

Defect production and deuterium retention in quasi-homogeneously damaged tungsten



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ABSTRACT

To understand the deuterium (D) bulk retention behavior in plasma facing materials under reactor-relevant conditions, tungsten (W) foils were irradiated with 122 MeV ²⁰Ne ions and then exposed to D₂ gas at 773 K. A quasi-homogeneous distribution of atomic displacement damage to 0.3 dpa within a depth of 50 μm was produced in W using an energy degrader in the irradiation chamber. Results of positron annihilation lifetime spectroscopy showed a long positron lifetime component of ~400 ps appeared after irradiation, indicating the formation of vacancy clusters with up to 12 vacancies in W. Thermal desorption spectra showed a broad D desorption temperature range (730–1173 K) with a high release peak at ~1010 K for the irradiated W specimens. The amounts of D retained in the irradiated W were significantly larger than the annealed ones, which could be attributed to the trapping effects of the vacancy clusters formed by cascade collisions and the clustering of mono-vacancies.

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1. Introduction

Due to the advantageous thermal mechanical properties and relatively low solubility of tritium (T) under high heat and high particle fluxes loading, tungsten (W) is currently considered as the leading plasma facing material (PFM) for future fusion reactors [1,2]. However, T retention in W may greatly increase owing to the T trapping effects of accumulated defects created by the 14 MeV fusion neutrons. Besides, neutrons (n) have long mean free paths on the order of centimeters in solids, and W with a thickness of 1–2 cm could be volumetrically damaged [3]. Therefore, T bulk retention in n-irradiated W becomes a significant safety concern during the maintenance of plasma facing components and is critical to the global T fuel cycle of fusion reactors.

Heavy ion beams is widely used to investigate the influence of n-produced defects on T retention in W [4–14] because heavy ions can produce damage structures, i.e., dense cascades with large

vacancy clusters, which are similar to those produced by fast neutrons [15]. However, the damaged layer of heavy ions provided by ordinary ion accelerators is usually limited to a few micrometers beneath the specimen surface and the damage profile is strongly peaked the end of their range. Hence the effects of volumetric traps produced by irradiation on T retention in W have not been fully understood.

In this study, W was irradiated with extremely high energy heavy ions – 122 MeV ²⁰Ne ions. Using an energy degrader in the irradiation terminal, a quasi-homogeneous distribution of atomic displacement damage within ~50 μm was produced. After irradiation, specimens were characterized with positron annihilation spectroscopy, which is sensitive to characterize radiation defects on an atomic scale [16,17]. To investigate the effects of volumetric defects on T retention, the irradiated samples were exposed to D₂ gas. Herein D is used as a surrogate of T. Gas loading is performed instead of plasma exposure (i) to avoid further modification of near surface structure by plasma irradiation (ii) and to introduce an uniform distribution of D in the damaged W. The high temperature D bulk retention behavior then was studied with thermal desorption spectroscopy (TDS).

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2. Experimental details

2.1. High-energy heavy-ion irradiation

Tungsten foils (99.95% purity, rolled product) with dimensions of $\varnothing 20 \times 0.11$ mm and $\varnothing 20 \times 0.05$ mm were used in this study. The foils were stress relieved at 973 K for 1 h under H_2 atmosphere and then electro-polished with NaOH solution in the manufacturing process. To outgas the hydrogen and to reduce the densities of intrinsic defects, all samples were annealed at 1373 K for 2 h in vacuum before irradiation.

The high-energy heavy-ion irradiation was performed in a terminal chamber of the Sector-focused Cyclotron at HIRFL (Heavy Ion Research Facility in Lanzhou). The incident ion species used in the present study was $^{20}\text{Ne}^{7+}$, which was accelerated to a kinetic energy of 6.17 MeV/u (123.4 MeV in total). The ion beam then passed through an Al-foil assembly which was used for beam current monitoring (Before the irradiation of specimens, the assembly was calibrated with a Faraday cup located at the position of specimen stage). The energy of Ne ions after the Al-foil assembly was reduced to ~ 122 MeV. Before reaching the sample, the beam penetrated through an energy degrader, which was a wheel made of 30 pieces of Al-foils [18]. The thickness of each foil is shown in the right column of Fig. 1. By rotating the wheel, Al-foils with different thicknesses passed in front of the sample (i.e., the travel distance of Ne ions in the Al-foils kept varying). That is to say, instead of performing many separate irradiations at varying energies, the rotating wheel allowed a single and continuous irradiation to be performed that resulted in varying Ne ion energies to impact the sample. With this technique, a quasi-homogeneous distribution of defects was achieved in the sample. The irradiation area was 15 mm in diameter in the center of the sample limited by an aperture. After irradiation for 12 h (i.e., one side of the sample was irradiated to a fluence of 3×10^{20} ions/m²), the sample was turned around and the opposite side was irradiated to the same fluence. The background pressure in the chamber was about 2×10^{-5} Pa. The temperature of the specimens was kept around (713 ± 15) K.

For the ion induced damage calculations, SRIM-2008 [19] was used in the “full damage cascades” mode for 1000 projectile ions with a displacement threshold energy of 90 eV [20]. Fig. 1 shows the depth distribution of the displacement per atom (dpa) at one side of tungsten after irradiation for 12 h. Clearly, a quasi-homogeneous distribution of atomic displacement damage of 0.3 dpa within 22 μm is produced. The actual incident range of ions should

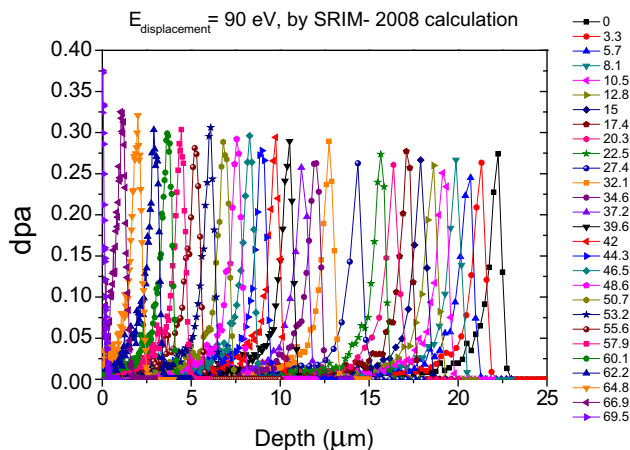


Fig. 1. Estimated depth profile of displacement damage at one side of W calculated using the SRIM code, corresponding to 122 MeV ^{20}Ne ions with a total fluence of 3×10^{20} ions/m² passed through the energy degrader after irradiation for 12 h. The right column shows the thickness (μm) of Al-foils used in the energy degrader.

be deeper than 22 μm for the SRIM calculation gives only the average incident range and the effects of target crystallinity are not taken into account [21]. Thus the tungsten specimen with a thickness of 50 μm can be considered to be quasi-homogeneously damaged after double-side irradiation.

2.2. Positron annihilation lifetime spectroscopy (PALS)

After irradiation, PALS measurements to characterize the irradiation defects in W were carried out at Wuhan University using a fast-fast coincidence positron lifetime spectrometer. PALS [16,17] measures the time between the positron implantation and its annihilation in the matter. The positron lifetime τ is a function of the electron density at the annihilation site. Due to the locally reduced electron density in the open volume regions, specimens with defects (such as dislocations, vacancies and their clusters) will exhibit longer positron lifetimes. The intensity of this component is directly related to the defect concentration.

Positrons were from a ^{22}Na radioactive source (2.6 years half-life, 0.545 MeV maximum energy). The incident range of positron R_+ in material can be calculated using equation [22,23]:

$$R_+[\text{cm}] = \frac{E^{1.19}[\text{MeV}]}{2.8\rho\left[\frac{\text{g}}{\text{cm}^3}\right] \cdot Z^{0.15}} \quad (1)$$

which takes into account material density ρ [g/cm³], atomic number Z and the positron energy E [MeV]. For tungsten ($\rho = 19.25$ g/cm³), the maximum incident range R_+ is ~ 47 μm . So W samples in this study were thick enough to ensure all positron annihilation processes took place in the material. The measured positron lifetime spectra were analyzed based on a two-state trapping model [17] by the programs Resolution and Positronfit [24].

2.3. D₂ gas loading and thermal desorption spectroscopy measurement

To check the hydrogen bulk retention behavior in irradiated tungsten, samples with and without Ne-ion irradiation were exposed to D₂ gas with a pressure of 1.2×10^5 Pa at 773 K for 2 h. The D₂ gas pressure was monitored with a Baratron capacitance manometer. At the end of the exposure, the device was quickly evacuated and the samples were immediately cooled by liquid nitrogen. Since all samples were processed together in one treatment, the difference in deuterium thermal releasing during the cooling process can be neglected. Samples after cooling were kept in a sample box for one or two days before TDS measurements were performed.

Thermal desorption experiments were carried out in a TDS facility at ASIPP [25]. Samples were heated in an infrared furnace up to 1273 K with a heating rate of 1 K/s. Desorption signals of mass 4 and mass 3 were both monitored with a quadrupole mass spectrometer, which was calibrated with a D₂ standard leak.

3. Results and discussion

3.1. Positron annihilation lifetimes in irradiated tungsten

The measured positron lifetime spectra are shown in Fig. 2 for the annealed and irradiated W. The curves for two irradiated W samples located significantly higher, indicating the occurrence of long-lived lifetime components. The extracted positron lifetime components and their relative intensities deduced by decomposition of the positron lifetime spectra are summarized in Table 1.

It has been reported that the lifetime of positrons in defect-free tungsten (τ_b) is ~ 105 ps and the lifetime of positrons trapped at mono-vacancies (τ_{1v}) is ~ 190 ps [26–28]. The lifetime of positrons trapped at dislocations is slightly shorter than that of positrons

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