

## Impact of Surface Modification of Tungsten on Deuterium Retention

### タングステンの重水素吸蔵に対する表面改質効果

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Effects of surface modification by He bubble formation and by tungsten deposition on hydrogen isotope retention in tungsten have been studied in the compact PWI simulator APSEDAS. Low-energy deuterium plasmas ( $\sim 28$  eV,  $\sim 2.2 \times 10^{25}$  m<sup>-2</sup>) are exposed to a W sample with He bubbles with the size of  $\sim 10$  nm to  $\sim 200$  nm beneath the surface and a W sample with a tungsten deposited layer (thickness  $\sim 30$  nm) as well as a bulk W sample without surface pre-treatment at a surface temperature of  $\sim 500$  K, and then D retention is determined by a TDS method. The diffusion of D in W is a dominant process of D retention in W. The modification layer of the surface may play a role of the diffusion barrier.

### 1. Introduction

Tungsten (W) is an important candidate material in the divertor region in ITER, because it has good properties such as a high energetic threshold for physical sputtering, a high melting temperature and a low retention of hydrogen (H) isotopes. However, surface condition of a plasma facing material continues to change during plasma operation due to plasma-wall interaction (PWI) such as erosion, redeposition, radiation damage, bubble formation and so on. Such a surface modification could affect the physical properties of W as well as fuel retention. Codeposition is an important mechanism for the fuel retention of the metal wall as well as carbon. A He irradiation effect in W is another critical problem for H isotope retention, because radiation damage such as bubbles and dislocation loops, which are created by He irradiation, acts as both a strong trap site and a diffusion barrier of H isotope [1-5]. It is important to investigate the mechanism of surface modification and its effect on fuel retention.

In the compact PWI simulator APSEDAS, W samples with a deposited W layer and with

helium (He) bubbles by pre-implantation were exposed to low energy and high flux deuterium (D) plasma to investigate surface modification and its impact of H isotope retention.

### 2. Experimental Results

Three samples of polycrystalline tungsten with 0.1 mm thickness provided by Nilaco Co. were used in this work. The quoted purity was 99.95 %. The grain sizes were estimated to be in the range of  $\sim 1$  nm to 10 nm. A sample W-1 was pre-annealed at 1170 K in vacuum for 1 h. A sample W-2 was not pre-annealed but was exposed to low energy He plasma at the surface temperature of  $\sim 1770$  K for 50 min. Many fine irregularities were produced on the W-2 surface. Figure 1(a) shows a micrograph of a scanning electron microscope (SEM) of the W-2 surface. Many holes and fine irregularities were produced by the He plasma exposure. Figure 2(b) shows a cross-sectional micrograph of W-2 by using a focused ion beam (FIB) process and a transmission electron microscope (TEM). It is found that bubbles with the size of  $\sim 10$  nm to  $\sim 200$  nm were generated beneath the surface. A sample W-3 was mechanically polished and pre-annealed at

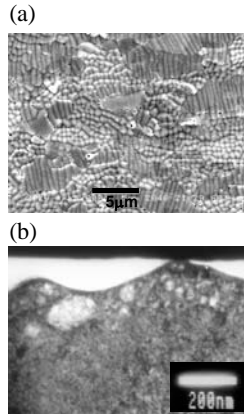


Fig.1. (a) SEM micrograph and (b) cross-sectional micrographs of the tungsten sample W-2.

1170 K in vacuum for 1 h and then a tungsten deposited layer was produced on the surface by a PVD method. The thickness of the deposited layer was  $\sim 30\text{nm}$ , which was estimated from a data of a quartz microbalance monitor (QMB), which was installed below the sample holder, and the geometric arrangement of the sample and the QMB.

Each sample was exposed to the low energy D plasma in APSEDAS. The exposed area was  $8\text{ mm}\phi$  and the surface temperature during the D plasma exposure was about 500 K. Typical parameters during the D plasma exposure in this work were  $n_e \sim 2.4 \times 10^{17}\text{ m}^{-3}$ ,  $T_e \sim 8\text{eV}$  and  $V_s \sim 28\text{ V}$  ( $\Gamma_i \sim 3 \times 10^{21}\text{ m}^{-2}\text{ s}^{-1}$ ). Although the plasma parameters of the He plasma exposed to the sample W-2 could not be measured directly, ion energy and fluence are considered to be in a range of 20 to 40 eV and  $\sim 4 \times 10^{25}\text{ m}^{-2}$  from the measurement of another similar plasma in APSEDAS, respectively. After D plasma exposure, D retention was measured using thermal desorption spectroscopy (TDS) with a quadruple mass spectrometer (QMS). During TDS, the sample was heated linearly at 1 K/s to 1127 K.

Figure 2 shows the fluence dependence of the total retention of W-1, W-2 and W-3 samples. In the case of the sample without the surface modification (i.e. W-1), the retention increased proportional to 0.52th power of the fluence. This means that diffusion is a dominant process in D retention in W. The retention of the W sample with the deposited layer significantly reduced to one-fifth of that of W-1. The deposited layer may play a role of diffusion barrier.

As for the W sample with He bubbles (i.e. W-3), the retention in the low fluence region was much higher than that of W-1, but it decreased slightly with the fluence and saturated. This decrease may

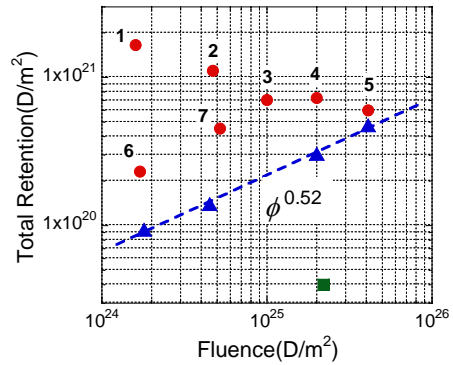


Fig.2. Fluence dependence of total retention of the bulk W without surface modification (triangle), W with helium bubbles (circle) and that with W deposited layer (square). The number next to the symbol indicates the order of the D plasma exposure.

be attributed to the characteristic change of the He bubble layer due to the repetition of the D plasma exposure. The decrease in the retention of 6th and 7th exposures is considered to be caused by the same reason. The high retention at the low fluence is attributed to a number of trap sites created by the He bubble. And the saturation of the retention is considered to be caused by suppression of the diffusion of D due to the He bubble layer.

### 3. Summary

Effects of surface modification of tungsten on hydrogen isotope retention have been studied in the compact PWI simulator APSEDAS. The D retention is in good agreement with a square-root dependence of the fluence, indicating that the D diffusion is a dominant process for the retention. The He bubble layer has a substantial potential of hydrogen isotope retention, and the retention decreases with fluence and saturate. The retention of W with a W deposited layer is significantly reduced to one-fifth of that of W without surface modification. Both surface-modified layers of the He bubbles and W deposition may play a role of a diffusion barrier.

### References

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