Tungsten Large-Scale Fiberform Nanostructures Retained under High Temperature Conditions

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Large-scale fiberform nanostructures (LFN) are formed on the tungsten (W) surface with He-W co-deposition environments. In this study, we conducted annealing experiments at 1473 - 1673 K for 30 min using an infrared heating furnace. It was found that the LFN retained their structures after annealing at 1673 K. Through detailed observations using an optical microscope, a scanning electron microscope (SEM), and a transmission electron microscope (TEM), the morphological changes are discussed in relation to the high-temperature stability.

Keywords: He-W co-deposition, fuzz, tungsten, annealing, LFN

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Fiberform nanostructures called *fuzz* are formed on the surface of metals such as tungsten (W) by helium (He) plasma irradiation [1–3]. Formation of fuzz on metal surfaces increases the surface area [4] and optical absorption [5], and there are expectations for industrial applications such as photocatalysis [6] and a gas sensor [7], as taking advantage of these characteristics. However, it has been confirmed that the fuzz structures disappear by annealing at 1300 - 1500 K for about 30 min, as they are reintegrated to the surface [8–11]. In this study, we focus on large-scale fiberform nanostructures (LFN) [12–15], which is orders of magnitudes thicker than that of conventional fuzz [2,15]. LFN can be formed with an auxiliary deposition and, like fuzz, leads to an increase in surface area and optical absorption. We have investigated the morphology and property changes of LFN by annealing at high temperatures to elucidate the stability of LFN at high temperatures.

The linear plasma device NAGDIS-II, which produced a high-density plasma in, steady state, was used for plasma irradiation. Tungsten wire was placed upstream of the W sample and biased to −400 V to induce sputtering [12]. The parameters for He irradiation are as follows: the surface temperature of 1300 K, incident ion energy of 60 eV, and He fluence of up to 6.0×10^{25} m^{-2}. Then, annealing experiments were performed in an infrared heating furnace equipped in a thermal desorption spectroscopy (TDS) device. The temperature was raised linearly at a heating rate of 0.5 K/s to 1473 - 1673 K in vacuum, and was kept at the maximum temperature for 30 min. An optical microscope, a scanning electron microscope (SEM), and a transmission electron microscope (TEM) were used for the surface analysis.

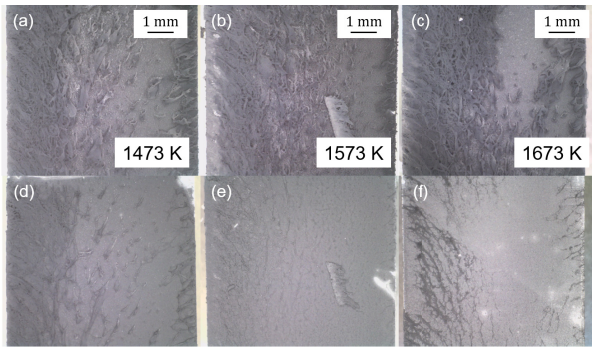

Fig. 1 Optical microscopy images of LFN (a, b, c) before and after annealing at (d) 1473 K, (e) 1573 K and (f) 1673 K.

Figure 1 shows optical microscope images of the LFN-formed W samples (a, b, c) before and (d, e, f) after annealing. When the annealing temperature was 1473 K (Figs. 1 (a, d)), the LFN shrunk and partially disappeared. At 1573 K (Figs. 1 (b, e)), most of the LFN started to disappear, and at 1673 K (Figs. 1 (c, f)), in addition to the disappearance of the LFN, the silver-whitening of the substrate was observed. This suggests that the reintegration of the surface is in progress. However, the LFN has not disappeared completely even at the annealing temperature of 1673 K.

Figures 2 (a-d) shows SEM images of the fibers of LFN before and after annealing at the three different temperatures, respectively. With increasing the annealing tem-

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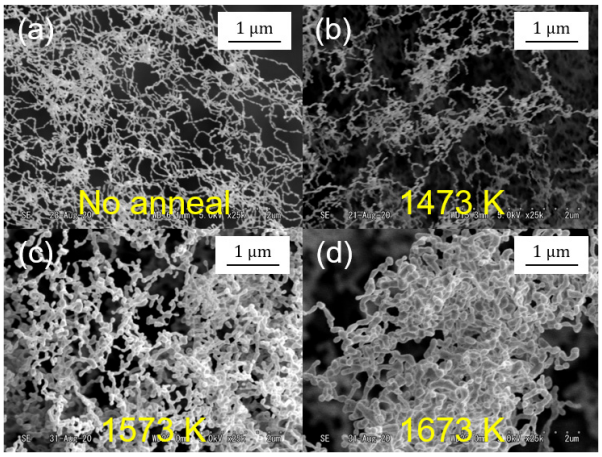

Fig. 2 SEM images of LFN (a) with no anneal and after annealing at (b) 1473 K, (c) 1573 K, (d) 1673 K.

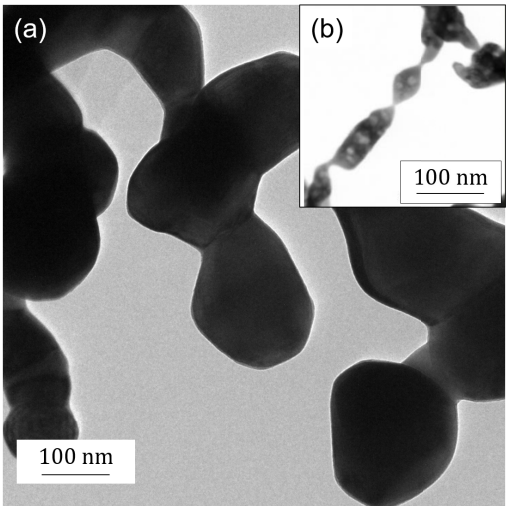

Fig. 3 TEM images of LFN (a) after annealing at 1673 K and (b) without annealing [17].

perature, the fiber diameter of LFN became larger from 30 nm to 100 nm. Fine fibers seemed to have merged and become thicker during annealing. The same trend was observed in the annealing experiment of conventional fuzz [11], although the fibers disappeared fully at 1473 K. It is noteworthy that the fibers still remained even after annealing at 1673 K (Fig. 2 (d)). It is suggested that the fibers of LFN are more stable at higher temperatures than those of conventional fuzz.

The behavior of He bubbles [16] and surface diffusion of W [2] are the factors that cause the conventional fuzz to lose its shape due to annealing. Figure 3 (a) shows a TEM image of the fibers of LFN after annealing at 1673 K. It was confirmed that there were no He bubbles in the fibers of LFN, though a large number of He bubbles were present in the fibers of LFN before annealing (Fig. 3 (b)). This indicates that the He bubbles inside the LFN fibers were fully released by increasing the temperature.

The fact that the structure of the LFN fibers does not change significantly is probably due to the insufficient surface diffusion of W compared to the length of fibers. LFN has approximately ten times higher porosity than that of conventional fuzz [15]. In other words, the distance between fibers of LFN is longer than those of conventional fuzz. If the distance between fibers is far, the required time for W surface diffusion becomes longer. Therefore, in the limited annealing time, sufficient W surface diffusion may not occur until the surface is reintegrated. Although the maximum temperature and time was 1673 K and 30 min in this study, it is of interest to investigate the morphological change at higher temperatures and times.

In this study, the LFN was annealed at 1473 - 1673 K and the morphological changes were observed. Even after annealing at 1673 K for 30 min, the LFN retained fiber shape. It was also observed that the fiber diameter of LFN became larger as the annealing temperature increased. Further detailed TEM observations revealed that the fibers of LFN after annealing retained their structure even after the release of He bubbles. This result shows that the LFN has better shape stability at high temperature than the conventional fuzz. In this respect, the LFN is considered to be superior to conventional fuzz for industrial applications under high temperature such as the emitter of thermophotovoltaic cells [18].

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