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Helium-induced hardening effect in polycrystalline tungsten

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ABSTRACT

In this paper, helium induced hardening effect of tungsten was investigated. 50 keV He^{2+} ions at fluences vary from 5×10^{15} cm^{-2} to 5×10^{17} cm^{-2} were implanted into polycrystalline tungsten at RT to create helium bubble-rich layers near the surface. The microstructure and mechanical properties of the irradiated specimens were studied by TEM and nano-indentor. Helium bubble rich layers are formed in near surface region, and the layers become thicker with the rise of fluences. Helium bubbles in the area of helium concentration peak are found to grow up, while the bubble density is almost unchanged. Obvious hardening effect is induced by helium implantation in tungsten. Micro hardness increases rapidly with the fluence firstly, and more slowly when the fluence is above 5×10^{16} cm^{-2}. The hardening effect of tungsten can be attributed to helium bubbles, which is found to be in agreement with the Bacon-Orowan stress formula. The growing diameter is the major factor rather than helium bubbles density (voids distance) in the process of helium implantation at fluences below 5×10^{17} cm^{-2}.

1. Introduction

Tungsten (W) and tungsten alloys are considered as leading candidate materials for facing plasma components in ITER and other future fusion reactors mainly because of its high melting point, low sputtering yield and high thermal conductivity [1,2]. Tungsten has to bear bombardment of high fluence of helium ions and other kinds of irradiation [3]. Yet current divertor designs also require the use of tungsten in structural components [4]. In both cases, high levels of displacement damage and helium concentration can be produced in tungsten materials. One of the key issues of tungsten materials is the understanding of helium-induced embrittlement and hardening effects [5]. It has been reported that self-ion implantation can lead to the increase of micro hardness in tungsten and tungsten 5-wt% rhenium alloy, and it can be attributed to dislocation loops and small rhenium clusters [5], but there is few experiment focuses on helium induced hardening effect in tungsten.

However, the presence of helium atoms is widely believed to induce hardening effect in metals. P. Lei, Y. Dai and co-works [6–8] have reported helium implantation can cause remarkable increase of micro hardness in ferritic/martensitic steels. Q.M. Wei studied the helium induced hardening effect in nanoscale V/Ag multilayers [9]. N. Li studied the hardening mechanisms in high dose helium ion implanted Cu and Cu/Nb multilayer thin film [10]. They all proposed that high density of helium bubbles was the reason of hardening effect. While helium bubbles have been observed in tungsten materials too [11], theoretically, bubbles in tungsten should bring out the increase of hardness too.

In this work, we designed the experiment to explore helium-induced hardening effect in polycrystalline tungsten. After implantation of helium atoms, the changes of microstructure and mechanical properties were studied.

2. Experimental

The specimen used in the present work was high-purity (99.95%) polycrystalline tungsten produced by Goodfellow Cambridge Limited in form of sheets with thickness of 1 mm. The samples were cut into 10×10 mm^2 size and mechanically polished to mirror-like finish, and then annealed at 1200 K for 1 h in high vacuum environment with a background pressure of 1×10^{-5} Pa to release internal stresses and impurity introduced by polishing processes.

In order to obtain a helium rich region with a relatively accurate helium concentration at an appropriate depth, 50 keV He^{+} ions was implanted into tungsten by an ion implantation system based on an ECR ion source at Peking University [12]. The temperature was kept below 50 °C by using water-cooling system attached to the back of samples and measured by a thermocouple plugged into a hole at the flank surface of sample. Five fluences of helium (5×10^{15}, 2×10^{16}, 5×10^{16}, 2×10^{17} and 5×10^{17} cm^{-2}) were

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implanted into tungsten samples, which confirmed helium rich layers with peak concentration of 4.86×10^3, 1.94×10^4, 4.86×10^4, 1.94×10^4, and 4.86×10^5 appm respectively, according to the full damage cascade simulations of SRIM-2013. It should be noted that the calculation was conducted without considering the influences of the helium diffusion. All implantation was completed at the same He^{2+} flux about 4.5×10^{13} ions·s^{-1} cm^{-2}. The depth profile of helium concentration at fluence of 5×10^{17} cm^{-2} is shown in Fig. 1. Helium atoms were stored in a near surface layer with a thickness about 300 nm, and the depth of concentration peak was about 125 nm.

The cross-sectional transmission electron microscope (TEM) samples were prepared with the following method: (1) stick the samples together face to face, (2) grind the samples perpendicular to the surface to about 30 μm thick, (3) mill the sample to get a hole in the sticky sew using a ion mill, (4) electrolytically polish for about 0.5–0.8 s in NaOH solution with mass fraction of 1% to decrease damage caused by ion milling process. The samples were observed with Tecnai F20 high-resolution TEM to reveal helium bubbles. Nano-indentation measurements were carried out in the Continuous Stiffness Measurement (CSM) mode which enables a continuous measure of hardness during loading, and not just at the point of initial unload, using a Berkovich indenter on Nano-indenter G200 produced by Agilent Technologies. 15 points were indented for each sample to obtain the average hardness value, and the maximum indentation depth is about 600 nm.

3. Results and discussion

3.1. Helium bubbles

Under TEM bright-field view, a distinguished bubble-rich layer appears in cross-section each He ions implanted sample. As shown in Fig. 2, the thickness of the layer expands from 196 nm at the lowest He^{2+} fluence (5×10^{15} cm^{-2}) to 301 nm at the highest fluence (5×10^{17} cm^{-2}).

Refer to the He depth profiles calculated by SRIM2013 (Fig. 1) and the thickness of bubble rich layer (Fig. 2), it can be deduced that the lowest He concentration to form distinguished bubbles is 1.5×10^3 appm for our specimens. It is widely understood that helium atoms implanted into tungsten will be caught by trap sites such as vacancy defects, then more and more helium atoms will be attracted into the trap site and helium bubbles will be formed [13]. So there must be enough helium atoms around a trap site for the nucleation and growth of bubble. For example, as in Fig. 1, SRIM calculation confirms that the concentration of helium atoms is

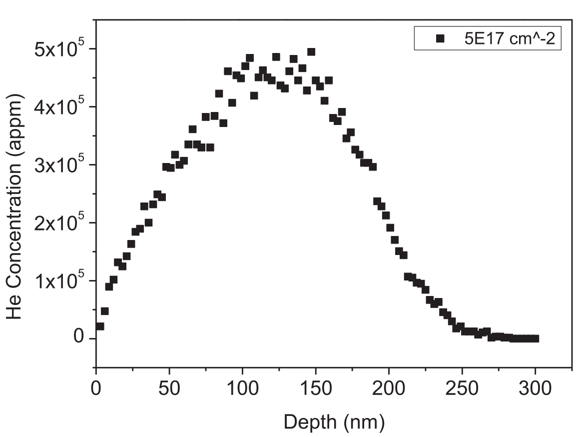

Fig. 1. Distribution of helium concentration in W irradiated with 50 keV He^{2+} at the fluence of 5×10^{17} cm^{-2}, data was calculated by SRIM-2013.

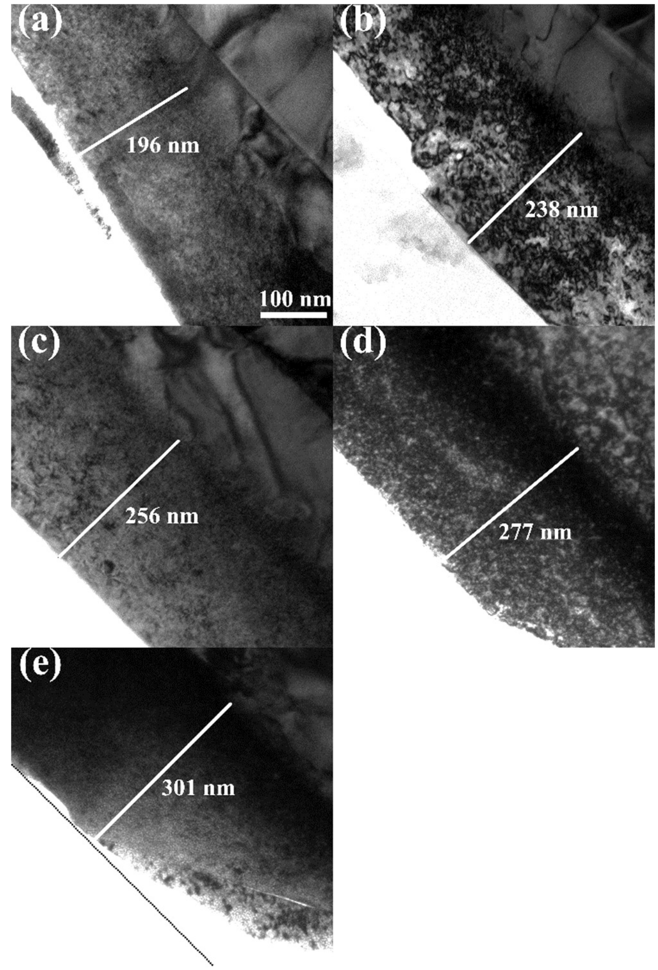

Fig. 2. Cross-section Bright-field TEM images of tungsten samples irradiated with He^{2+} ions at different fluences of (a) 5×10^{15} cm^{-2}, (b) 2×10^{16} cm^{-2}, (c) 5×10^{16} cm^{-2}, (d) 2×10^{17} cm^{-2}, and (e) 5×10^{17} cm^{-2}. Straight lines perpendicular to the surface are drown from the surface to the bottom of the bubble-rich layers.

lower than 1.5×10^3 appm in the tail deeper than 300 nm, so no bubble is observed beneath the depth of 300 nm because the helium concentration is not enough for bubble formation. While the fluence increases, the He concentration tail uplifts synchronously, so that bubbles can form in wider tail area, the bubble-rich layer becomes thicker. However, helium bubbles are observed near the surface at the minimum fluence, even the concentration of helium atoms is lower than that in the tail area. As shown in Fig. 1, the concentration of helium atoms rises very steeply in the surface area, and it has been reported that helium atoms tend to diffuse towards the surface [14], so helium atoms in the surface area may be enough for the bubbles formation earlier than that in the tail area.

SRIM calculation result shows that the maximum of He concentration in tungsten for 50 keV He ions appears at 125 nm beneath the surface. We take high resolution TEM observation in this area, and the images are shown in Fig. 3. Fig. 3(a) demonstrates that helium bubbles have been formed at the fluence of 5×10^{15} cm^{-2}. With the rise of fluence, helium bubbles grow up clearly (see Fig. 3(b)–(e)).

Average diameter of helium bubbles in each sub image of Fig. 3 is shown in Table 1. At fluence of 5×10^{15} cm^{-2}, average diameter of helium bubbles is 0.4 nm, which is approximate to the lattice distance of BCC tungsten crystals. It means that bubbles shown