In Situ TEM Observation of Helium Bubbles Collapsing on Nanostructured Tungsten during Annealing

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A fiberform nanostructured layer is formed on a tungsten (W) surface using helium (He) plasma irradiation. The behavior of the nanostructures and He bubbles were observed during annealing via in situ transmission electron microscopy. Notable changes in the nanostructures occurred at 1223 K. A collapse of the nanostructures was observed along with a collapse of the He bubbles during annealing.

Keywords: tungsten, fiberform nanostructure, annealing, transmission electron microscopy, helium bubble

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It is known that strong morphology and property changes, such as the formation of helium (He) bubbles [1–3] and fuzzy fiberform nanostructures [4, 5] (hereafter referred to as "nanostructures"), occur when tungsten (W) surfaces are irradiated by He plasma. Studies have revealed that nanostructures are formed when the temperature is in the range of 1000 - 2000 K and the incident ion energy is higher than 20 eV [6].

These nanostructures reintegrate onto the surface of W when heated above ~1000 K without He-ion irradiation [7–12]. In a study, annealed samples subjected to heating in stages were observed using transmission electron microscopy (TEM), and it was found that the recovery of the nanostructures occurred at 1173 K during the surface diffusion of W [13]. However, the behavior of He bubbles during annealing and their relation to collapsing of the nanostructures are yet to be identified.

In this study, we investigate the behavior of He bubbles during the annealing process using a special TEM device that enables observations to be made while the sample is being heated. Our study is the first to capture the moment when a He bubble bursts during annealing. Moreover, unlike previous studies [13], our nanostructures are coated by carbon during annealing. This simulates the effect of depositions, which can occur during an actual annealing processes, on the nanostructures. The influence of coating will be discussed later.

Mechanically polished powder metallurgy W disks (Nilaco Co., Ltd.) with a diameter of 5 mm and a thickness of 0.2 mm were used as samples. The polished samples were irradiated with He plasma in the linear divertor plasma simulator NAGDIS (NAGoya DIvertor Simulator)-II [14]. The incident ion energy was controlled by biasing the sample, and it was approximately 55 eV. The sample was exposed to He plasma at 1330 K up to 2.0×10^{25} m^{-2}. The surface temperature of the sample was measured using a radiation thermometer.

To evaluate the damage to the surface, a thin (~100 nm) sample fabricated using a focused ion beam (FIB) milling apparatus was used for TEM observation. The irradiated surface was coated with an oil-based ink, and W was successively deposited on it to protect it against the sputtering erosion that could occur during the FIB process. The main component of the ink was C. It was confirmed via TEM observation that the surface of each W fuzz was completely covered by the ink. The samples were heated stepwise to 1273 K during the observation. The dynamic behavior of the sample surface was recorded using a framing camera. High-resolution images were also taken at random intervals.

Figure 1 (a) shows a TEM image of the cross-sectional sample. As expected, the irradiated surface is covered by typical nanostructures, with each having a thickness of approximately 600 nm on an average. The change in the nanostructure, which was observed during heating in area I shown in Fig. 1 (a), is shown in detail in Fig. 1 (b). Many He bubbles (white images) with a diameter <10 nm were formed in the thin-curved W fibers. They were very stable; up to 1173 K, the sizes and positions of the He bubbles scarcely changed. However, a notable change occurred at 1223 K. After heating for 15 min at this temperature, we started to take a series of pictures using the framing camera. The TEM pictures are shown in Fig. 2. Many of the He bubbles, such as the ones in area II, became faint and finally disappeared. Interestingly, in area III, the number of small bubbles near the fiber surface increased as they began to continuously combine with neighboring small bubbles until a large bubble appeared very close to the surface. After some time, this large bubble reached the surface, and the opened hole spread quickly and changed the shape of the fiber. We believe that the collapse of large bubbles en-

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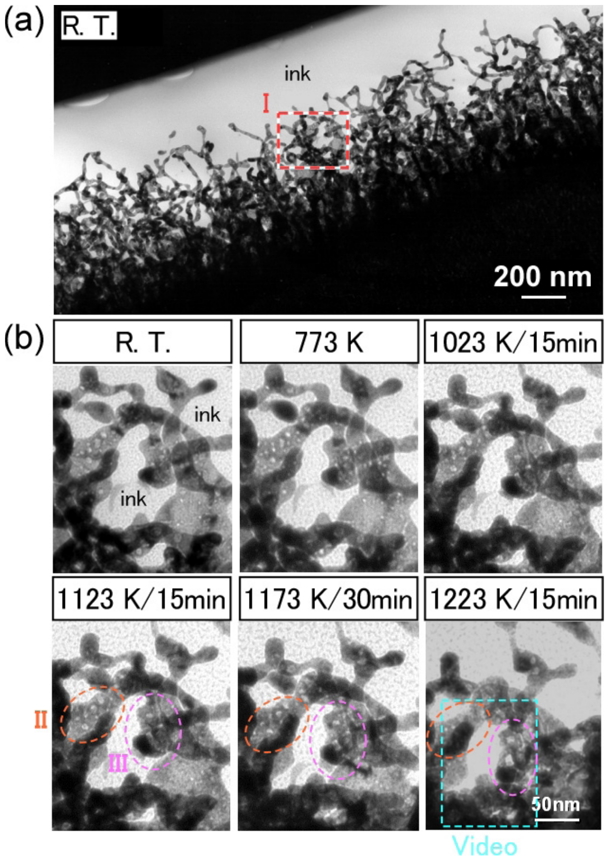

Fig. 1 Cross-sectional TEM images of the annealed nanostructured sample.

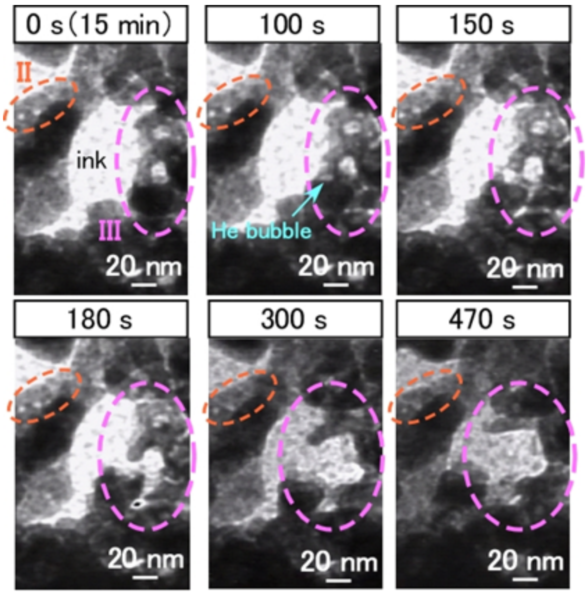

Fig. 2 Detailed TEM images at different heating times in the area of the annealed nanostructured sample seen in Fig. 1 (b). The sample temperature was maintained at 1223 K.

hanced the collapse of the nanostructure and the desorption of He trapped in the fibers.

A previous study [5] found that W surface diffusion is enhanced at ~1173 K. However, in the present experiment, the nanostructures sustained even at 1173 K; this was probably due to the C coating on the surface. Small bubbles in the nanostructures covered by C started to move at ~1223 K. Therefore, it is likely that the collapse of the nanostructures was mainly due to the collapse and diffusion of the He bubbles when the surface diffusion of the W atoms was suppressed by the deposition of impurities on the nanostructures.

In this study, the thermal behavior of the nanostructures that gained a carbon coating was studied using in situ TEM observations during annealing. Notable changes in the nanostructures occurred at 1223 K, which was significantly higher than the temperature value reported in a study [13]. This was probably due to suppression of the carbon coating process by surface diffusion. Collapsing of a He bubble was observed using TEM analysis, the results of which suggest that the collapse of the carbon-coated nanostructures was mainly caused by the collapse of the He bubbles. In ITER, it is possible that the surface will be covered by beryllium. In those situations, the processes and necessary surface temperatures required for the reintegration of the nanostructures could be different than for nanostructures that do not have such a coating.

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