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Effect of temperature on microstructures and retention properties in tungsten exposed to D+He+Be mixture plasma in PISCES

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Abstract

The influence of temperature on microstructures and D retention properties in tungsten exposed to D+He+Be mixture plasmas is investigated. For the low temperature exposure case at 573 K, the Be seeding to D+He mixture plasmas results in the suppression of high density He nano-bubbles, which are distinctive internal defects observed in He irradiated/exposed metals. In contrast, fine and high-density He bubbles appear for W exposed to D+He+Be mixture plasmas at higher temperatures of > 773 K. TDS measurements also show that the influence of Be seeding to D+He mixture plasmas, which counteracts the significant reduction in D retention, becomes weaker with increasing temperature. From XPS analyses, it is

considered that Be_2W is formed by interdiffusion and solid-state-reaction at the high temperature exposure case, and the influence of the metallic Be on surface properties ultimately disappears with increasing temperature.

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1. Introduction

Tungsten (W) was chosen as a divertor material in the international thermonuclear experimental reactor (ITER) because of its high melting temperature, high thermal conductivity and low sputtering erosion yield [1]. In the ITER DT phase, the burning plasma will expose W simultaneously to helium (He) and other trace impurities such as Be besides hydrogen isotopes. To investigate the hydrogen isotope retention and surface damage for W in the burning plasma condition, multiple irradiation/exposure by D (or H) and He have been actively performed with ion beams [2-4] and linear plasma simulators [5,6]. Our previous work found that seeding of He and Be into D plasma causes significant microstructual changes and impacts on D retention properties [7-10]. However, available information about the effect of sample temperature during plasma exposures is limited, while W will be used at rather high temperature in ITER. In this study, therefore, the influence of temperture on microstructures and D retention in W exposed to D+He+Be mixture plasmas was examined.

2. Experimental

Samples used in this study are stress-relieved pure W (99.99 %) with diameter of 25.4 mm and thickness of 1.5 mm with a grain size of ~100 μm supplied from A.L.M.T. Corp. The samples were annealed at 1173 K for 0.5 h to relieve internal stresses. The rolling direction of the samples is perpendicular to the surface, similar to the proposed grade of W to be used in

ITER. Mirror polishing and ultrasonic cleaning were performed prior to exposure to plasma. In addition to the bulk samples, pre-thinned and vacuum annealed W discs of 3 mm diameter were also prepared by twinjet-electropolishing with sodium hydroxide electrolyte in order to carry out TEM observations after exposure to plasma. The pre-thinned samples were installed with a specially-fabricated Mo holder with the same size as the W bulk samples and were similarly treated as the bulk samples.

The samples were exposed to plasmas in the linear divertor plasma simulator PISCES-B [11] at the University of California, San Diego (UCSD). The Be^+ (c_{Be}) and He^+ (c_{He}) ion concentrations were spectroscopically determined [12,13], and were set to be ~0.1% and ~10%, respectively. The incident ion energy, E_i, was ~60 eV, achieved by negatively biasing the sample with respect to the plasma potential. At this E_i, most of incoming Be onto the W surface is re-sputtered [10]. The ion flux, Γ_i, was determined from single probe measurements, and was about ~$5x10^{22}$ ions $m^{-2}s^{-1}$ in all plasmas. The fluence of D to all samples, Φ_D, was derived from the measured ion flux by taking into account the contribution of ~10% He^+ ions, and was unified to be $5x10^{25}$ m^{-2}, assuming that D^+ ions are the dominant species in the plasma. The sample temperature, T_s, during the plasma exposures was measured by a thermocouple attached to the backside of the sample, and kept fixed at various temperatures (573-973K) by adjusting the flow rate of forced-air cooling.

Subsequent to the plasma exposures, internal microstructures were examined using a

transmission electron microscope (TEM) JEOL-JEM2010 for pre-thinned samples. To observe microstructures of the top surface region in the samples, thinning ion beam processing was also applied using the focused ion beam (FIB) milling with HITACHI-FB-2100. 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. During TDS, the samples were heated linearly with ramping rate of 1 K/s from room temperature to 1173 K. The D_2 signal was quantified with a D_2 calibrated leak bottle, and the total amount of D retained in a sample was obtained by integrating the TDS signal. X-ray photoelectron spectroscopy (XPS) was also used to evaluate the chemical state of the sample surface. While depth profiles were acquired by sputtering with a scanning 10 keV Ar^+ ion beam and its flux was ~$7x10^{15}$ ions $m^{-2}s^{-1}$, the sputter time cannot be converted to a depth scale because the sputter yield of the mixed and porous layers is presently unknown.

3. Result

3.1 TEM observations

Pre-thinned W samples were exposed to the Be-mixed plasmas at T_s ~573, 773, and 973 K for internal microstructure examinations with TEM. A sample exposed to the plasma at T_s ~973 K was unfortunately stuck on the Mo sample holder probably due to a temperature

rising. Fig. 1 shows microstructures of the samples exposed to the various plasmas. Cavities are seen as white contrasts at this image condition. There is no significant difference in the microstructure for the samples exposed to D plasma with and without Be seeding regardless of the sample temperature. On the contrary, while high density He bubbles are observed in the sample exposed to D+He mixture plasma, cavities are hardly seen in the sample exposed to D+Be+He mixture plasma at T_s ~573 K. Fig. 2 shows the cross-sectional TEM images obtained from the W bulk samples thinned with FIB milling after the exposure to (a) D+He mixture plasma and (b) D+He+Be mixture plasma at T_s ~573 K. In these images He bubbles are observed as bright and dark contrasts in under and over focused images, respectively, if they are present. Cavities are hardly seen for the sample exposed to D+He+Be plasma, while high density He bubbles with a depth distribution far beyond the He ion range of a few nm are clearly observed in the sample exposed to D+He plasma. On the contrary, He bubbles reappear in the sample exposed to D+Be+He mixture plasma at T_s ~773 K as shown in Fig. 1, although the size is much smaller than those in the sample exposed to D+He mixture plasma at T_s ~573 K. These results indicate that Be seeding suppresses the formation of He bubbles at T_s ~ 573 K, and such a effect disappears at elevated temperature.

3.2 TDS measurements

In Fig. 3, thermal desorption spectra of D_2 from the W samples exposed to various

plasmas at Φ_D ~5x10^{25} ions m^{-2}, E_i ~60 eV and T_s ~ (a) 573, (b) 773, and (c) 973K are displayed. For the samples exposed to Be seeding mixture plasmas at T_s ~573K, a desorption peak appears at the temperature of around 800 K, and is an order of magnitude smaller than that for the sample exposed to the pure D plasma. However, the desorption rate of these Be-containing samples is higher at higher temperature > 900 K. With increasing T_s, the desorption at >1000 K disappears, and then the peak at ~800-900 K becomes smaller and falls below the detection limit for both samples exposed to D+Be and D+He+Be mixture plasmas at T_s ~973 K.

From TDS spectra, the total D retention of each sample was derived as shown in Fig. 4. Compared with the sample exposed to pure D plasma at T_s ~573 K, the samples exposed to D+Be and D+He+Be at T_s ~573 K retain an order of magnitude smaller D. In addition, it should be noted that the influence of He seeding on the retention, which is significant without Be seeding, is almost eliminated for the samples exposed to D+He+Be plasma at T_s ~573 K, and D retention in D+Be and D+He+Be samples is similar for all T_s.

3.3 XPS measurements

Be depth profiles on the surface region of the samples exposed to D+Be mixture plasma at T_s ~773 and 973 K are obtained by the integration of the peak area over the energy range from 106 to 120 eV with a Shirley background subtracted, and are displayed in Fig. 5.

The both samples show a similar tendency, while the intensity for the sample exposed at T_s ~973K is slightly smaller than that at T_s ~773K. This reduction can be explained by the enhancement of Be erosion due to the surface adatom formation at elevated temperatures [14]. On the contrary, the chemical states of Be for both samples were not necessarily the same, as displayed in Fig. 6. The chemical states were examined after the surface sputtering with Ar ion beam for 0.5 and 1.6 minutes. In this figure, the binding energies of Be1s, which originate from metallic Be (111.8 eV), Be_2W (111.1 eV), and BeO (114.0 eV) [15,16], are also displayed with the broken lines. The chemical state near the top surface is dominated by beryllium oxide for both samples, which seems to be caused by air exposure. However, in the middle of the Be layers, Be_2W alloy becomes dominant for the sample exposed at T_s ~973 K, while the metallic Be is dominant at T_s ~773 K. It is considered that Be_2W is formed by interdiffusion and solid-state-reaction during the plasma exposure at T_s ~973 K.

4. Discussion and Conclusion

For the low temperature exposure case of T_s ~573 K, the Be seeding to D+He mixture plasma suppressed the formation of He bubbles, and accordingly counteracted the remarkable He effects on the significant reduction in D retention. As shown in TDS spectra in Fig. 3 (a), the Be seeding also caused additional desorption at higher temperature of ~1000 K with and without He seeding, indicating that Be creates rather strong trapping sites for D at T_s ~573 K.

However, the influence of Be seeding becomes smaller with increasing T_s: fine and high-density He bubbles appear, and the deuterium desorption at higher temperature of ~1000 K disappears. From these results, one can say that the influence of Be seeding on microstructures and D retention properties of tungsten is more remarkable at lower T_s.

The XPS analyses reveal the small reduction of Be and the formation of Be_2W alloy on the sample exposed to D+Be mixture plasma at T_s ~973 K. These results seem to correspond with the reappearance of He bubbles with increasing T_s, and with the reduction of desorption at ~1000K, as shown in the TEM observations and the TDS experiments, respectively. It is reasonable to suppose that the influence of metallic Be on surface properties ultimately disappears due to the quantitative reduction and to the change in the chemical state at elevated temperature.

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Figure captions

Fig. 1. TEM images of pre-thinned W samples exposed to various plasmas.

Fig. 2. Cross-sectional TEM images of W samples exposed to (a) D+He and (b) D+He+Be mixture plasmas at T_s ~573K.

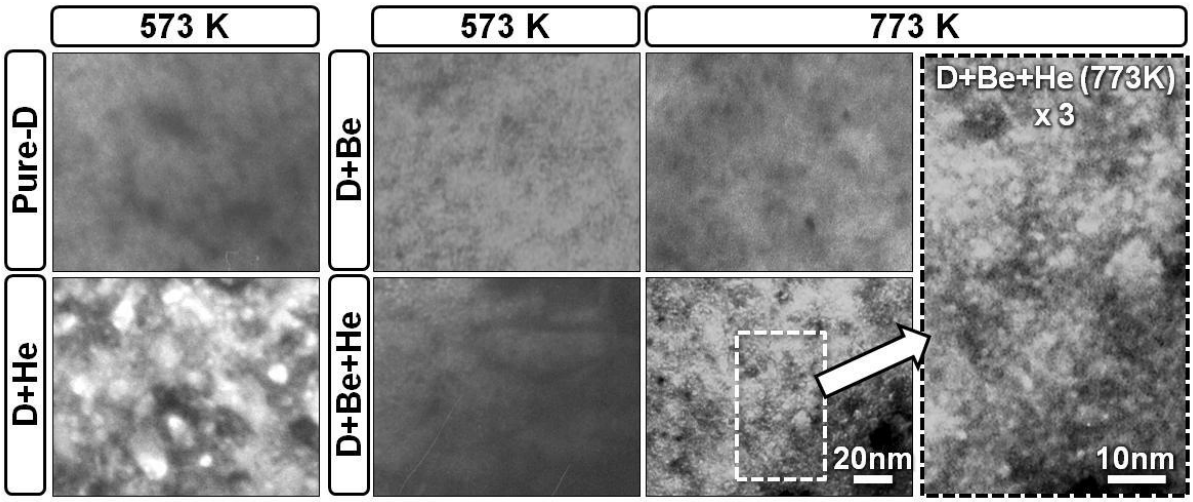
Fig. 3. Thermal desorption spectra of D_2 for W samples exposed to various plasmas at T_s ~ (a) 573, (b) 773, and (c) 773 K

Fig. 4. Total retentions of D for W samples exposed to various plasmas. The fluence of D is unified to be $5x10^{25}$ m^{-2} for all the samples.

Fig. 5. Depth profile of Be for the samples exposed to D+Be mixture plasmas at T_s ~773 and 973 K.

Fig. 6. XPS spectra of the Be 1s energy range for the samples exposed to D+Be mixture plasmas at T_s ~773 and 973 K in the surface region.

Figure 1 – N. Iijima, two columns



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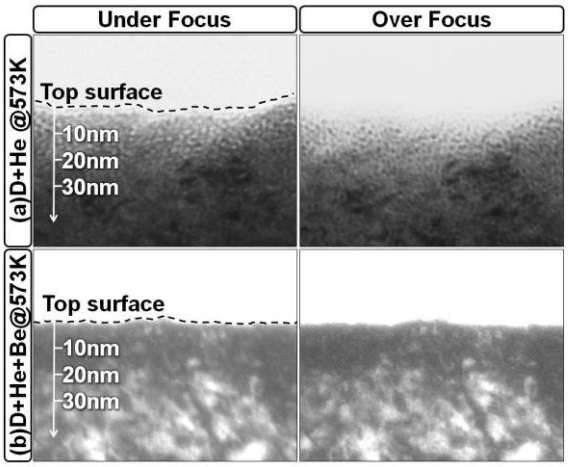

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Figure 3 – N. Iijima, two columns

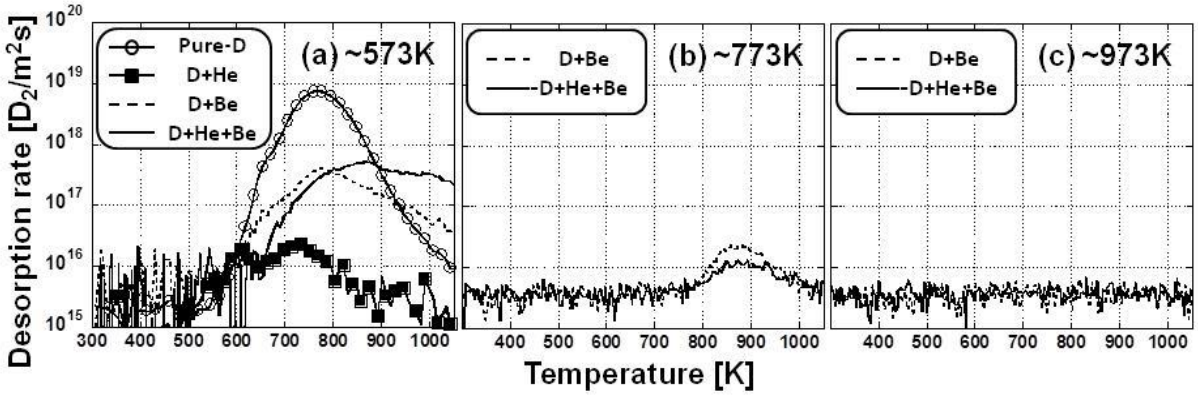

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Figure 4 – N. Iijima, one column

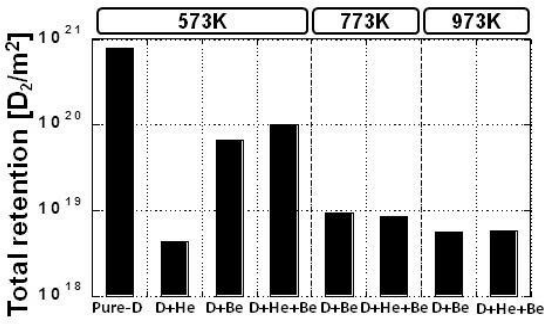

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Figure 5 – N. Iijima, one column

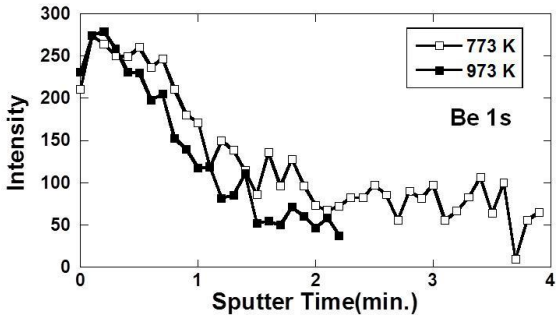
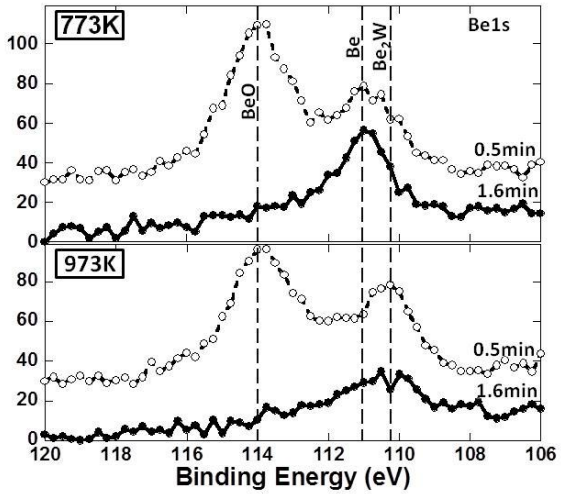


Fig. 5. Depth profile of Be for the samples exposed to D+Be mixture plasmas at T_s ~773 and 973 K.

Figure 6 – N. Iijima, one column



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