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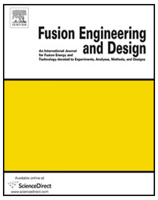

Full Length Article

Vacancy like defects and hardening of tungsten under irradiation with He ions at 800 °C

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HIGHLIGHTS

- The bulk-equivalent hardness values and their increments of tungsten irradiated with 200 keV He^+ at 800 °C were investigated.
- Vacancy like defects in tungsten induced by 200 keV He^+ at 800 °C were investigated.
- The irradiation induced hardening behavior in tungsten could be explained by the dispersed barrier hardening model.
- The irradiation induced hardening in tungsten was consistent with the vacancy like defects.

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ABSTRACT

The irradiation induced hardening and vacancy like defects are investigated as a function of irradiation fluences with 200 keV He ion implantation at 800 °C in this work. Nano-indentation tests show the irradiation induces hardening and the hardness values increase with increasing irradiation fluences. Doppler broadening spectroscopy analyses of slow positron annihilation find that a large amount of vacancy like defects are produced under irradiation. When the He/dpa ratio is less than about 2.2% He/dpa, the vacancy like defects include mainly empty vacancy clusters and loops, and He_nV_m complexes. When the He/dpa ratio is more than about 2.2% He/dpa, the vacancy like defects become mainly He_nV_m complexes with increasing irradiation fluences. Meanwhile, these complexes increase in size and He/V ratio while the irradiation fluences increase. Based on the Orowan hardening mechanism, we discuss the relationship between irradiation induced hardening and defects under indentation, especially the contribution of He_nV_m complexes to the hardening.

1. Introduction

Nuclear fusion energy is one important method to solve the future energy of the world. One most important issue in the development of future reactors is the material problem, such as the selection and estimation of divertor and structural components. Tungsten is a potential candidate divertor material, due to its high melting point, high thermal conductivity, low activation, low sputtering and low H isotope retention etc. The divertor component, i.e. tungsten, serves under severe environments such as the bombardment of hydrogen isotopes (D and T) and helium (He) plasma, the irradiation of neutron and high temperatures etc. Irradiation induced damages such as the displacement damage (up to 15 dpa within 5 years in DEMO-like reactor for the armor materials of divertor) and He atom depositions often influence the microstructure and mechanical properties of materials and possibly relates to the safe operation of reactors. He ion irradiation has been testified to be an effective means to simulate the He effects in materials under the irradiation in reactors. There were lots of He irradiation experiments on many kinds of materials such as low activation ferrite/martensite steels [1–4], tungsten [5–11] and Si [12]. The researches showed that He irradiation produced dislocations, and vacancy like defects such as vacancies (Vs), complexes of He and

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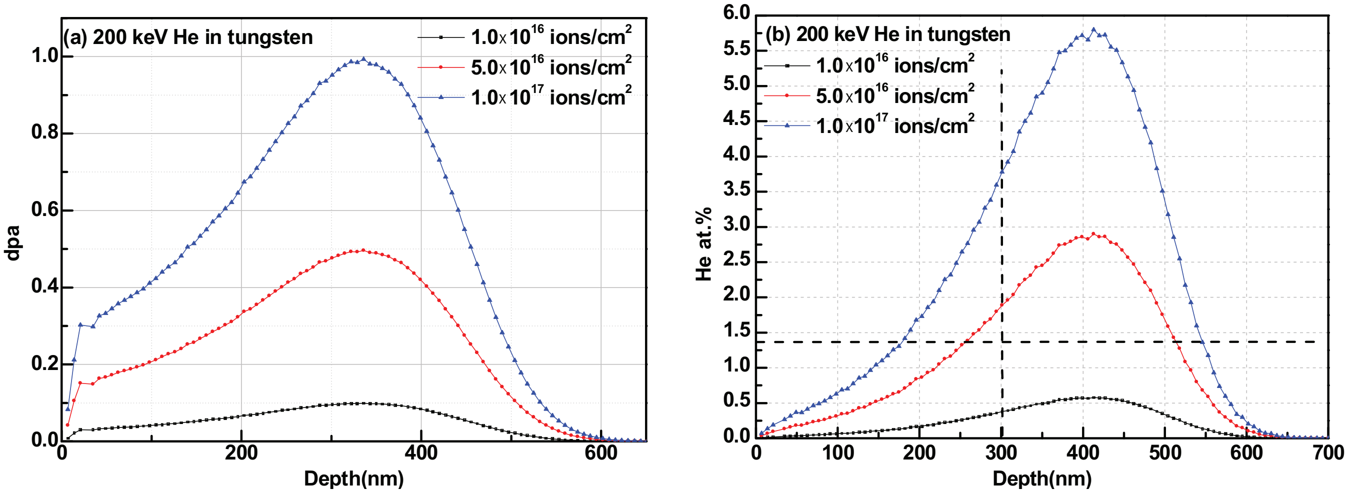

Fig. 1. depth profiles of (a) dpa and (b) He concentration (C_{He}) for irradiated tungsten with 200 keV He to three fluences calculated with SRIM.

vacancies (He_nV_m, in which n and m are the number of He atoms and vacancies, respectively), and He bubbles. Vacancy like defects, especially the He_nV_m complexes, play a very important role in the degradation of materials' mechanical properties [5,8,13] and surface morphology [14], because the vacancies are very attractive to He atoms [15–17] and the complexes are the nuclei of He bubbles. The fundamental understanding of He irradiation induced vacancy like defects and their effects on hardening in tungsten is very important in understanding the mechanical behavior under irradiation.

The investigation about vacancy like defects and their effects on hardening of tungsten induced by He irradiation under different temperatures has been investigated by several papers. Monovacancies were introduced by 800 keV He irradiation in the track region of tungsten [10]. They became mobile from about 200 °C, which gave rise to the formation of vacancy clusters [18]. The complexes of He_nV_m formed by 350 keV He ion irradiation in the near surface of tungsten. The ratios of He/V in complexes at 600 °C were lower than the ratios at room temperature (RT). The hardness at 600 °C was also lower than the hardness at RT [5]. However, the studies at higher temperatures are rare but important because the operation temperature is in a wide region of about 400–1400 °C [6] and the nature of vacancy like defects are different at different temperatures.

Recently developed nano-indentation technology (NIT) has been widely used to characterize the hardness in the thin damage layer induced by ion irradiation [3,5,7,19–22]. The positron annihilation technology was an effective method in detecting the vacancy like defects because of its sensitivity on point defects with atomic scales [10]. Many works related the S parameters derived from the Doppler Broadening Spectrum (DBS) of positron annihilation with the nano-hardness from the NIT to investigate the effects of vacancy like defects to nano-hardness under irradiation [3,5,7,19,20].

In this work, we choose pure tungsten as the study objects. To investigate the fundamental process and effects of He_nV_m complexes on hardening at higher temperatures, 200 keV He ion irradiation in tungsten were completed at 800 °C which is in the working temperature range of divertor. Three different irradiation fluence of 1.0×10^{16}, 5.0×10^{16}, 1.0×10^{17} ions/cm^2 were completed to get a relatively low displacement damage and He centration in shallower depth, which are favorable in the investigations of DBS and NIT. The changes of hardness and vacancy like defects after irradiation are investigated by the NIT tests and the DBS measurements of slow positron annihilation, respectively. The influences of irradiation induced defects on the hardness is discussed.

2. Experiments

The investigated tungsten samples were cut from the high pure tungsten plate provided by Advanced Technology & Materials Co., Ltd. The plate was produced by the powder metallurgy method followed by warm rolling at 1200 °C to 75% reduction in thickness (final thickness ~3 mm). The elongated grains varied in size from several to a few hundred μm. The tungsten's purity was higher than above 99.99% and contained impurities such as ~23 appm Mo, ~13 appm Fe, ~9 appm Cr and ~3 appm Ni. The samples were mechanically polished until the diamond paste is 0.25 μm before irradiation and then were irradiated with 200 keV He ions provided by the 320 kV Multi-Discipline Research Platform for Highly Charged Ions in Institute of Modern Physics, Chinese Academy of sciences (IMP, CAS), Lanzhou, China. The ion beam was swept in two perpendicular directions to a uniform distribution. The irradiation temperature is 800 °C and the irradiation fluences are the lowest 1.0×10^{16} ions/cm^2, the modest 5.0×10^{16} ions/cm^2 and the highest 1.0×10^{17} ions/cm^2.

When the incident He ions irradiate into the samples, the majority damages are the displacement damage and the He atom deposition. The theoretical results of displacement damage levels (i.e. dpa, displacement per atom) and He concentrations (C_{He}) for irradiated tungsten with three fluences are calculated with SRIM (The stopping and Range of Ions in Matter) and shown in Fig. 1(a) and (b). The displacement energy of tungsten was set to be 90 eV [10]. As recommended in Ref. [23], the surface and binding energies were set to be 0 eV, meanwhile we selected the "Ion Distribution and Quick Calculation of Damage" option to gain the data related to dpa and C_{He}. C_{He}/dpa ratios are equal to the C_{He} divided by dpa. It should be noted that the dpa values are possible maxima because the calculation does not consider the recombination of point defects and the diffusion of atoms. We can see that the dpa and C_{He} peak are the depths of 330 and 400 nm, respectively from Fig. 1(a) and (b). The integrated average dpa and C_{He} in two regions of first 160 nm and 160–300 nm from surface are given in Table. 1. In addition, C_{He}/dpa ratio is another important factor in the discussion of hardness and defect evolution. Its profile has been shown in Ref. [7] which shows its value increases with increasing depths and equals to about 2.2 at.%/dpa at 160 nm from surface.

After irradiation, NIT tests were carried out using an Agilent Nano Indenter G200 with a Berkovich tip in the continuous stiffness mode (CSM). The indenter was normal to the samples' surface. Six indentations were carried out on each sample and hence six hardness continuous distributions versus depths were given. Each

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