained from the cross-sectional FE-SEM image of the W substrate, which was confirmed to be in good agreement with the result shown in Fig. 4. It can be seen that d at $V_b = -112$ V is more than three times thicker than at $V_{\rm b} = -82$ V. When $V_{\rm b} = -162$ V, the growth of fiber-form nanostructures is further enhanced and d is obtained as ~ 5.5 μ m although He ions with $E_i > E_s$ are also irradiated. On the other hand, d has its maximum value when $V_{\rm b}$ is between -162 and -202 V, and it tends to decrease when $V_{\rm b} = -242$ V. This roll-over phenomenon is thought to be caused by the competition between the growth of nanostructures and sputtering erosion. In spite of the decrease in d at $V_{\rm b} = -242$ V, the W fiber diameter becomes thinner as shown in Fig. 3. Therefore, it can be considered that the relationship between d and X_d is different from that



Fig. 2. FE-SEM images of the W surface irradiated by He arc discharge plasma with biasing voltage of -52 V, -112 V, -162 V, and -242 V under a gas pressure of 5 kPa.



Fig. 3. W fiber diameter on the top surface of the fiber-form W nanostructures as a function of the biasing voltage.



Fig. 4. Thickness of the fiber-form nanostructured layer as a function of the biasing voltage.

in the low gas pressure plasma irradiation experiment [5].

In the collisional plasma irradiation experiments such as this study, because the distribution of the incident energy of He ions due to collisions between ions and helium neutral particles in the ion sheath appears, both He ions that contribute to the growth of fiber-form nanostructures and those that cause erosion due to sputtering are irradiated simultaneously. Although the growth process of fiber-form nanostructures under such energetic conditions is still unresolved, it was found that the W surface is blackened (d= 1-2 µm) after as short as 10 minutes of plasma irradiation.

4. Summary

It was found that the high-speed formation of fiber-form W nanostructures can be achieved by appropriately selecting the biasing voltage applied between the W sample and the vacuum chamber during the collisional He arc discharge irradiation. In the future, we plan to conduct experiments in which the irradiation time (He fluence) is varied in order to clarify the effect of sputtering on the formation rate of fiber-form nanostructures.

References

- [1] S. Takamura et al., Plasma Fusion Res. 1, 051 (2006).
- [2] Y. Kikuchi et al., J. Appl. Phys. 131, 123301 (2022).
- [3] Y. Noiri et al., J. Nucl. Mater. 463, 285 (2015).
- [4] S. Kajita et al., Nucl. Fusion **49**, 095005 (2009).
- [5] S. Feng et al., Plasma Fusion Res. 16, 1206098 (2021).