# 高密度直線型プラズマ発生装置PISCESにおいて D-He-Be混合プラズマ照射したWの微細組織と重水素リテンション特性 Microstructure and Deuterium Retention Property of W Exposed to D-He-Be Mixture Plasma in the linear divertor plasma simulators PISCES

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Microscopic damage and D retention in tungsten have been investigated for W exposed to ITER-relevant mixed species (D, He, Be) plasmas in the linear divertor plasma simulators PISCES-A and -B. It was observed that seeding of He into pure D plasma resulted in a significant reduction of D retention and suppression of surface blistering. TEM observations and ellipsometric measurements revealed that nano-sized high-density He bubbles were formed and percolated in the near surface region. A similar reduction of deuterium retention was also observed by ion irradiation experiments with an increasing fluence of He ions. Injected D atoms are thought to diffuse back to the surface through the percolating bubbles during exposure. The seeding of Be into D+He mixture plasma eliminates this He effect on the reduction in D retention.

#### 1. Introduction

In the ITER DT phase, the burning plasma will expose tungsten (W) simultaneously to helium (He) and other trace impurities such as C and Be besides hydrogen isotopes. Thus, the effect of these impurities on hydrogen isotope retention in W needs to be explored under mixed species plasma exposure for a more reliable prediction of T retention.

In this study, systematic mixed species (D, He, Be) plasma exposure experiments on W and complementary ion irradiation experiments have been conducted to investigate microscopic damage and hydrogen isotope retention for W in the burning plasma conditions.

## 2. Experimental

Mirror-polished W samples were exposed to pure D plasmas, He-mixed D (D+He) plasmas, Be-mixed D (D+Be) plasmas, or He and Be mixed D (D+He+Be) plasmas in PISCES-A and -B at the University of California, San Diego [1,2]. The He<sup>+</sup> ( $c_{\text{He+}}$ ) and Be<sup>+</sup> ( $c_{\text{Be+}}$ ) ion concentrations were spectroscopically determined [3], and were set to be ~1-20% and ~0.1%, respectively. The incident ion energy,  $E_i$ , was ~60 or ~120 eV, and the sample temperature,  $T_s$ , during the plasma exposures was kept fixed at ~573 or ~773 K by adjusting the rate of flow of forced-air cooling.

Subsequent to the plasma exposure, surface morphology and internal microstructures were examined by means of a scanning electron microscopy (SEM) and a transmission electron microscopy (TEM), respectively. To investigate D retention properties of the bulk samples, gas-desorption profiles were measured using a thermal desorption spectroscopy (TDS) system, capable of distinguishing between mass four He and  $D_2$  signals.

In addition to the plasma exposure experiments, sequential ion irradiation experiments were also performed with 3 keV-He<sup>+</sup> and then  $1.5 \text{ keV-D}^+$ .

## 3. Results and discussion

Figure 1 shows SEM images of the W samples exposed to pure D plasma and D+He mixture plasmas at  $E_i \sim 60 \text{ eV}$ ,  $\Phi_D \sim 5 \times 10^{25} \text{ m}^{-2}$  and  $T_s \sim 573 \text{ K}$ . On the surfaces of stress-relieved (SR-W) and re-crystallized (RC-W) pure W exposed to pure D plasma, high density micron-sized blisters are clearly seen, while the samples exposed to D+He mixture are as comparably smooth as the surfaces of the unexposed samples. In contrast, toughness enhanced, fine-grained W-1.1wt.%TiC (TFG W-TiC) [4] shows no significant changes in the surface morphology at all for exposure to both pure D and D+He mixture plasmas. D retention in TFG W-TiC was much smaller than that in



Fig.1. SEM images of several W samples exposed to pure D and D+He ( $c_{\text{He+}} \sim 20\%$ ) mixture plasmas at  $T_{\text{s}} \sim 573$  K,  $E_{\text{i}} \sim 60$  eV, and  $\Phi_{\text{D}} \sim 5 \times 10^{25} \text{ m}^{-2}$ .

SR-W and RC-W by around two orders of magnitude because of no blisters [5]. Since TFG W-TiC contains more grains than either SR-W or RC-W, it is speculated that a larger fraction of implanted D atoms can diffuse back to the surface along grain boundaries and leave from the surface during plasma exposure.



Fig.2. TDS of D<sub>2</sub> from SR-W samples exposed to pure-D and D+He ( $c_{\text{He+}} \sim 20\%$ ) mixture plasmas at  $T_{\text{s}} \sim 573$  K,  $E_{\text{i}} \sim 60$  eV, and  $\Phi_{\text{D}} \sim 5 \times 10^{25} \text{ m}^{-2}$ .



Fig.3. Cross-sectional microstructure of SR-W exposed to D+He mixture plasma.

In figure 2, thermal desorption spectra of  $D_2$  from the SR-W samples exposed to pure-D and D+He mixture plasmas are displayed. D retention in the sample exposed to D+He mixture plasma is significantly lower than that in the pure D exposed sample. To clarify the mechanisms of reducing the retained amount of D, near surface cross-sections of the plasma-exposed samples thinned by ion beam processing were examined with TEM. As can be seen in figure 3, the high density He bubbles (circular white contrasts) were created in the near surface region of the sample exposed to D+He mixture plasma. The bubble density and the mean diameter were estimated to be  $7.6 \times 10^{25}$  m<sup>-3</sup> and 1.8 nm, respectively, and then the volume fraction of bubbles in the damaged layer is ~23%. A comparable value was also with obtained from optical measurements spectroscopic ellipsometry [6]. When a filling fraction of percolating particles exceeds the percolation threshold of ~16%, the particles mutually percolate and form an infinitely large cluster regardless of their size and alignment [7]. Thus, the bubbles can interconnect and link between the surface and the deep region. In fact, the interconnected bubbles were observed with TEM [6]. These linked bubbles can act as a diffusion path to the surface for implanted D atoms during the plasma exposure, resulting in the observed significant reduction in D retention.



Fig.4. TDS of  $D_2$  from RC-W samples irradiated with  $3\text{keV-}D_2^+$  ions to fluence of  $1 \times 10^{21} \text{ m}^{-2}$  after the pre-irradiation of  $3\text{keV-He}^+$  ions.

A similar reduction of D retention was also observed by sequential ion irradiation experiments as shown in figure 4. Pre-irradiation of 3 keV-He<sup>+</sup> above the fluence of  $1.0 \times 10^{23}$  He<sup>+</sup>/m<sup>2</sup> caused a large D desorption peak to disappear, while the desorption peak increased with increasing the He fluence at a lower fluence up to  $1.0 \times 10^{22}$  He<sup>+</sup>/m<sup>2</sup>.



Fig.5. Total D retention in W samples exposed to various plasmas at  $T_{\rm s} \sim 573$  K,  $E_{\rm i} \sim 60$  eV, and  $\Phi_{\rm D} \sim 5 \times 10^{25}$  m<sup>-2</sup>.

On the other hand, a sample exposed to D+He+Be mixture plasma at  $E_i \sim 60$  eV and  $T_s \sim 573$  K showed no significant He effects. At this  $E_i$ , most of incoming Be onto the W surface is re-sputtered. Figure 5 shows the total D retention in samples exposed to the D+Be and D+He+Be mixture plasmas, compared with those exposed to the plasmas with no Be seeding. It is found that He seeding to D+Be plasma does not reduce D retention, as opposed to He mixture to pure D plasma. Thus, one can say that Be seeding has a more dominant influence on the D retention property in plasma-exposed W than He seeding.

The temperature dependence of microstructures and retention properties will be discussed in the presentation.

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