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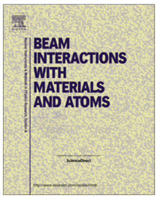

Influence of Au ions irradiation damage on helium implanted tungsten

Fanhang Kong [a], Miao Qu [a], Sha Yan [a,*], Xingzhong Cao [b,*], Shixiang Peng [a], Ailin Zhang [a], Jianming Xue [a], Yugang Wang [a], Peng Zhang [b], Baoyi Wang [b]

[a] State Key Laboratory of Nuclear Physics and Technology, Institute of Heavy Ion Physics, Peking University, Beijing 100871, People's Republic of China
[b] Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100049, People's Republic of China

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ABSTRACT

The damages of implanted helium ions together with energetic neutrons in tungsten is concerned under the background of nuclear fusion related materials research. Helium is lowly soluble in tungsten and has high binding energy with vacancy. In present work, noble metal Au ions were used to study the synergistic effect of radiation damage and helium implantation. Nano indenter and the Doppler broaden energy spectrum of positron annihilation analysis measurements were used to research the synergy of radiation damage and helium implantation in tungsten. In the helium fluence range of 4.8×10^{15} cm^{-2}–4.8×10^{16} cm^{-2}, vacancies played a role of trappers only at the very beginning of bubble nucleation. The size and density is not determined by vacancies, but the effective capture radius between helium bubbles and scattered helium atoms. Vacancies were occupied by helium bubbles even at the lowest helium fluence, leaving dislocations and helium bubbles co-exist in tungsten materials.

1. Introduction

Tungsten (W) is proposed as the leading candidate material for plasma-facing materials in fusion reactors because of its high melting point, low sputtering yield and high thermal conductivity [1]. It will be subjected to intense hydrogen isotope plasmas, helium particles, and 14 MeV neutrons produced by the fusion reactions [2,3]. Energetic neutrons will induce the production of athermal lattice defects (dislocations and vacancies) in tungsten [4]. In our previous research [5], when helium ions is implanted into tungsten materials, they will aggregate and nucleate to form helium bubbles. Both helium bubbles and lattice defects will play roles of destroyer in tungsten materials, their status will influence the mechanical behavior, hydrogen isotope retention [6], and further determine the service life of nuclear reactors. According to previous Refs. [7,8], vacancies in tungsten will play a role of trapper for helium ions because the high binding energy with helium ions. Nucleation will prefer to happen in vacancies then bubbles will be formed. However, the physical process of helium bubble formation in defect existing tungsten materials is still not clear, and neither is the effect of helium implantation on irradiation-induced defects.

In the present work, the synergistic effect of radiation damage and helium implantation was studied. Au ions irradiation was used to simulate the damage induced by neutrons, then helium atoms were implanted to research the formation of helium bubble. It should be noted that the maximum primary Knock-on atom (PKA) energies and the damage rates using heavy ions irradiation are generally higher than by neutrons, and the damage distribute in the sample surface layer, instead of the average distribution of neutron irradiation. The distributions were coupled by selecting the energy of Au and Helium ions. For a quantitative analysis of helium bubbles formation, a "cross-section" observation experiment using TEM is conducted. Nano-indenter tests and Doppler broaden energy spectrum of slow positron annihilation (DBS-SPA) were used to illustrate the influence of pre-irradiation damage on helium bubble formation in tungsten.

2. Experimental

The tungsten samples used in this work was foils with size of $10 \times 10 \times 1$ mm^3, which were cut off from a high-purity polycrystalline tungsten foil with thickness of 1 mm produced by Goodfellow Corporation. Samples were mechanically polished to mirror-like finish before any irradiation treatments. In order to release internal stresses and impurity introduced by polishing processes, they were annealed at 1200 K for one hour in high vacuum environment with a background pressure better than 10^{-5} Pa. Polycrystalline tungsten samples were pre-irradiated by Au ions to generate radiation-induced damages and then they were injected with helium ions to study the influence of radiation damage on helium.

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* Corresponding authors.
E-mail addresses: syan@pku.edu.cn (S. Yan), caoxzh@ihep.ac.cn (X. Cao).

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In general case, 14 MeV neutrons are deemed to be the culprit for radiation damage of structure materials in fusion devices. While considering the operability and economic reasons, 3 MeV Au ions produced by a 2 × 1.7 MV tandem accelerator were used to simulate the n-like radiation damages with the fluence up to $9.3 \times 10^{13}\,\mathrm{cm}^{-2}$. The background pressure was better than 10^{-5} Pa too in the irradiation process. Damage profile results was calculated using SRIM-2013 in the mode of 'full damage' with a threshold displacement energy of $E_{\mathrm{th}} = 90$ eV [9,10]. As shown in Fig. 1, the Au ions irradiation produced 1.0 displacement per atom (dpa) in tungsten materials at the damage peak located at the depth of 125 nm beneath the surface.

Helium ions were implanted into tungsten materials on an Electron Cyclotron Resonance (ECR) ion source device at Peking University [11]. Among the helium ions offered by this device, about 94.7% are He^{+} ions and 5.3% are He^{2+} ions. With an extraction voltage of 50 kV, these helium ions were accelerated to 50 keV and 100 keV respectively. The temperature was kept at 300 K using a water cooling system through the target holder attached to the back of samples. Three fluences (4.8×10^{15}, 1.9×10^{16}, $4.8 \times 10^{16}\,\mathrm{cm}^{-2}$) were used to generate helium rich layers near surface with peak concentration of 4.4×10^{3}, 1.8×10^{4}, 4.4×10^{4} -appm respectively, according to the full damage cascade simulations of SRIM-2013. The depth profile of helium concentration at the highest fluence is shown in Fig. 1, and the depth of concentration peak was about 125 nm too.

Cross-sectional TEM samples were prepared with a 'flash-polishing' method to avoid damage caused by Ar ions in the ion milling process. Samples were electrolytically polished for about 0.5–0.8 s in NaOH solution with mass fraction of 1% after ion milling. Nano-indentation measurements and Doppler broaden energy spectrum of slow positron annihilation (DBS-SPA) measurements were used to research the synergy of radiation damage and helium implantation in tungsten. Nano-indentation measurements were carried out in the Continuous Stiffness Measurement (CSM) mode which enables a continuous measure of hardness during loading, 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. The mean projective depth of energetic positrons in DBS-SPA tests can be estimated using the empirical equation $R = (40/\rho)E^{1.6}$ [12], where ρ $(\mathrm{g\,cm}^{-3})$ is the density of materials, E (keV) is the energy of positron and the unit of R is nm. In this work, positron energy of 0.03–20.03 keV corresponds to the mean projective rang of 0.01–250.07 nm.

In the DBS-SPA measurement, a high-purity Ge detector was used to record the gamma rays with energy 504.2–517.8 keV. The ratio of the central low-momentum area (510.2–511.8 keV) to the whole spectrum area defines the S parameter. Correspondingly, the ratio of the wing high momentum area (504.2–508.4 keV and 513.6–517.8 keV) to the whole one determines the W parameter. Therefore, the S parameter corresponds to the annihilation with valence electrons and is sensitive to open volume defects. While the W parameter corresponds to the annihilation with core electrons and is sensitive to chemical substances around the annihilation sites. An increase in S parameter indicates the presence of vacancy defects. More details of DBS-SPA test can be found elsewhere [6,12,13].

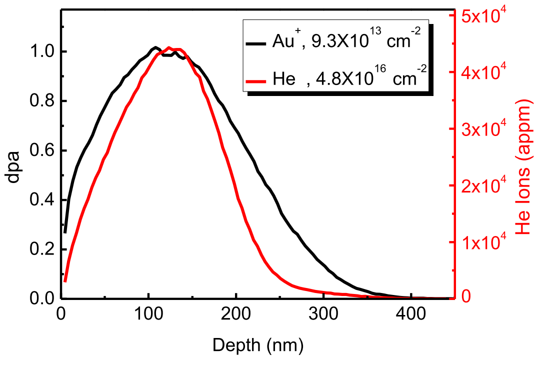

Fig. 1. Damage profile caused by 3 MeV Au ions irradiation and the distribution of helium atom concentration at the fluence of $4.8 \times 10^{16}\,\mathrm{cm}^{-2}$. Data was calculated by using SRIM-2013.

3. Results and discussion

3.1. Radiation caused by Au ions

Fig. 2 shows the cross-sectional TEM picture of tungsten samples irradiated by 3 MeV Au ions with a peak damage of 1.0 dpa. Clearly, a radiation damage layer near the surface was produced with a thickness of about 420 nm as shown in Fig. 2(a). Fig. 2(b) displays the enlarged view of the region near surface with depth range of 0–60 nm, low-density defect clusters and dislocation lines can be seen, and the half-moon-shaped pairs can be recognized as dislocation loops. When focus on the region near the damage peak depth (125 nm) shown in Fig. 2(c), dislocation lines and defect clusters both become denser obviously. And at the tail region of damage profile, only large amount of defect clusters can be observed without dislocation lines as shown in Fig. 2(d).

DBS-SPA test is known as an ideal method for vacancy defects because its sensitivity to the electrons' momentum in where positrons anneal [14]. The S parameter profiles of tungsten materials with and without Au ions irradiation is displayed in Fig. 3. For vir-

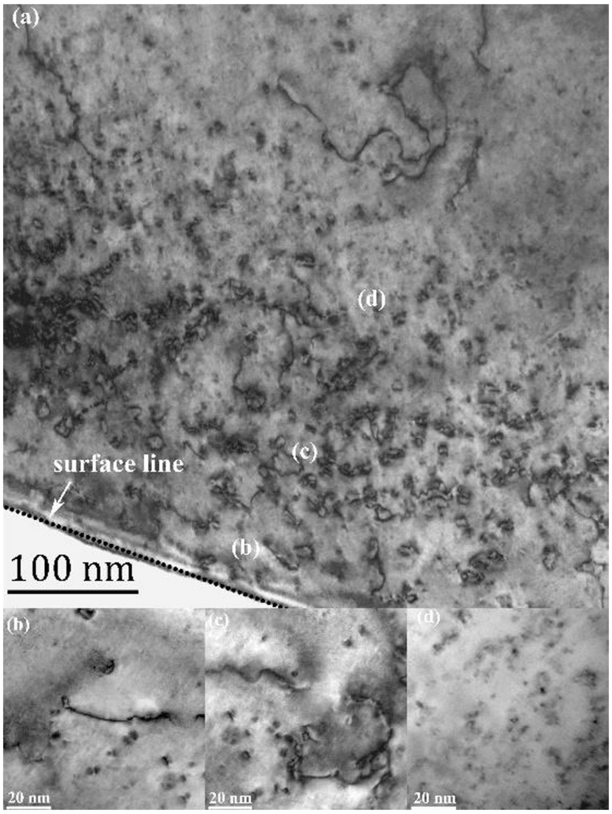

Fig. 2. Cross-section Bright-field TEM images of tungsten samples irradiated by Au ions. Subpictures in (b), (c), and (d) correspond to the corresponding regions shown in (a).

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