Fuzz Growth Process under He-W Co-Deposition Conditions

Wanqi SHEN, Shin KAJITA[1)], Hirohiko TANAKA, Quan SHI[2)] and Noriyasu OHNO
Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan
[1)]*Graduate School of Frontier Sciences, The University of Tokyo, 5-1-5 Kashiwanoha, Kashiwa, Chiba 277-8561, Japan*
[2)]*National Institute for Fusion Science, 322-6 Oroshi-cho, Toki 509-5292, Japan*

(Received 13 June 2022 / Accepted 5 July 2022)

Helium (He)-Tungsten (W) co-deposition experiments were conducted in the linear plasma device Co-NAGDIS at a temperature of 1223 K to observe the characteristics of fuzz growth on the W surface with auxiliary W deposition. The dependence on the deposition rate of W was investigated and a clear difference in fuzz thickness was found between He-only and co-deposition experiments. In addition, the fuzz structures on a single sample exhibited a spatially nonuniform distribution, which was probably caused by the nonuniformity in the deposition rate.

Keywords: fuzz, helium, co-deposition, tungsten

DOI: 10.1585/pfr.17.1302094

Tungsten (W) is chosen as the armor material for the divertor in ITER due to its high melting point and low tritium retention [1]. Exposure to helium (He) plasma has been shown to cause morphological changes on the surface of tungsten, such as blisters, bubbles, and fiber-form nanostructures called fuzz, which can change a material's thermal conductivity and pose a risk of an increase in high-Z impurities [2]. In addition, plasma-facing components are easily sputtered and ejected by particles from plasma; they are then deposited back to the surface, together with hydrogen (H) and He.

Recently, detailed features of the He-W co-deposition layer within a temperature range from 473 K to 773 K have been revealed from experiments in the linear plasma device Co-NAGDIS [3, 4]. It has been found that when the substrate temperature is higher than 1100 K and co-deposition is present, mm-scale, fur-like structures called large-scale fiber-form nanostructures (LFNs) form in the device NAGDIS-II device, and such structures are always grown from the edges of samples and expanded in the surface direction [5]. In normal operating conditions in ITER, the divertor is supposed to face a temperature of 1273 K [6]. In this study, we mainly investigate the effects of He-W co-deposition on the morphology of W surface at 1223 K. In contrast to previous studies in which LFNs were formed, this study selects conditions in which all edges of samples are covered. Thus, we focus on the structures growing from the surface rather than from the edges. Although mm-thick LFNs are not grown, this study shows that the fuzz growth rate increases significantly with auxiliary W deposition.

Experiments were carried out in the linear plasma device Co-NAGDIS, where high density (~10^{18} m^{-3}) He plasma was produced in a steady state [3]. During the experiments, the substrate temperature was measured using a non-contact infrared thermometer (LTC Company Limited, KTL-PRO). As shown in Fig. 1, the sample holder was placed 45 degrees to the window (i.e., 45 degrees to the plasma columns) for sample temperature observation. Therefore, different locations on the sample were not at the same distance from the source along the direction of the plasma column. In the following paragraphs, the edge of the sample with a smaller distance to the plasma source is referred to as "Close side", while the opposite edge is called "Far side". The co-deposition was achieved by introducing a 1-mm diameter W wire at a position of 2~10 mm away from the sample stage, as shown in Fig. 1. The wire was bent to an "m" shape and was installed parallel to the surface of the sample. When He plasma was generated, it was able to hit the sample surface together with the sputtered W from the wire simultaneously. The experiments in this study were performed in three differ-

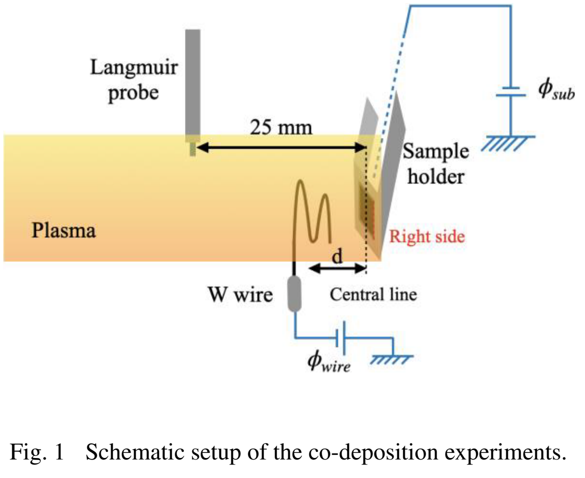

Fig. 1 Schematic setup of the co-deposition experiments.

---

author's e-mail: kajita@edu.k.u-tokyo.ac.jp ent conditions. In Case (1), the sample was irradiated only with He plasma. In Case (2), a W sputtering wire with a bias of −300 V was placed 10 mm away from the sample biased at −60 V. In Case (3), the wire was 2 mm away, with a bias of −500 V, and the sample was −90 V biased. Cases (2) and (3) correspond to low and high deposition conditions, respectively.

Figures 2 (a-c) show cross-sectional scanning electron microscope (SEM) images of fuzz structures formed in Cases (1-3), respectively, at 1223 K with a He ion fluence of $3.5\times10^{25}\ \mathrm{m^{-2}}$. All the observations shown in Fig. 2 were taken around the center of Far side. In Case (1) (Fig. 2 (a)), a fuzz layer with a thickness of 0.65 μm is observed. Compared with Figs. 2 (b, c), a clear difference in fuzz thickness was found between the He-only and co-deposition experiments. For the co-deposition cases, the layer thickness increased with the sputtering yield of the W wire. Especially in Case (3), the fuzz layer had an average height of 5.3 μm. Preliminary conclusions can be drawn as follows: first, the sputtered W atoms are able to co-deposit with He, contributing to the morphological changes in the W surface; second, a higher sputtering and depositing rate of W can enhance this effect. One thing to note is that although the experiment's parameters, such as He fluence and substrate temperature, have met the requirements to form LFNs in NAGDIS-II [5], the maximum fuzz thickness here is still μm-scale.

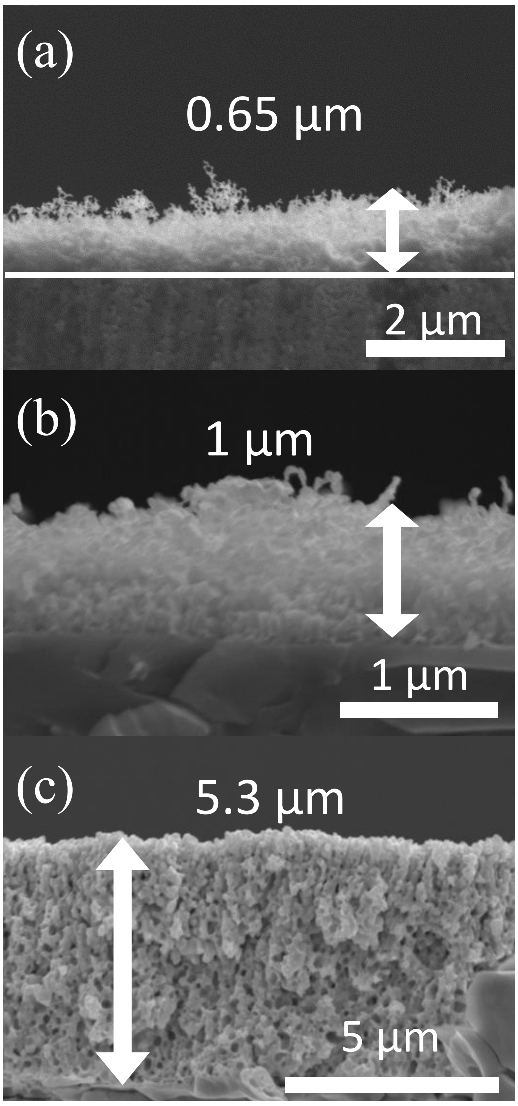

Fig. 2 (a-c) SEM images of the cross section of the fuzz layer formed in Case (1–3), respectively, at 1223 K when the He fluence is $3.5 \times 10^{25}\ \mathrm{m^{-2}}$.

A common feature found among the experiments conducted in Case (3) was that the darkness of the irradiated surface showed a positive gradient from Close side to Far side. This might be a result of the growth difference in the co-deposition layer. In order to observe the cross sections of the sample, it was cut along the lines shown in Fig. 3 (a). After an overview of the whole surface by SEM, the bottom area of Close side (Region A) and the top area of Far side (Region B) (marked with white dots) were picked out for detailed observation because of the obvious smooth and rough features they presented. Magnified views of regions A and B are shown in Figs. (b, c). Figures 3 (d, e, g, h) are high magnification images of regions A and B, while Figs. 3 (f, i) are their cross sections, respectively. It can be clearly seen from Figs. 3 (d-f) that plump and dense clusters of fuzz with a thickness of around 2.8 μm were grown in Region A. Figures (g, h) show a membrane-like structure previously observed in deposition experiments [7]. Unlike fuzz structures, the membranes are emerged independently and with a much larger size. These structures are considered to form from fiber-form structures that are extended two-dimensionally with deposited W atoms. Figures 3 (f, i) show that the layer thickness of the Region B is approximately 5 μm thicker than that of the Region A.

In addition, the thickness profile of the fuzzy layer obtained from the SEM cross section along the axis from Region A to Region B of the sample is shown in Fig. 4 as a function of the distance from Close side (left edge of the sample). The fuzz thickness increased more than 2.8 times from Close side to Far side. At 8.5 mm, the thickness was about 8 μm while it was only 2.8 μm at 1 mm. However, the value is still four times larger than the fuzz thickness formed without co-deposition.

Figure 5 illustrates the relationship between He fluence and fuzz thickness based on different irradiation conditions, both in this study and previous studies [8, 9]. For the nonuniform property presented by samples in Case (3), error bars are used to represent minimum and maximum thickness. The dotted line at the bottom of this figure is drawn from data obtained from previous He-only experiments in NAGDIS-II [8]. The sample with a fuzz thickness of 0.65 μm in Case (1) was in perfect agreement with the trend line of the thickness variation at $3.5 \times 10^{25}\ \mathrm{m^{-2}}$. However, the samples for the other two cases in this study had thicker fuzz structures. In Case (2), the fuzz thickness was 0.45 μm at $2.2\times10^{25}\ \mathrm{m^{-2}}$ and 1.0 μm at $3.5\times10^{25}\ \mathrm{m^{-2}}$.

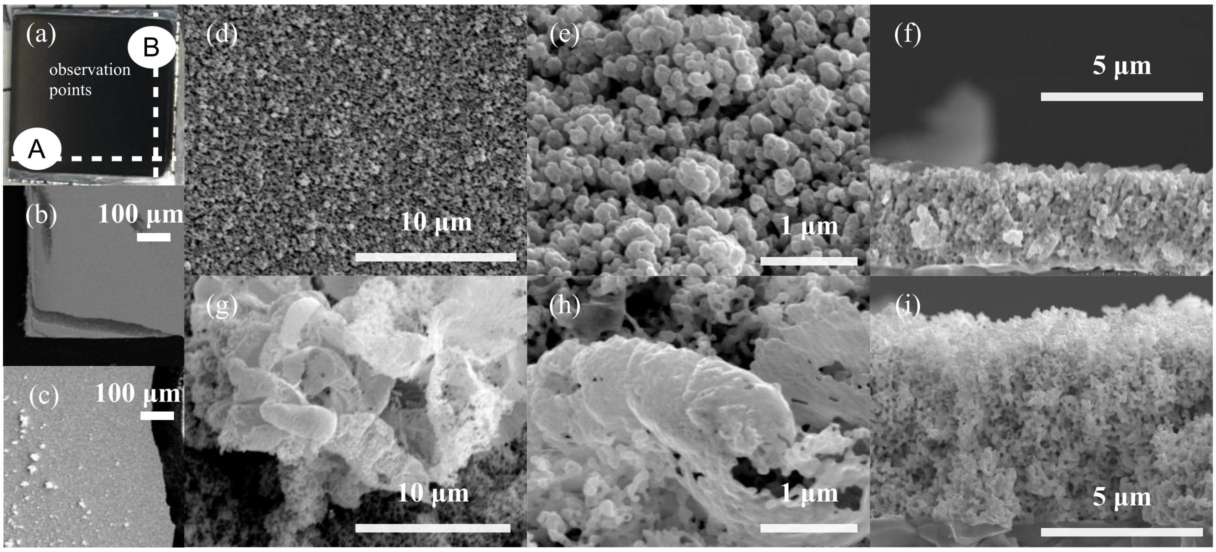

Fig. 3 Photo (a) and SEM micrographs (b–i) of the W sample. Circles A and B in (a) mark relatively thinner and thicker regions, respectively. (b, d, e, f) are the overview, the low magnification, high magnification view and the cross section of region A, respectively; (c, g, h, i) are the overview, the low magnification, high magnification view, and the cross section of region B of the sample, respectively.

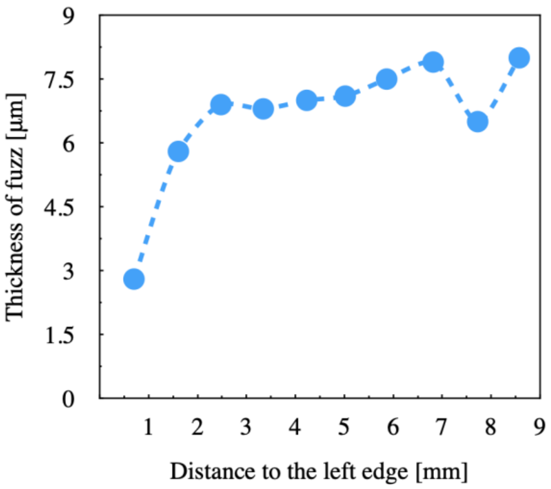

Fig. 4 Transverse surface distribution of fuzz thickness.

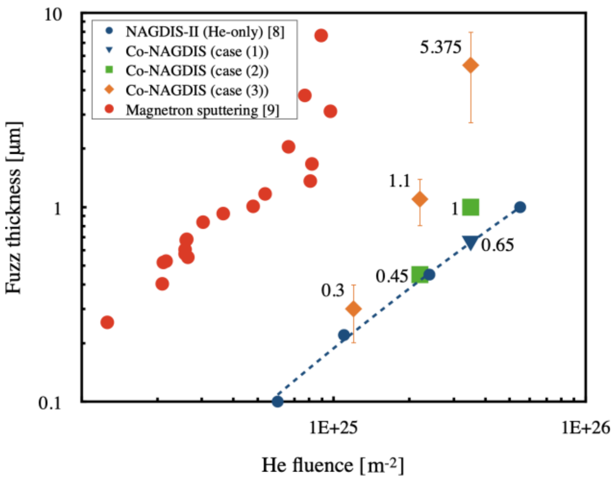

Fig. 5 A comparison of fuzz thickness as a function of He fluence between this study and previous studies: He-only irradiations from NAGDIS-II [8] and He-W co-depositions in a magnetron sputtering device [9]. Error bars were calculated from the minimum and maximum thickness values.

For Case (3), the average fuzz thickness was much greater than that in Cases (1, 2). The thickness identified in Case (3) was much less than that in magnetron sputtering co-deposition experiments [9], which is shown with red markers in Fig. 5, at the same He fluence. The ratio between the amount of W and He reaching the sample surface is considered as a key factor affecting the formation of W fuzz. McCarthy found that thick W fuzz can form when W/He ratio is within the range from 0.003 to 0.009 over a certain range of He ion fluence [9]. In addition, Kajita concluded that fuzz formation was enhanced when the W/He has a value higher than 0.0004 [10].

To calculate the W/He ratio for Case (3), we took the mass difference of the sample before and after the experiment as the W value and defined the incident He fluence as He value. The W/He value obtained in Case (3) was ~0.007, which is almost comparable to the values found in McCarthy's work [9]. One potential explanation is the difference in exposure time, namely, the difference in He and W flux. In this study, the He flux was more than an order of magnitude greater than that in McCarthy's research [9]. For conventional fuzz, no clear dependence on the He flux has been identified [11]; thus, co-deposition cases could have flux dependence. It is of interest to perform further experiments under different flux conditions in the future.

In this study, fuzz structure He-W co-deposition experiments were carried out in Co-NAGDIS at 1223 K. The dependence on the sputtering rate of the W wire was investigated at this temperature. A clear difference in fuzz thickness can be found between He-only and co-deposition experiments, as well as between experiments with high and low sputtering rates. The co-deposition of He and W has been proven to contribute to morphological changes. A high sputtering yield and a shortened distance between the sputtering source and the substrate are two crucial factors speeding the growth process of fuzz structures as well. In addition to comparisons between different samples, the fuzz structures of a single sample exhibited a spatially uneven distribution. Membranous structures were found in samples with a fuzz thickness lower than 10 µm. These phenomena are probably related to the distance between the sputtering source and the substrate; in the He-only irradiation and sputtering wire far-placed experiment, no similar findings were found. With the distance between the sputtering source and the sample approaching 2 mm, regions of the sample that are closer to the sputtering source tend to form thinner fuzz than in the farther regions. A definitive explanation for this phenomenon still requires further research.

This work was supported in part by a Grant-in-Aid for Scientific Research 19H01874 and 21K18617, and Fund for the Promotion of Joint International Research 17KK0132 and 21KK0048 from the Japan Society for the Promotion of Science (JSPS), and NIFS collaboration research program (NIFS22KIIH006 and NIFS22HDAF009). Authors thank Mr. T. Eda and Mr. R. Zhang for useful discussion and comments.

[1] T. Hirai *et al.*, "Use of tungsten material for the ITER divertor", Nucl. Mater. Energy **9**, 616 (2016).

[2] M. Thompson, *Helium Nano-Bubble Formation in Tungsten: Measurement with Grazing-Incidence Small Angle X-Ray Scattering* (Springer, 2018).

[3] K. Asai *et al.*, "Microstructure and Retention in He-W Co-Deposition Layer", Plasma Fusion Res. **15**, 1201004 (2020).

[4] S. Kajita *et al.*, "Helium-W co-deposition layer: TEM observation and D retention", Nucl. Mater. **540**, 152350 (2020).

[5] S. Kajita *et al.*, "Tungsten fuzz: Deposition effects and influence to fusion devices", Nucl. Mater. Energy **25**, 100828 (2020).

[6] ITER Organization. Available at: https://www.iter.org/mach/divertor

[7] S. Kajita *et al.*, "Growth of membrane nanostructures on W co-deposition layer", Nucl. Mater. Energy **18**, 339 (2019).

[8] S. Kajita *et al.*, "TEM observation of the growth process of helium nanobubbles on tungsten: Nanostructure formation mechanism", Nucl. Mater. **418**, 152 (2011).

[9] P. McCarthy *et al.*, "Enhanced fuzzy tungsten growth in the presence of tungsten deposition", Nucl. Fusion **60**, 026012 (2020).

[10] S. Kajita *et al.*, "Morphologies of co-depositing W layer formed during He plasma irradiation", Nucl. Fusion **58**, 106002 (2018).

[11] T.J. Petty *et al.*, "Tungsten 'fuzz' growth re-examined: the dependence on ion fluence in non-erosive and erosive helium plasma", Nucl. Fusion **55**, 093033 (2015).