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Formation condition of fiberform nanostructured tungsten by helium plasma exposure

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Tungsten samples were irradiated by high density helium plasma in the divertor plasma simulator NAGDIS-II to investigate the formation condition of the fiberform nanostructured tungsten. Experimental result indicates that the surface temperature threshold exists around 1000 K. When the surface temperature is above 1130 K, when the incident ion energy is 50 eV, the fiberform nanostructure thickens with the increase of the sample temperature. Three samples manufactured by different procedure (ultra fine grained tungsten, ITER-reference tungsten grade and powder metallurgy tungsten) were exposed to helium plasma, and the nanostructure was formed in all the samples.

Keywords: Helium plasma, Tungsten, Optical reflectivity, Nanostructure, NAGDIS-II

1. Introduction

Recently, it has been found that the fiberform nanostructured tungsten is formed on tungsten surface by irradiating helium plasma [1]. Tungsten is a candidate material for divertor armor in ITER [2] because of its good thermophysical properties, high melting point, low sputtering yield and a low tritium inventory. Additionally, tungsten is also a candidate material for in-vessel mirror material in fusion devices [3]. When it is used as a material of these positions, the nanostructure may cause serious problems because it could lead to the decrease of the optical reflectivity [4] and the thermal conductivity [5]. Therefore, it is necessary to investigate the formation condition and the formation mechanism of the fiberform nanostructure.

Until now, it has been revealed that the high helium ion fluence ($>10^{25}$ m^{-2}) is necessary to cover the surface with the fiberform nanostructured tungsten [1,4,6]. Moreover, it has been found that the incident ion energy is an important parameter to form the nanostructured tungsten. When the incident helium ion energy was above 30 eV, the fiberform nanostructure was formed at the surface temperature of 1800 K, although only the helium bubbles and holes were formed when the incident ion energy was 15 eV [4]. Additionally, it is reported that a nanostructured layer thickness of 5μm is observed by irradiating helium plasma at the fluence of 1.1 x 10^{27} m^{-2} [6].

In this paper, to investigate the formation condition in detail, helium plasma irradiations are performed at different surface temperatures with fixed incident ion energy. In addition, experiments are performed by using the tungsten samples manufactured by different procedure. The fluence of the helium ion to form the nanostructure is evaluated by the measurement of the optical reflectivity because the optical reflectivity decreases to ~ 0 % due to its complicated structure when the surface is covered with the nanostructure. The surface is analyzed by scanning electron microscope (SEM) after the helium plasma exposure.

2. Experimental setup

Experiments were performed in the divertor plasma simulator NAGDIS-II (NAGoya university DIvertor Simulator-II). Figure 1 shows a schematic view of the experimental setup. High density helium plasma, which is produced by dc arc discharge, was irradiated to tungsten samples situated in parallel to the magnetic field line. The sample, which is powder metallurgy tungsten provided by Nilaco Co. with 99.95 % purity, was polished by sandpapers and alumina suspension. In order to control the incident ion energy to the samples, the samples were electrically biased in the helium plasma. The optical reflectivity of the sample was measured by using a He-Ne laser, of which wavelength is 632.8 nm, and a photodiode with a band-pass optical filter to exclude the emission of plasma. The surface temperature of the samples was measured by a pyrometer and the incident ion flux was derived by the ion current. After the helium plasma irradiation, the samples were analyzed by a SEM and were

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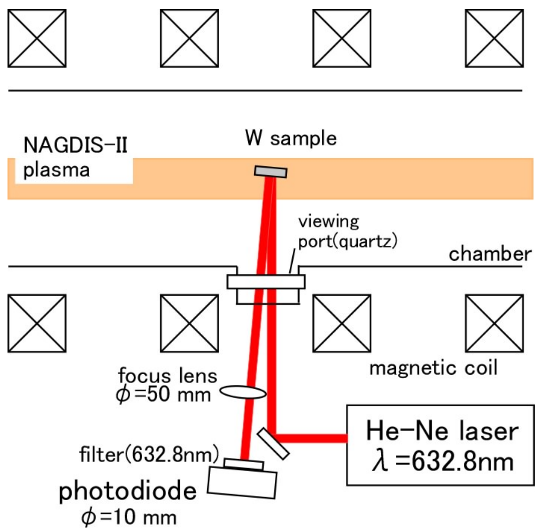

Fig. 1 Schematic illustration of the experimental setup in the divertor simulator NAGDIS-II.

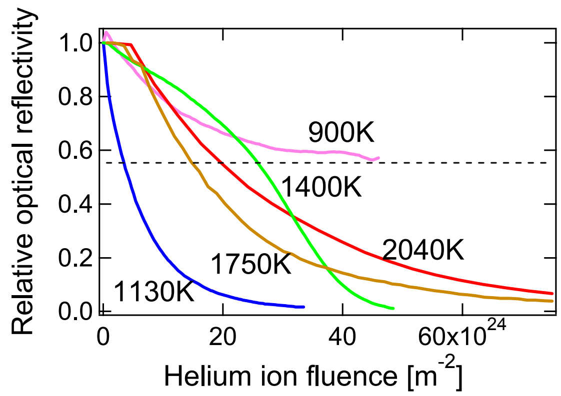

Fig. 2 Measured reflectivity of tungsten under helium plasma exposure at different sample temperatures from 900 K to 2040 K

measured with a spectrophotometer (Nihon Bunkosya: ARV-47S) for the wavelength between 200 and 900 nm**.**

3. Results

3. 1. Surface temperature dependence

Figure 2 shows the relative optical reflectivity as a function of helium ion fluence while helium plasma was irradiated. Five samples were irradiated to the helium plasma at different surface temperatures from 900 K to 2040 K. The incident helium ion energy was fixed to 50 eV. When the sample temperature was 900K, the relative optical reflectivity decreased to 0.55 at the fluence of 4.5 x 10^{25} m^{-2} and the surface had a metallic luster even after the helium irradiation. On the other hand, when the sample temperature was 1130 K, the relative optical reflectivity decreased to ~ 0 at the fluence of 3.0 x 10^{25} m^{-2} and the surface color changed to black. Figure 3 shows the SEM micrographs of samples after the helium plasma exposure. The surface irradiated at 900 K was modified to the wavy structure (Fig. 3, (a)). However, the surface irradiated at the temperature from 1130 K to 2040 K was covered with the fiberform nanostructure (Fig. 3, (b)~(e)). It is indicated that the sample temperature should be greater than ~ 1000 K to form the nanostructure.

The size of the fiberform nanostructured tungsten is different by the temperature of the samples. The size of the structure are, typically, < 40 nm at 1130 K (Fig. 3 (b)), < 200 nm at 1750 K (Fig. 3 (c)), and < 1 μm at 2040 K (Fig. 3 (e)). The structure irradiated at 1750 K is complicated, but the average size is < 1 μm (Fig. 3 (d)). It is indicated that the size of nanostructure thickened with the increase of the sample temperature. Besides, more helium fluence was required to decrease the relative optical reflectivity to ~ 0 with the increase of the sample temperature from Fig. 2.

Figure 4 shows the wavelength dependence of reflectivity of polished sample, and samples after the helium plasma exposure. The optical reflectivity at the wavelength of 632.8 nm corresponds to the one that was measured by the photodiode. For the wavelength λ = 900 nm, the reflectivity decreases by ~ 20 %; on the other hand, it decreases by ~ 70 % for λ = 200 nm. The degree of reflectivity reduction increases as the decrease of the wavelength. From Fig. 3 (a), the size of the surface roughness is approximately several tens nm. Thus, it is suspected that the roughness does not nave significant influence on the optical reflectivity if the wavelength is ten times greater than the structure. When the irradiation temperature was higher and the surface is covered by the fine structure, the optical reflectivity becomes 0 in the range of 200 < λ < 900 nm.

It has been reported that helium ions produce bubbles and holes on the surface at the incident ion energy below the threshold value of physical sputtering [7-9]. When the surface temperature is higher than ~ 1600 K and the incident ion energy exceeds ~ 5 eV, the micrometer-sized bubbles and holes are formed [9]. This paper shows that the necessary minimum sample temperature to form the fiberform nanostructure is approximately 1000 K when the incident ion energy is 50 eV. This temperature is lower than the threshold of the micrometer-sized bubble and hole formation. Meanwhile, the nanometer-sized bubbles formation is reported at the temperature of 700 K [10]. Since the bubble size increases due to the high sample temperature, the nanostructure thickens with the increase of the sample temperature. We believe that the nanometer-sized bubbles formation and their coalescence

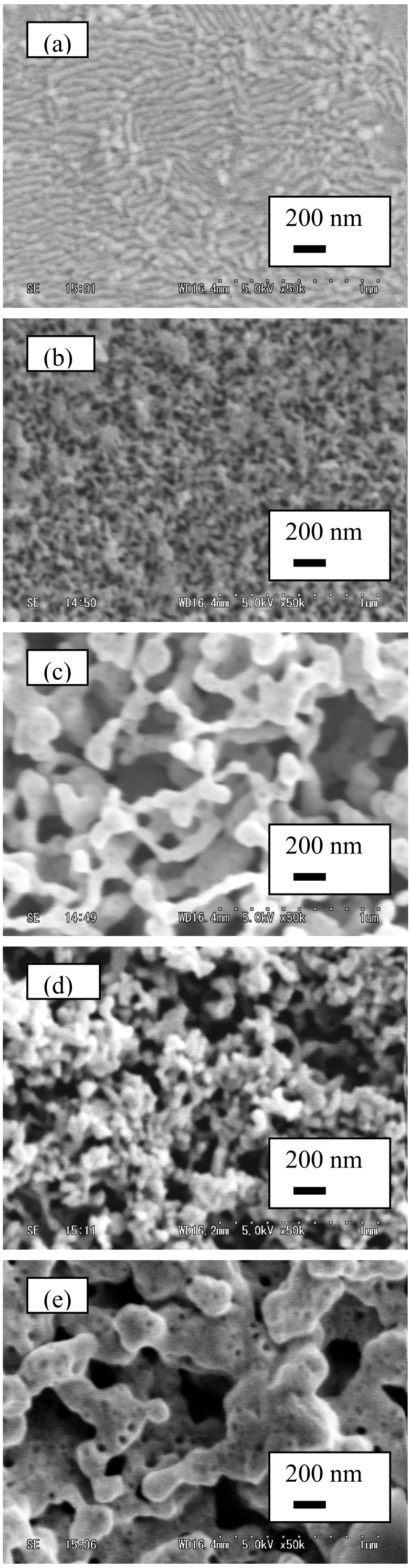

Fig. 3 SEM micrographs of the tungsten surface after the helium plasma exposure. The sample temperatures are (a) 900 K, (b) 1130 K, (c) 1400 K, (d) 1750 K and (e) 2040 K, respectively.

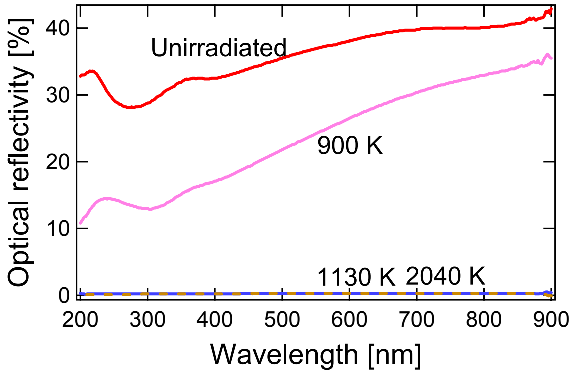

Fig. 4 Reflectivity of polished samples and samples irradiated at 900 K, 1130 K, 2040 K.

play a role to form the fiberform nanostructured tungsten. The incident helium ion energy threshold of the nanostructure formation is ~ 30 eV which is higher than that of the bubble formation at the surface temperature of ~ 1800 K. The formation mechanism of the nanostructure is not understood well yet.

3. 2. Different manufacturing procedure

Figure 5 shows the optical reflectivity as a function of helium ion fluence. Three samples, which were manufactured by different procedure, were irradiated to the helium plasma. First one is the ultra fine grained (UFG) W-0.5 wt. % TiC consolidate which is fabricated utilizing mechanical alloying in purified Ar and hot isostatic pressing [11]. The grain size is approximately 70 nm. Second one is the ITER-reference tungsten grade (ITER). The grain size is approximately 20 μm. Third one is the powder metallurgy tungsten. The incident ion energies were 50 eV and the surface temperatures were 1700 K, 1880 K and 1750 K, respectively. The fiberform nanostructured tungsten was formed on all the samples. Figure 6 shows the SEM micrographs of UFG and ITER after the helium plasma exposure. The size of the nanostructure is almost same. The fluence to form the nanostructure was almost same at ~ 10^{26} m^{-2} from Fig. 5. Clear differences on the formation of the nanostructure were not observed even when the manufacturing procedure and grain size are different. It is noted that the surface temperature was higher than the recrystallization temperature. Thus, the recrystallization process may modify the originality of each sample. For further work, it is expected that the irradiation at lower temperature make it clear the difference between them.

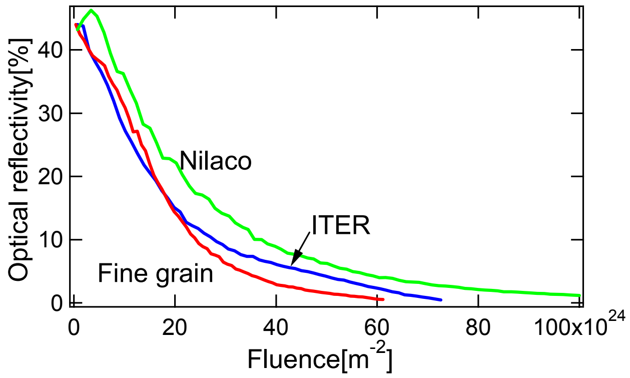

Fig. 5 Measured reflectivity of three samples which were manufactured by different procedure

4. Conclusion

Helium plasma irradiation experiments have been performed to investigate the necessary conditions for the formation of the fiberform nanostructure. This paper reveals that the necessary minimum sample temperature to form the nanostructure is approximately 1000 K when the incident ion energy is 50 eV. Moreover, it has been reported that the incident ion energy threshold of the nanostructure formation is ~ 30 eV. Considering the use of tungsten as divertor and mirror materials, the formation of the fiberform nanostructured tungsten has to be prevented. Thus, it is necessary for the fusion device materials to keep a material temperature under ~ 1000 K or to keep the incident helium ion energy under ~30 eV.

The nanostructure thickens with the increase of the sample temperature. We consider that the size of helium bubbles influence the size of the nanostructure. No clear differences on the formation of the nanostructure were observed at the temperature of ~ 1800 K even when the manufacturing procedure and the grain size are different.

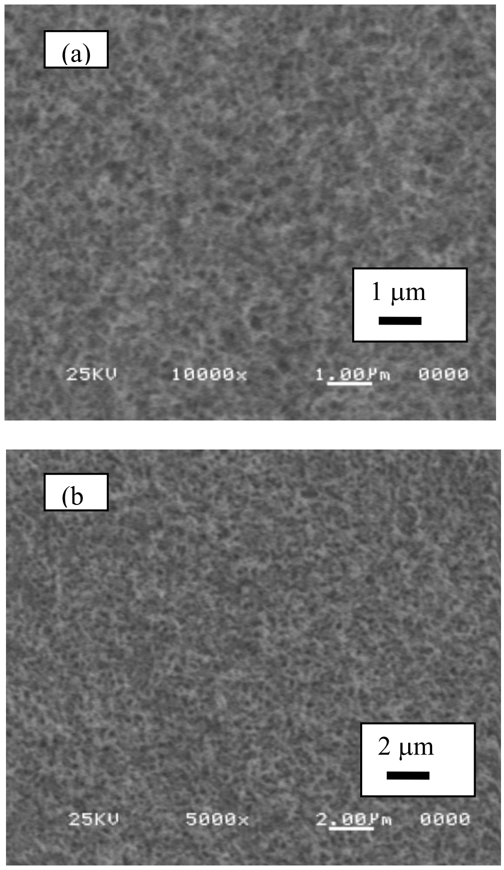

Fig. 6 SEM micrographs of tungsten surface (a) UFG and (b) ITER after helium plasma exposure.

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