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Blistering of tungsten films deposited by magnetron sputtering after helium irradiation

Jiangang Yu[a], Wenjia Han[a], Zhe Chen[a], Kaigui Zhu[a,b,c,*]

[a] Department of Physics, Beihang University, Beijing, 100191, PR China
[b] Beijing Key Laboratory of Advanced Nuclear Energy Materials and Physics, Beihang University, Beijing, 100191, PR China
[c] Key Laboratory of Micro-nano Measurement-Manipulation and Physics, Ministry of Education, Beihang University, Beijing, 100191, PR China

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A B S T R A C T

In this paper, tungsten films were deposited on tungsten substrates by magnetron sputtering and some films were annealed at 1273 K for 1 h. Subsequently, the morphology and microstructure of as-deposited and pre-annealed films were measured by scanning electron microscopy and X-ray diffraction. In addition, both films were exposed to helium ions with energy of 60 keV and fluence of $1.0 \times 10^{22}\,m^{-2}$ at room temperature. After helium irradiation, the blisters formed on surface of films induced by helium irradiation were observed by scanning electron microscopy. Here we report the differences in blistering on surface between as-deposited and pre-annealed films. In addition, in spite of similar microstructure of both films including grain size and preferred orientation, the different compressive stresses within both films lead to the different behaviors of blistering.

1. Introduction

Tungsten (W) is not only regarded as a typical industrial material used in the fields of aerospace, war industry, electron optics, mechanical workout and others, due to its favorable thermodynamic properties [1–3], but also a promising candidate for plasma-facing materials (PFM) in fusion reactors due to its low hydrogen permeability, low sputtering erosion yield, high thermal conductivity and no chemical reaction with hydrogen [4–6]. Tungsten considered as PFM has been researched for several decades. In the actual nuclear fusion devices like International Thermonuclear Experimental Reactor (ITER), tungsten must be simultaneously subjected to various particles irradiation with high flux ($10^{22} \sim 10^{25}$ ion/m^2 × s), including hydrogen and its isotopes, helium, neutron and other trace impurities [7–9]. These issues can cause diffusion and retention of deuterium and helium in materials, as well as blistering on surfaces of irradiated PFMs, which could lead to partial exfoliation and local melting. So these phenomena may be harmful to the stability of a fusion reactor [10–13].

It has been reported by many researchers that nanostructured materials have the characteristics of radiation resistance, because it can introduce a high density of grain boundaries and interfaces which are regarded as sinks for irradiation-induced defects [14–17]. For this reason, The fine crystalline strengthening is a method to improve anti-radiation properties of PFMs, which can restraint blistering on surface and reduce retention of deuterium/helium [18–22]. Magnetron sputtering as a means of physical vapor deposition is able to prepare thin films with a distinct nanostructure [23,24]. With respect to the PFMs [6,25], the thickness of the films must exceed several micrometers to cover the diffusion depth of hydrogen isotopes and helium in a film. Moreover, the tungsten films or coatings have been studied for decades [26–30], especially in the retention behavior of hydrogen and deuterium [27,28,30–32] and thermal shock resistance [26,33]. In addition, the spherical- crown-shaped blisters induced by helium irradiation with large plastic elongation on surface of polycrystalline tungsten and nanocrystalline tungsten film have been reported in our previous works [34,35]. We proposed that mass flow resulting from point defect diffusion in collision cascades can be responsible for this special shape of blister [34]. And then we have found that the different structure between polycrystalline tungsten and nanocrystalline tungsten film lead to differences in density and size of blisters [35]. Besides, our group has investigated the different behaviors of blistering on various tungsten samples with different microstructures; the results show that the microstructure in tungsten materials plays a dominant role in the blistering after irradiation [20,30,36]. However, in recent years, few researches have been studied about the effect of pre-existing internal stress in material on blistering behaviors after helium irradiation. In this work, we have prepared the W films with a thickness of about 10 μm deposited by magnetron sputtering and some films were annealed at 1273 K for 1 h. Subsequently, both as-deposited and pre-annealed films were exposed to helium ions with the energy of 60 keV

* Corresponding author at: Department of Physics, Beihang University, Beijing 100191, PR China.
E-mail address: kgzhu@buaa.edu.cn (K. Zhu).

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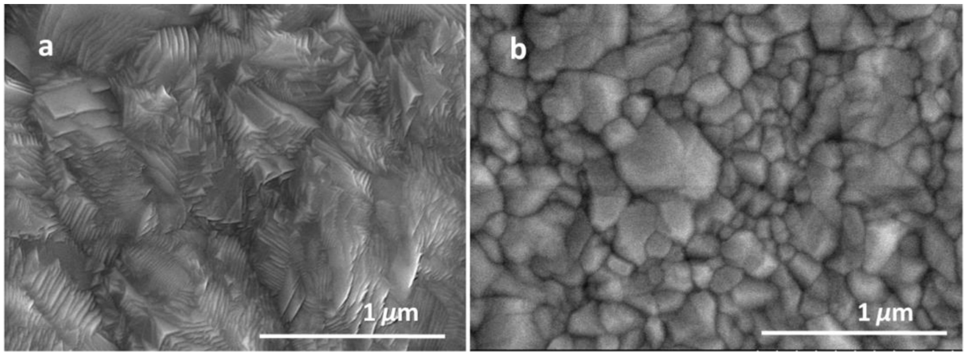

Fig. 1. SEM images of morphology of (a) as-deposited and (b) pre-annealed films.

and fluence of $1.0 \times 10^{22}\,m^{-2}$ at room temperature. There is a difference in blistering behavior between both the films before and after annealing. It is founded that the different compressive stress in W film leads to the different behaviors of blistering after helium irradiation.

2. Experiments

2.1. W films preparation

W films with a thickness about 10 μm were deposited on- polished tungsten substrates using a commercial magnetron sputtering device (KYKY MP 650-A). The tungsten substrates with size of 4 mm × 4 mm and thickness of 1.5 mm were cut from a polycrystalline tungsten plate with the purity of 99.99%. The tungsten plate was purchased from Advanced Technology & Materials Co., Ltd (AT&M). Mirror-like surface was obtained by mechanical and electrochemical polishing. Before sputtering, all polished substrates were ultrasonically cleaned with acetone, alcohol and deionized water for 15 min respectively.

The sputtering system was pumped down to a base pressure of less than 5×10^{-4} Pa and deposition was performed in argon atmosphere of 1 Pa. Prior to the deposition the target was bombarded by argon plasma for 10 min in order to remove impurities from the surface. During deposition the DC power applied to the tungsten sputter target with the purity of 99.95% was kept constant at 100 W. At these settings the deposition rate of tungsten is about 1 μm per hour. No extra substrate bias was applied. In order to enhance adhesion and prevent delamination, the substrate was heated up to 773 K during the sputtering and kept constant for the first 2 h, and then the temperature was reduced to 523 K to finish the next 8 h.

After deposition, a tactile profilometer (DEKTAK 6 M, Veeco) was applied to measure the thickness of deposited films. For measuring the density of the films, the mass change before and after deposition was determined by a microbalance with an accuracy of 0.1 mg, which was mentioned in Ref. [31]. Based on the weight and thickness of W film, the density can be figured out, which is closed to 90% of bulk tungsten density. Furthermore, some films were annealed at 1273 K for one hour.

2.2. Sample analysis before irradiation

Scanning electron microscope (SEM) (HITACHI S-4800, at Beijing Center for Physical and Chemical Analysis) was used to measure the morphology of W films. The secondary electrons produced by a 10 keV electron beam were detected by the in-lens detector system. The crystallographic textures of W films were determined by x-ray diffraction (XRD) (Shimadzu XRD-6000 Type) using a Cu Kα source with the wavelength of 0.154 nm. Diffraction patterns were acquired from 20° to 90° to study the microstructure of films and orientation of crystal grains. In addition, the total stresses in W films parallel to the surface before and after annealing were carried out by Formula (1) according to the inter-planar spacing measured by XRD.

2.3. Helium irradiation and analysis

The irradiation experiments were performed at 320 keV multi-discipline research platform for Highly Charged Ions equipped with an Electron Cyclotron Resonance (ECR) plasma source in the Institute of Modern Physics, Chinese Academy of Sciences (IMP, CAS), in Lanzhou. The as-deposited and annealed tungsten films were simultaneously irradiated with 60 keV He^+ ions with a fluence of $1.0 \times 10^{22}/m^2$ at room temperature, and the average flux was $2.44 \times 10^{17}/(m^{-2}\,s^{-1})$. The base pressure of the chamber was better than 5×10^{-4} Pa.

The blisters formed on the surface of the tungsten films after helium irradiation were observed by scanning electron microscope (HITACHI S-4800 Type, at China Telecommunication Technology Labs, CTTL). Meanwhile, tilting observation with the dip angle of 80° was performed in order to analyze the geometries of the blisters.

3. Results and discussion

3.1. Morphologies and microstructures of W films before irradiating

As seen in Fig. 1 (a), the surface of as-deposited film has a distinct nanostructure like a ridge, which is characterized as the piles of platelets with random orientation on the surface. This "nano-ridge" structure is a characteristic of high melting-point metal film deposited under relatively low temperature [37,38]. Fig. 1 (b) shows the morphology of pre-annealed film, and the grain size is in the range of tens to hundreds nanometers. As seen in Fig. 1, a considerable change of the surface morphology is observed after thermal treatment.

In order to compare the grain size of as-deposited and pre-annealed films, the both samples were electro polished with the electrolytes containing 2.5% sodium hydroxide. The differences in the morphologies of smooth surfaces on both films are shown in Fig. 2. Although there is little difference in the grain size of the films, it is worth noting that the grain shape of the pre-annealed film seen in Fig. 2 (b) was more regular and distinct than that of the as-deposited film seen in Fig. 2 (a). The small difference in grain size indicates that the W films did not significantly recrystallize during thermal treatment at 1273 K, because the annealing temperature is lower than the recrystallization temperature of tungsten, which is about 1773 K [39].

The x-ray diffraction patterns of as-deposited and pre-annealed W films are shown in Fig. 3. They indicate that both W films have the body centered cubic structure (BCC) and are dominated by α-phase tungsten without any β-W and tungsten oxide. The both W films show three diffraction peaks located in the vicinity of 40°, 58° and 73°, corresponding to a lattice plane with indices of (110), (200) and (211), respectively. The sharp peaks indicate a high degree of crystallinity of the W thin films. As shown in Fig. 3, the samples prepared on tungsten substrates show strong (211) preferred orientation. However, there seems to be little difference in the preferential orientation of the films before and after annealing at 1273 K.