Nuclear Instruments and Methods in Physics Research B 358 (2015) 124–130

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Multi-scale characterization of surface blistering morphology of helium irradiated W thin films

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ARTICLE INFO

Article history:
Received 16 February 2015
Received in revised form 1 June 2015
Accepted 2 June 2015
Available online 22 June 2015

Keywords:
Surface morphology
Irradiation
Thin film
Multi-scale

ABSTRACT

Surface blistering morphologies of W thin films irradiated by 30 keV He ion beam were studied quantitatively. It was found that the blistering morphology strongly depends on He fluence. For lower He fluence, the accumulation and growth of He bubbles induce the intrinsic surface blisters with mono-modal size distribution feature. When the He fluence is higher, the film surface morphology exhibits a multi-scale property, including two kinds of surface blisters with different characteristic sizes. In addition to the intrinsic He blisters, film/substrate interface delamination also induces large-sized surface blisters. A strategy based on wavelet transform approach was proposed to distinguish and extract the multi-scale surface blistering morphologies. Then the density, the lateral size and the height of these different blisters were estimated quantitatively, and the effect of He fluence on these geometrical parameters was investigated. Our method could provide a potential tool to describe the irradiation induced surface damage morphology with a multi-scale property.

1. Introduction

Surface blistering is one of the most important modes of material failures under He irradiation environment, which results in the modification of surface physical–chemical properties and the loss of structural integrity, and ultimately degrades material component performance [1–3]. During recent decades, numerous studies have been devoted to this surface damage and its underlying microscopic mechanisms, in which several blistering theories including gas pressure model and lateral stress model are developed [4,5]. In these studies, a key strategy is to obtain the geometrical parameters such as the lateral size, the height and the density of surface blisters, so as to fully understand the blistering process. It is thus crucial to quantitatively characterize the surface blistering morphology of He irradiated materials.

Nanostructured thin films are attracting more and more interest as an excellent irradiation tolerant material, because the films have abundant interior interfaces that can act as sinks to annihilate or disperse the irradiation induced defects [6–8]. Under He irradiation impact, thin films also exhibit surface blistering like the bulk materials due to the nucleation and growth of He bubbles. However, the surface blistering of thin film is more complex, which could be additionally influenced by the film/substrate interface failure. The introduction of He into thin film often leads to an enhancement of compressive stress. The subsequent stress relaxation may occur in the form of the film/substrate interface delamination, which will induce the formation of surface blisters [9,10]. This kind of surface deformation accompanied by the intrinsic He blisters could yield a complicated film surface morphology, which usually exhibits a multi-scale property. Therefore, it is intensively desirable to develop an effective method of distinguishing and characterizing these different blistering morphologies so as to understand their formation mechanisms.

Unfortunately, most traditional characterization methods such as surface roughness measurement and statistical function analysis, which only obtain the parameters reflecting the global surface morphology, fail to separately describe these different surface blisters [11]. In the 1980s, Mallat introduced a multi-resolution space-scale (or frequency) transformation method, called wavelet transform (WT), which can be used to analyze multi-scale space signals [12]. During recent years, the WT approach has been successfully applied to characterize the material surface morphology with a multi-scale property [13–15]. This approach can distinguish the surface features with different characteristic sizes and/or shape. Moreover, it can separately extract and quantitatively estimate these different surface features. A more accurate and elaborate characterization of surface morphology is thus realized.

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http://dx.doi.org/10.1016/j.nimb.2015.06.010
0168-583X/

Based on its advantages, WT could provide a powerful tool for characterizing the complicated blistering morphology of He irradiated film.

In the present study, W thin films deposited by magnetron sputtering were irradiated by 30 keV He ion beam with different fluencies. The interest of this material lies in its wide range of applications, such as plasma facing components [16], radiation shielding coating [17], etc. After film irradiation, obvious surface blistering morphologies were observed; especially a multi-scale blistering morphology emerges for higher He fluence. Then the WT approach was for the first time proposed to quantitatively characterize this complicated surface morphology. Several important parameters including the density, the lateral size and the height of these blisters were studied quantitatively and their relation to He fluence was also analyzed.

2. Experimental details

W thin films with a thickness of 200 nm were deposited on Si substrates covered with a thin Ta buffer layer in a magnetron sputtering system (base pressure of 1.0×10^{-3} Pa). During W film deposition, the substrate temperature was held at room temperature. The Ar pressure and the sputtering power were 0.3 Pa and 200 W, respectively. The sample was placed 6 cm away from the target and its surface normal was perpendicular to the target surface. The sample holder was operated with a rotation of 20 rpm to assure the uniformity of film thickness. After film deposition, the samples were irradiated with 30 keV He ions with four fluencies: 5×10^{20}, 1×10^{21}, 5×10^{21}, and 8×10^{21} He ions/m^2. The He dose rate was about 1.67×10^{18} He ions/m^2 s and the incident direction of He ion beam was perpendicular to the sample surface. The film samples were mounted on the water-cooled substrate holder and the temperature rise of the samples was less than ~300 K during He irradiation. Meanwhile, the stopping and range of ions in matter (SRIM) analysis was used [18]. As can be seen in Fig. 1, the depth of irradiation damage and He distribution was smaller than the W film thickness (200 nm), implying the direct effect of He implantation on the underlying Ta buffer layer and Si substrate can be negligible.

Film phase structure was investigated by grazing incidence X-ray diffraction (GIXRD) measurement with Cu-K_α radiation (λ = 0.15406 nm). The incidence angle was chosen as 1.3° to ensure that the X-ray is only reflected from W films. Film surface and cross-section morphologies were studied with scanning electron microscope (SEM). To quantitatively characterize surface blistering morphologies, atomic force microscopy (AFM) was performed with tapping mode under atmospheric conditions. The AFM provided surface height data with a resolution of 512 × 512 pixels. A planar background was subtracted from the data to compensate for the tilt of the sample relative to the scanning plane. The results were checked for reproducibility by imaging several regions of the same sample.

3. Results and discussion

3.1. Phase structure and grain size

Fig. 1 shows typical GIXRD patterns of W films. All W films exhibit polycrystalline structure nature. For as-deposited sample, several obvious peaks can be indexed as the orientations (200), (211), (320), and (321) of β-W phase. Two slight peaks of (200) and (211) orientations of α-W phase can be also detected. Meanwhile, the peak located about 40° could be indexed as the mixture of β-W(210) and α-W(110) orientations. Obviously, it shows that the as-deposited W film is composed of α-W and β-W phases. This phase mixture was demonstrated in the previous studies on vapor-sputtered W films, which is related to the deposition conditions such as the sputtering gas pressure, sputtering powder and the burial of impinging impurities (e.g., O, Ar) [19,20]. For irradiated films, with increasing He fluence, the peaks of β-W phase weaken gradually and those of α-W phase become stronger and stronger. Additionally, as shown in the inset of Fig. 1, the major peak located at about 40° also shifted towards a higher diffraction angle (Standard peaks of β-W (210) = 39.88° and α-W (110) = 40.26°, JCPD). These results indicate that the He irradiation leads to a transition from β-W phase to α-W phase. This phenomenon was already observed in Wang et al.'s study [21]. They suggested that this transition behavior arises from the fact that α-W is thermal stable phase as compared to β-W. Further, we approximately estimate the film grain size with Scherrer formula [22]. The results showed that the average grain size of W films gradually increases from 9 nm to 25 nm with the increase of He fluence. It should be noted that, though the He irradiation induced the grain growth, the film grains are still tiny (not larger than 25 nm nominally) (See Fig. 2).

3.2. Surface damage morphology

To study the irradiation induced surface damage, surface SEM observations were carried out, as shown in Fig. 3(a)–(c). For the as-deposited W film, its surface is very smooth and no well-defined surface fluctuations are observed, which arises from the fine grain structure of the film. After He irradiation, the change

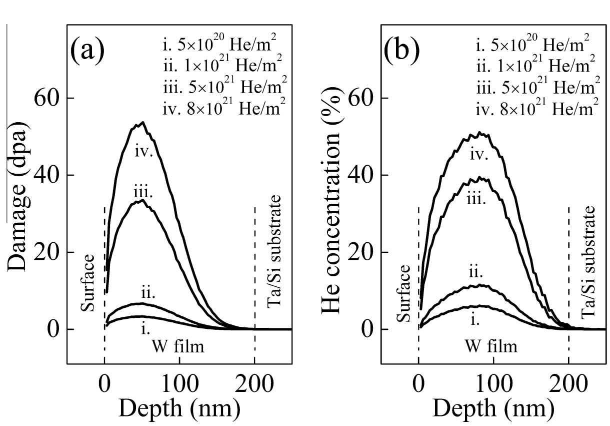

Fig. 1. The SRIM results of (a) irradiation damage level and (b) He concentration as a function of depth in W film.

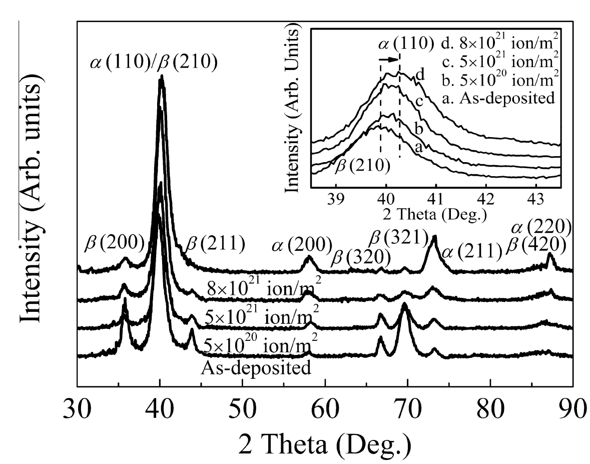

Fig. 2. Typical GIXRD patterns of as-deposited and irradiated W films subjected to different He irradiation fluences.