



Deuterium trapping by irradiation damage in tungsten induced by different displacement processes



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HIGHLIGHTS

- By light ion (C^+ , He^+) irradiation, the dislocation loop and the vacancy were formed in tungsten.
- D retention by vacancies was almost saturated at the damage level of 0.2 dpa.
- D was trapped by voids resulted from cascade collision induced by the heavy ion irradiation.
- D could accumulate in the voids, resulting in the formation of blisters in tungsten.

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ABSTRACT

The deuterium trapping behaviors in tungsten damaged by light ions with lower energy (10 keV C^+ and 3 keV He^+) or a heavy ion with higher energy (2.8 MeV Fe^{2+}) were compared by means of TDS to understand the effects of cascade collisions on deuterium retention in tungsten. By light ion irradiation, most of deuterium was trapped by vacancies, whose retention was almost saturated at the damage level of 0.2 dpa. For the heavy ion irradiation, the deuterium trapping by voids was found, indicating that cascade collisions by the heavy ion irradiation would create the voids in tungsten. Most of deuterium trapped by the voids was desorbed in higher temperature region compared to that trapped by vacancies. It was also found that deuterium could accumulate in the voids, resulting in the formation of blisters in tungsten.

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1. Introduction

Tungsten (W) is a candidate for plasma facing material of fusion reactors. In the reactor operation, tungsten will be exposed to hydrogen isotope plasma including tritium. The solubility of hydrogen isotope in tungsten is relatively low, compared to that in graphite and so on [1]. However, it has been reported that irradiation damage in tungsten will be trapping sites for hydrogen isotope and therefore, enhance the hydrogen isotope retention [2–5].

The hydrogen trappings by irradiation damages have been simulated by higher energy ion irradiation technique [2–5] to introduce the defects, such as dislocation loops, vacancies and voids and so on. In TDS (thermal desorption spectroscopy) spectra, the

characteristic hydrogen desorption peak was observed in the temperature above 800 K for tungsten damaged by higher energy ion irradiation [3–5]. The influences of neutron irradiation on hydrogen retention behaviors were also investigated in the framework of US–Japan joint research project TITAN (Tritium, Irradiation, and Thermofluids for America and Nippon). The TDS experiments for tungsten damaged by neutron showed deuterium desorption peak in the temperature region around 800 K, similar to that observed for tungsten damaged with higher energy ion irradiation [6–8].

In TITAN project, Shimada et al., have examined deuterium retention in tungsten damaged up to about 0.3–3 dpa (displacement per atom) by different types of impact ions, such as 2.8 MeV Fe^{2+} , 20 MeV W^{2+} and 0.7 MeV H^- [6]. The deuterium TDS spectra for these specimens showed different deuterium desorption behaviors even in the comparable damage level. For example, the deuterium desorption in higher temperature region around 800 K was observed for tungsten damaged by 20 MeV W^{2+} irradiation but

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not appeared in the case of 2.8 MeV Fe²⁺. The difference was also found in deuterium retentions in each desorption peak. In tungsten damaged by 2.8 MeV Fe²⁺ or 0.7 MeV H⁻, deuterium TDS spectra were concentrated in lower temperature region below 700 K. The deuterium retentions in higher temperature region and lower temperature region were almost equivalent in the case of 20 MeV W²⁺ irradiation. These facts indicate that the concentration and type of trapping sites for deuterium are changed by the energy and/or species of impact ions, showing that the deuterium retention behaviors cannot be estimated only by the parameter of dpa level.

The irradiation damage would be introduced by a displacement of lattice atom of tungsten by energetic particles. As the energy transfer from incident particle to lattice atom increases, the displaced lattice atom (primary knock-on atom, PKA) can induce displacements of other lattice atoms (cascade collision), resulting in the larger size defects like voids and vacancy clusters. These differences in displacement processes would induce the different concentration of these defects, resulting in the differences of hydrogen retention behaviors in tungsten since the concentration of hydrogen trapped by each type of defects are different as reported by Roth and Schmid [9]. As the energy transfer rate is controlled by mass of impact ion as well as its energy, we have categorized the impact ions into two groups in the present study to clarify the effect of impact ions on formation of irradiation damage and consequent hydrogen retention behaviors in tungsten. The first is named as light ion with lower energy, namely 10 keV C⁺ and 3 keV He⁺ in this work. The second group is heavy ion with higher energy which is 2.8 MeV Fe²⁺. Deuterium was injected into tungsten after these ion irradiations mentioned above. The deuterium retention and desorption behaviors were evaluated by means of TDS measurement. The surface morphology was also observed by scanning electron microscope (SEM) for tungsten after TDS measurements.

2. Experimental

The tungsten samples (6 mm ϕ \times 0.2 mm^t) were prepared by cutting polycrystalline tungsten rod (99.99 at. % purity, from A.L.M.T. Corp.) annealed under stress relieved condition. The samples were mechanically polished with abrasive papers and diamond powders (9 and 3 μ m). Then, these samples were annealed at 1173 K for 0.5 h in ultra-high vacuum ($\sim 10^{-6}$ Pa) to remove the impurity and stress introduced by polishing processes.

The 10 keV C⁺ and 3 keV He⁺ were implanted into tungsten at room temperature by triple ion implantation system at Shizuoka University [10]. The flux of each ion was set to be $1.0 \times 10^{17} \text{ m}^{-2} \text{ s}^{-1}$. Several samples with different ion fluences were prepared to introduce various dpa levels in tungsten. The C⁺ fluence was ranged in 1.0×10^{19} – $1.0 \times 10^{21} \text{ C}^+ \text{ m}^{-2}$. The corresponding dpa levels are ranged in 0.78–78 dpa. The He⁺ fluence was changed in similar range, and dpa levels were around 0.03–3 dpa. The damage by 10 keV C⁺ and 3 keV He⁺ was distributed up to about 25 nm from surface. The dpa levels and distributions of damaged regions were estimated by SRIM (stopping range of ions in matter) code calculation assuming the displacement energy of tungsten atom as 90 eV [11].

The 2.8 MeV Fe²⁺ were irradiated into tungsten at room temperature by the Tandetron Accelerator, RAPID (Rutherford backscattering spectroscopic Analyzer with Particle induced X-ray Emission and Ion implantation Devices), at the University of Tokyo. The ion fluence was $3.3 \times 10^{19} \text{ Fe}^{2+} \text{ m}^{-2}$, corresponding to 3.0 dpa. The damage was distributed up to ~ 1000 nm beneath the surface.

Thereafter, the deuterium ion (D₃⁺) implantation and deuterium plasma exposure were carried out in TRIIX (Tritium Ion Implantation Experiment) and TPE (Tritium Plasma Experiment) systems

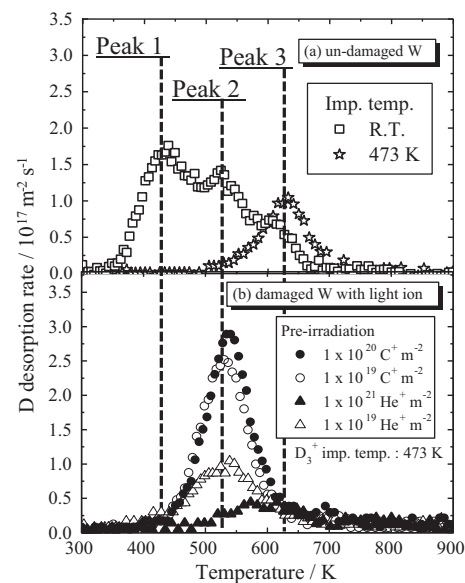


Fig. 1. Deuterium TDS spectra for (a) tungsten with different D₃⁺ implantation temperatures and (b) tungsten damaged with light ion irradiation prior to D₃⁺ implantation at 473 K.

at Idaho National Laboratory (INL), US. The energy of D₃⁺ was set to be 900 eV to prevent damage creation in tungsten during the deuterium ion implantation. The D⁺ flux and fluence were set to be $1.6 \times 10^{19} \text{ D}^+ \text{ m}^{-2} \text{ s}^{-1}$ and $3.2 \times 10^{23} \text{ D}^+ \text{ m}^{-2}$, respectively. The implantation temperature was controlled at 473 K. The D₃⁺ implantation for the non-damaged tungsten was also carried out at 473 K and room temperature. D⁺ flux and D⁺ fluence for deuterium plasma exposure were $6 \times 10^{21} \text{ D}^+ \text{ m}^{-2} \text{ s}^{-1}$ and $5.5 \times 10^{25} \text{ D}^+ \text{ m}^{-2}$, respectively. The energy of D⁺ and the sample temperature were set as 100 eV and 473 K in this plasma exposure experiment, respectively. Details of deuterium plasma exposure can be found in Ref. [8]. Thermal desorption experiments were carried out at INL after the D₃⁺ implantation. The samples were heated in ultra-high vacuum and by infrared furnace up to the temperature of 1173 K with the heating rate of 10 K min⁻¹. Heating at the maximum temperature of 1173 K was continued for 30 min and then sample temperature was decreased at the rate of 20 K min⁻¹. The mass species of 3 and 4, which corresponded to HD and D₂, respectively, were measured in TDS experiments for the estimation of deuterium retention. SEM observations were also conducted for these tungsten samples after TDS experiments.

3. Results and discussion

Fig. 1(a) shows deuterium TDS spectra for non-damaged tungsten samples implanted with D₃⁺ at room temperature and 473 K. The desorption temperature of deuterium for the sample implanted at room temperature was ranged in 300–700 K, which could be divided into three peaks located at 440 K, 530 K, 630 K, namely Peaks 1, 2 and 3, respectively. It was found that most of deuterium was desorbed as Peaks 1 and 2 under the D₃⁺ implantation at room temperature. The deuterium TDS spectrum for tungsten implanted with D₃⁺ at 473 K consists of almost single desorption stage as Peak 3. Because the desorption temperatures for Peaks 1 and 2 are located close to or lower than the implantation temperature of 473 K, almost all deuterium trapped as Peaks 1 and 2 would be desorbed during D₃⁺ implantation. The deuterium TDS spectra for tungsten damaged by lower energy light ion subsequently irradiated with D₃⁺ at 473 K showed clear differences compared to those for the non-damaged sample as found in Fig. 1(b). The

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