

## Elucidation of annealing effects on deuterium retention in damaged tungsten

欠陥導入タングステン中の重水素滞留挙動に及ぼす熱アニーリング影響

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Thermal annealing effects on deuterium (D) retention for Fe<sup>2+</sup> damaged tungsten were studied. It was found that total D retention was clearly reduced as the annealing temperature was increased. In particular, D retention trapped by voids was significantly decreased by annealing at 1173 K. Transmission electron microscope observation showed that the aggregation of dislocation loops was initiated at the temperature of 573 K and their recovery was completed at 1173 K. On the other hand, the aggregation of voids was not observed even if the annealing temperature was reached to be 1173 K.

### 1. Introduction

Tungsten (W) is a reference material of the divertor in ITER and a candidate for plasma facing materials for DEMO due to its good physical properties, such as lower sputtering rate and higher melting point. It is considered that W will be irradiated with 14 MeV neutron generated by the result of fusion reaction, which will introduce the radiation-induced defects [1]. It is well-known that the solubility of hydrogen isotope is quite low compared to that in graphite. However, the radiation-induced defects may contribute on the retention enhancement of hydrogen isotopes, which refrains the development of effective fuel cycle in fusion reactor. In addition, the defects are recovered and/or aggregated to form voids by thermal annealing [2]. Therefore, it is important to understand the thermal annealing effects on deuterium retention for damaged W to estimate the hydrogen isotopes retention behavior.

To demonstrate the actual fusion environment, the best way is the use of fusion neutrons to introduce the damage. However, the production of radioactive materials by neutron irradiation will regulate the various surface analyses, the heavy ion implantation method was used to introduce the damages in W [1 - 3]. The annealing effect on deuterium retention was studied at the

temperature between 573 K and 1173K.

### 2. Experimental

The polycrystalline W (A.L.M.T.Corp., Japan) with the size of 10 mm diameter and 0.5 mm thickness was used as a sample. These samples were heated up to 1173 K for 30 minutes under ultrahigh vacuum ( $< 10^{-6}$  Pa) as a pretreatment. The 6 MeV Fe<sup>2+</sup> were irradiated at 3 MV tandem accelerator, TIARA (Takasaki Ions Accelerators for Advanced Radiation Application), up to the damage concentration of 0.3 dpa (the damage level at depth of D implantation were calculated by SRIM 2008). Then, the samples were annealed at the temperature between 573 and 1173 K for 30 min and 1 keV D<sub>2</sub><sup>+</sup> irradiation was performed with the ion flux of  $1.0 \times 10^{18}$  D<sup>+</sup> m<sup>-2</sup> s<sup>-1</sup> up to the ion fluence of  $1.0 \times 10^{22}$  D<sup>+</sup> m<sup>-2</sup> at room temperature. The D desorption behavior was evaluated by TDS from room temperature to 1173 K.

Transmission electron microscope (TEM) observation for damaged W was performed to elucidate the recovery/aggregate of defects at room temperature to 1173 K at Kyushu Univ.

### 3. Result and discussion

#### 3.1 Annealing effect for D retention

The D<sub>2</sub> TDS spectra for damaged W with various

annealing temperature were shown in Fig. 1. These desorption spectra were divided into 3 desorption stages to be located at 400, 600 and 790 K. Peak 1 is known to be derived from the desorption of deuterium adsorbed on the surface and trapped by dislocation loops [4]. Peaks 2 and 3 were assigned to that trapped by vacancies and voids, respectively [4]. It is found that the D retention was clearly reduced as the annealing temperature increased. In particular, Peak 3 was significantly reduced when the annealing temperature was reached to be 1173 K. It can be said that the defects were recovered by annealing between 573 and 1173 K. In addition, the D trapping by voids was refrained by annealing at 1173 K.

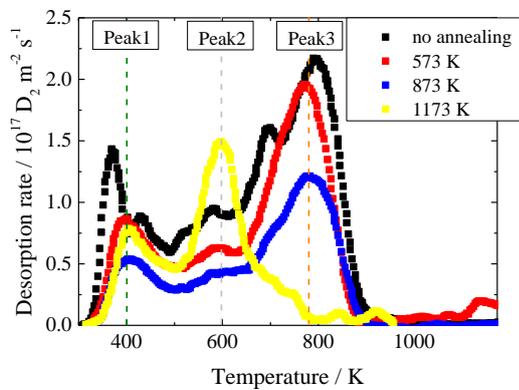


Fig. 1 D<sub>2</sub> TDS spectra for the damaged W with various annealing temperature.

### 3.2 TEM observation for damaged W

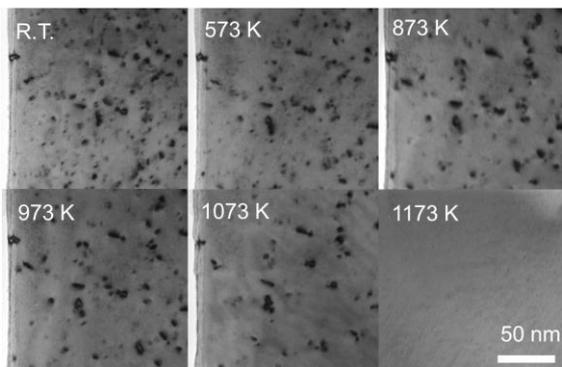


Fig. 2 Bright field TEM images of 6 MeV Fe<sup>2+</sup> irradiated W and their annealing behaviors.

Fig. 2 shows the typical TEM pictures for 0.3 dpa W focused on dislocation loops, It was found that the dislocation loops existed throughout the sample without un-annealed sample. The size of dislocation loops was clearly increased and its density was decreased after annealing above 573 K. After annealing at 1173 K, almost all dislocation loops were recovered. It can be said

that the aggregation and recovery of defects were initiated at the temperature of 573 K and most radiation defects were disappeared by 1173 K.

On the other hand, aggregation of voids was not observed in the present temperature region. This result suggests that it would be difficult to induce the aggregation of voids by annealing for the under 0.3 dpa sample because the distance of each void was longer and the density was lower. Therefore, further study is scheduled for 1.0 dpa sample to observe the aggregation behavior of voids and correlation between defect densities and annealing temperature will be revealed.

### 4. Reference

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