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Effect of crystal orientation on hardness of He^+ ion irradiated tungsten

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ABSTRACT

The effect of crystal orientation on hardness in the as-received, irradiated and post-irradiation annealed tungsten samples was investigated using a nanoindenter. An effective irradiation method of He^+ ions with a series of energy degraded from 200 keV to 20 keV was used to continuously irradiate polycrystalline tungsten at room temperature in order to obtain a relatively homogenous displacement damage and helium concentration from sample surface to a desired depth at a NEC 400 kV ion implanter. Some irradiated samples were then annealed at 900 °C. He^+ ion irradiation induced hardness increase, oppositely for the post-irradiation annealing effect. Meanwhile, the hardness of the irradiated samples was decreased sharply in the initial stage of annealing from 0 to 1 h, and then slowed down in the latter stage from 1 h to 3 h. Crystal orientation had an obvious effect on the nanoindentation hardness. The (001)-oriented grains had highest hardness at the as-received and irradiated samples. During the annealing process, the hardness in the irradiated grains with (111) crystal orientation decreased more quickly than that in the (001)-oriented grains. The mechanism of the effect of crystal orientation on hardness was analyzed and discussed.

1. Introduction

Pure tungsten and tungsten alloys have been considered as potential candidates for first wall materials in fusion reactors due to their high temperature properties, very low sputtering yields and low evaporative coefficients [1–4]. The reaction of deuterium and tritium is most likely to be achieved for a commercial fusion reactor and it will produce α particles that retaining in the plasma and providing the primary heating source. The energetic α particles will induce a considerable helium concentration in the damage layer of tungsten [5–6]. The diffused-in helium along with displacement damage induced by high dose of neutrons and other energetic ions will degrade the properties and performance. Irradiation hardening, embrittlement and swelling will be induced, which due to a large amount of defects generated in tungsten such as vacancies, dislocations, interstitials and bubbles. So, the evolution of helium in a tungsten and its relative research have been research hotspot.

In the past decades, the effect of helium irradiation on tungsten has been widely investigated [7–12], which mainly focused on the formation and evolution of complex defects under various kinds of irradiation conditions. Besides, the surface morphologies including blister, sputtering and nanostructures were also studied with a low energy helium ion implantation [13–15]. However, a few literatures concerned the mechanical properties after helium ion irradiation or then post-irradiation annealing. Gibson [16] reported the relationship between the irradiation hardening and the irradiation temperature of the tungsten implanted by 2 MeV He^+ ions with ~600 appm peak concentration. The implantation temperature was ~75 °C. Armstrong [17] analyzed the load-displacement curves of original state and W^+ ion implanted tungsten. The implantation temperature was approximately 300 °C. Zhang [18] compared the hardening behavior of the as-received and irradiated tungsten. Tungsten was irradiated with 6.4 MeV Fe^{3+} ions at 300 °C, 700 °C and 1000 °C. Hasegawa [19] studied the microstructure characteristics and the hardening behavior of pure W and W-Re-Os alloys irradiated with neutron up to 1.54dpa at a temperature range between 400 °C and 800 °C. However, the effect of crystal orientation on mechanical properties was almost not considered at their experiments. During the irradiation and post-irradiation annealing, temperature effect is a key factor that controlling the evolution of the irradiation-induces defects such as vacancies, voids, interstitial, gas bubbles. These defects will move, gather and grow, which directly affect the mechanical properties of tungsten. In a fusion reactor, tungsten is considered as a potential candidate for first wall material that will service at a high temperature environment. Therefore, the evolution of mechanical proper-

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ties such as hardness at high temperature conditions is very important for the life assessment of tungsten in service. However, it is difficult to obtain the hardness of tungsten during both irradiation and high temperature. Therefore, the evolution of hardness during the post-irradiation annealing is a suitable method to assess the mechanical properties of tungsten.

In fact, crystal orientation plays a very important role in the irradiation behaviors of tungsten. After bombardment with 80 eV helium ions at 1130 °C, various surface morphologies such as smooth, wavy, pyramidal and terraced waves were formed on polycrystalline tungsten surface, which depend on crystal orientation [20]. Liu [21] found that the density of irradiation-induced blisters was larger on (1 1 1) crystal plane than that on (0 0 1) crystal plane after irradiation with 50 keV helium ions with a fluence of 3.55×10^{18} ions/cm^2 and 35 keV proton with a fluence of 3.4×10^{19} ions/cm^2. In our previous research work [22], grain with a (0 0 1) direction parallel to the Ga^+ ion beam always maintained a much smoother surface morphology with less mass removal after ion bombardment, indicating a lower sputtering yield. Therefore, it is important to consider the effect of crystal orientation on the mechanical properties of tungsten after helium ion irradiation and then post-irradiation annealing.

The method using charged-particle radiation damage to simulate neutron radiation damage has been extensively accepted in studying radiation behavior of target materials. However, a single-energy ion beam was widely used by many researchers in studying irradiation-induced mechanical behavior [16–18], even in researching microstructure characteristics [23,24]. Although a lot of results have been obtained, ion irradiation with single energy has a problem that it will induce a normal distribution of displacement damage and ion concentration along with irradiation depth, not a homogenous manner, especially for heavy ions. However, in fact, displacement damage, helium and hydrogen in the target materials suffered from neutron irradiation are homogeneous. So, some existing results are inexact in describing and explaining the irradiation-induced change of mechanical properties if the nonuniformity of displacement damage and ion concentration are not considered after a single-energy ion beam irradiation.

Therefore, in the present work, helium ions with a series of energy were used to irradiate polycrystalline tungsten in order to obtain a relatively homogeneous displacement damage and helium concentration from sample surface to a desired depth. The hardness of tungsten grains with different crystal orientations at the conditions of the as-received, irradiated and post-irradiation annealed status were measured by a nanoindenter instrument. The mechanism of the effect of crystal orientation on hardness was also analyzed and discussed.

2. Experiments

The polycrystalline tungsten (purity > 99.98 wt.%, provided by Xiamen Tungsten Corporation of China) fabricated by powder metallurgy was used as the original material in the present work. The samples of 10 mm × 10 mm × 3 mm in dimensions were first cut from the as-received round rod by a precision diamond knife-cutting machine and then grinded by SiC sandpaper from 320 to 2500 grid, mechanically polished using 3–0.1 μm diamond paste. After that, the samples were finally electrochemical polished by a 2% NaOH aqueous solution to remove surface damage for electron back-scattered diffraction (EBSD) test and then ion irradiation experiment. The surface roughness of these polished tungsten samples was about 3 nm, which was measured in air at room temperature using atomic force microscopy (AFM) with a BRUKER Dimension Icon AFM.

Because the single-energy He^+ irradiation induced the nonuniformity of displacement damage and helium concentration along with irradiation depth in tungsten sample, such as 200 keV He^+ ion irradiation with a fluence of 7.0×10^{16} He^+/cm^2 as shown in Fig. 1(a). The displacement damage and helium concentration were simulated using the Stopping Range of Ions in Matter (SRIM) code in quick Kinchin-Pease mode. It is difficult to obtain surface hardness in tungsten gains with different crystal orientations at the conditions of homogeneous displacement damage and helium concentration. Therefore, in the present work, helium ions with a series of energy and corresponding ion fluence were used to continuously irradiate the polished tungsten in order to obtain a relatively homogenous displacement damage and helium concentration from sample surface to a desired depth, which was simulated by SRIM code in quick mode and shown in Fig. 1(b). He^+ ion irradiation was conducted in a vacuum of $\sim 10^{-5}$ Pa at room temperature using a NEC 400KV ion implanter at our research group. The ion flux was kept at about 2.0×10^{12} ions/cm^2·s to prevent excessive beam heating. The schedule of the helium-ion irradiation was shown as:

- 200 keV: 3.7×10^{16} He^+/cm^2 fluence;
- 150 keV: 2.5×10^{16} He^+/cm^2 fluence;
- 100 keV: 1.85×10^{16} He^+/cm^2 fluence;
- 50 keV: 1.85×10^{16} He^+/cm^2 fluence;
- 20 keV: 1.0×10^{16} He^+/cm^2 fluence.

The helium ions with 200 keV were first used to irradiate the samples. And then ion energy was degraded until 20 keV. According to the simulation results of SRIM 2008 software with quick mode, the helium-ion irradiation with above energy and fluence caused a 3.5% platform concentration of helium ions in the tungsten sample at the range of 70–400 nm depth. The platform displacement damage was about 0.7dpa at the range of 50–300 nm depth. The atom displacement threshold energy (E_d) of tungsten was designed to be 90 eV.

Before He^+ ion irradiation, orientation imaging microscopy (OIM) based on EBSD of the polished tungsten was performed using a TESCAN MIRA3 field emission scanning electron microscopy (FEG SEM). The crystal orientations of tungsten grains were then obtained by processing these data. The hardness of the tungsten grains with different crystal orientations at the conditions of the as-received, irradiated and post-irradiation annealed status were measured using a TI-950 TriboIndenter system with standard Berkovich tips in a load controlled mode. The nanoindentation test was performed at room temperature.

3. Results and discussions

The crystal orientation image of the as-polished tungsten is obtained by processing EBSD data as shown in Fig. 2(a). Various colors in the image represent tungsten grains with different crystal orientations. From the original test data, we can find tungsten grains with planes parallel to the sample surface in the interested area such as (1 0 0)-, (1 1 0)- and (1 1 1)-oriented grains. Meanwhile, the crystal size can be also obtained from the EBSD image, which is in the range of tens of microns to hundreds micrometers. However, we only focused on the hardness evolution of tungsten grains with crystal size over 100 μm during the ion irradiation and annealing process. The reasons are shown as: (I) reducing the plastic-deformation affected zone among multi-point measurements in a grain. To avoid the overlap of deformation zones, the distance between two adjacent indentations should be more than 10 μm. Therefore, tungsten grains with large size were selected. In the present work, the average hardness was used from five measurement points at same grain; (II) making sure that the measuring position

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