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Blister formation and deuterium retention on tungsten exposed to low energy and high flux deuterium plasma

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Abstract

Deuterium ion irradiation on tungsten has been carried out with incident energies of 100 eV and flux of 1×10^{22} D^+ m^{-2} s^{-1} at a temperature in range between 333 K and 1130 K up to a dose of 1×10^{26} D^+ m^{-2}. Three kinds of tungsten used are pure tungsten made by powder metallurgy tungsten (PM-W), vacuum plasma spray tungsten (VPS-W) and single crystal tungsten (SC-W). Surface morphology before and after the irradiation is observed with an SEM. In addition, retention property of deuterium after the irradiation is also examined with a TDS. Behavior of blister formation depends on the kind of the samples and the irradiation temperatures. TDS measurement also shows that deuterium is not retained in sample, which the blisters are not formed. The behavior of the blister formation and deuterium retention is influenced by the manufacturing process and the sample history of tungsten.

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Keywords: Bubbles & blister; Deuterium inventory; High-*Z* material; Ion-surface interactions; Tungsten coated carbon

1. Introduction

Recently, tungsten has became one of the candidate plasma facing materials for fusion devices due to its low sputtering yield and good thermal properties. The plasma facing materials will be subjected to high-flux low energy particle bombardment including fuel hydrogen isotope. In recent studies, surface modification and hydrogen retention on tungsten exposed to hydrogen isotope ion with energies (a few hundred eV) below that required to generate elastic displacement have been investigated [1–6]. The studies have demonstrated that hydrogen isotope implantation into tungsten can produce blustering. The blister formation affects the trapping behavior of hydrogen isotopes and erosion resulting from surface flaking. Tungsten is produced by different methods which introduce impurities and

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different lattice imperfections. These defects act as trapping sites of hydrogen isotope. Therefore, the behavior of the blister formation and hydrogen isotope retention may be influenced the manufacturing process and the sample history. In the present study, three kinds of tungsten have been exposed to a high flux deuterium plasma where the ion energy was kept low in order to acquire to a good understanding of the blister formation and deuterium trapping behavior.

2. Experimental

Three kinds of tungsten were used for the present experiment. They were powder metallurgy tungsten (PM-W), vacuum plasma spray tungsten (VPS-W) and single crystal tungsten (SC-W). PM-W was stress relieved pure tungsten fabricated by powder metallurgy supplied by Allied Material Corp. After rolling, heat treatment was performed at 1173 K for 15 min in hydrogen gas. Rolling ratio and purity of PM-W were 87.4% and 99.99 wt%, respectively. A disk of PM-W 25 mm in diameter and 2 mm in thickness was used. The surface of the PM-W was electrically polished. VPS-W with a thickness of 0.5 mm was coated by vacuum plasma spraying on isotropic fine graphite (VPS-W/IG-430U) (25.4 mmϕ1 $\times$ 3 mm) made by Toyo Tanso [5]. After coating, heat treatment was performed at 1173 K for 5 h to stabilize the microstructure of the sample. The density of VPS tungsten (VPS-W) was 98.0% of that of theoretical density. PM-W and VPS-W/IG-430U were degassed by heating up to 1273 K in high vacuum before installation into the plasma facility. A disk of SC-W 8 mm diameter and 1 mm in thickness, fabricated by the floating zone melting method, was also used. The surface of (1 0 0) plane of SC-W was electrically polished and was irradiated by deuterium ion.

The facility used in the present experiment was the Pisces-B, which is the liner plasma simulator device at the University of California, San Diego. In the present experiments, deuterium was used as working gas. The ion flux (D^+) to the sample was about $1.0 \times 10^{22}\ m^{-2}\ s^{-1}$. The energy of the impinging ions was 100 eV. The previous experiment have showed that blisters is formed by the irradiation of about $5 \times 10^{25}\ D\ m^{-2}$ on PM-W at relatively low temperature [5]. Therefore, irradiation was carried out up to a dose of $1.0 \times 10^{26}\ m^{-2}$.

Plasma parameters remained constant throughout the plasma exposure. The sample was clamped to a water or air cooled sample holder with a Tantalum cap. The temperature was measured at the center of the sample on the side opposite to the plasma with thermocouples. The thermocouples were mechanically attached to the backside of the sample. The samples were heated by the plasma and reached their final steady state temperatures within about 1 min. The sample temperatures were in a range from 333 K to 1130 K.

After the plasma exposures, the sample was removed from the Pisces-B facility and microstructure was examined by scanning electron microscopy (SEM). Weight loss before and after the exposure was also measured. In addition, retention of deuterium after the exposure was examined by thermal desorption spectroscopy (TDS). The sample was linearly ramped up to 1273 K for 65 min (0.25 K/s) and remained at 1273 K for 20 min, and was cooled down linearly to 298 K for 90 min (0.18 K/s).

3. Results

Shown in Fig. 1(a) and (b) are the SEM images taken from PM-W samples after the irradiation at 333 K and 673 K, respectively. Blisters with a diameter of $0.2 \sim 2\ \mu m$ was formed on surface of PM-W irradiated at 333 K. In addition, blisters with a diameter of $0.5 \sim 7$ m are formed on the surface of PM-W irradiated at 673 K. Shape of these blisters was not circular. The blisters with ellipse shape and holes were observed on the surface as shown in Fig. 1(b). However, blisters were not found on the surface of PM-W irradiated at 732 K and 1130 K.

Fig. 2(a) shows SEM image of the surface on VPS-W irradiated at 333 K. Modification occurs on the surface of VPS-W irradiated at 333 K and 723 K as shown in Fig. 2. They seem to be small blisters with a diameter of about 0.2 μm. In addition to this, fine surface modification was observed on VPS-W irradiated at 1123 K as shown in Fig. 2(b). This may be formed by chemical reaction of deuterium and VPS-W surface.

Shown in Fig. 3 are the surface of SC-W after the irradiation at 343 K. Blisters with a diameter of $1 \sim 2\ \mu m$ were formed. Directional cracks, which are seen to be black line as shown in Fig. 3, were also formed. However, no modification occurred on the surface of SC-W after the irradiation at 383 K, 623 K and 1123 K.

Shown in Fig. 4(a) and (b) are the thermal desorption spectrum of HD and D_2 from PM-W and VPS-W irradiated at 333 K. The major peaks appear at around at 750 K and 680 K, respectively. On the other hand, a peak appeared at 620 K from SC-W irradiated at 343 K. In the case of PM-W irradiated at 673 K, HD and D_2 desorption started at temperature below the exposure temperature. This is expected that as the sample cooled after the plasma exposure the diffusing D atoms already in the sample may become trapped in sites that remain unoccupied during the high temperature during plasma exposure. On the other hands, in the thermal desorption from VPS-W irradiated at 723 K and 1123 K, HD was only released but not D_2. However,

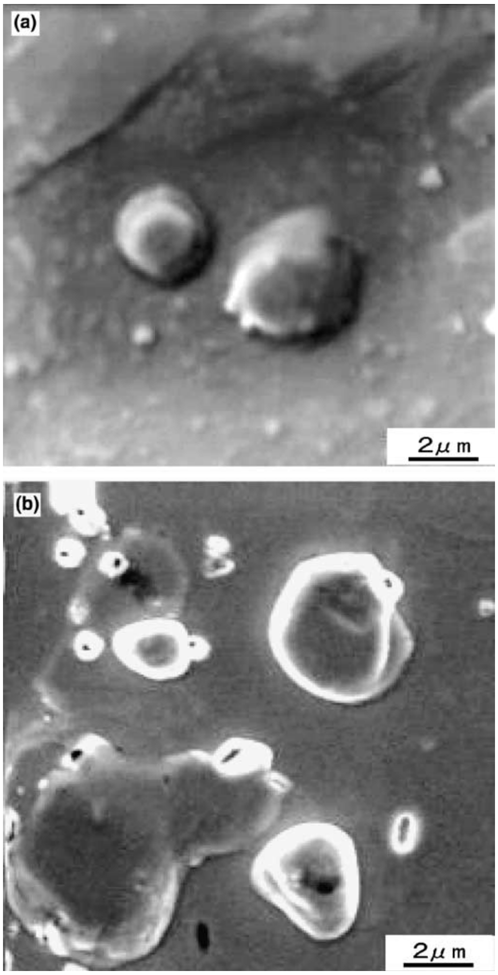

Fig. 1. SEM images of PM-W irradiated by 100 eV D^+ to a fluence of 1.0×10^{26} m^{-2} at 333 K (a) and at 673 K (b).

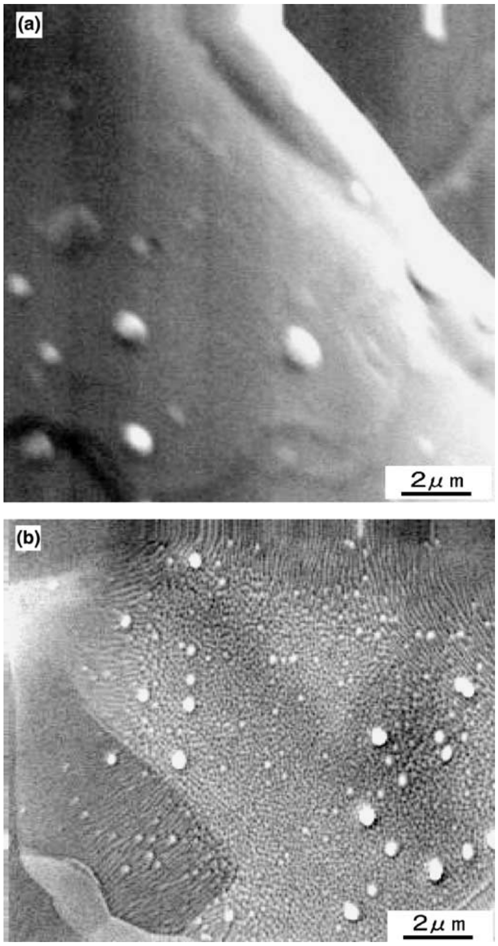

Fig. 2. SEM image of VPS-W irradiated by 100 eV D^+ to a fluence of 1.0×10^{26} m^{-2} at 333 K (a) and 1123 K (b).

in the thermal desorption from PM-W which was irradiated at 723 K, 1130 K and SC-W which was irradiated at 383 K and 623 K, release of HD and D_2 did not appeared. In these samples, the blisters were not formed on the surfaces.

Fig. 5 shows the total amount of desorption of D as a function of irradiation temperature. In the case of PM-W, the total amount of desorption of D at 673 K was larger than that of at 333 K and desorption was not detected at elevated temperatures between 723 K and 1133 K, which blisters were not formed. However, in the case of VPS-W, the total amount of desorption decreases with increasing irradiation temperature and desorption was seen from VPS-W irradiated at 1123 K. On the other hand, in the case of SC-W, the total amount of desorption of D was almost same as much as that of PM-W at 333 K and desorption was not seen above 383 K, which blister was not formed.

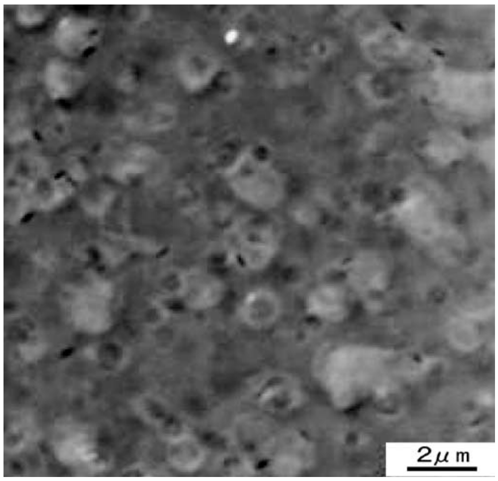

Fig. 3. SEM image of SC-W irradiated by 100 eV D^+ to a fluence of 1.0×10^{26} m^{-2} at 343 K.

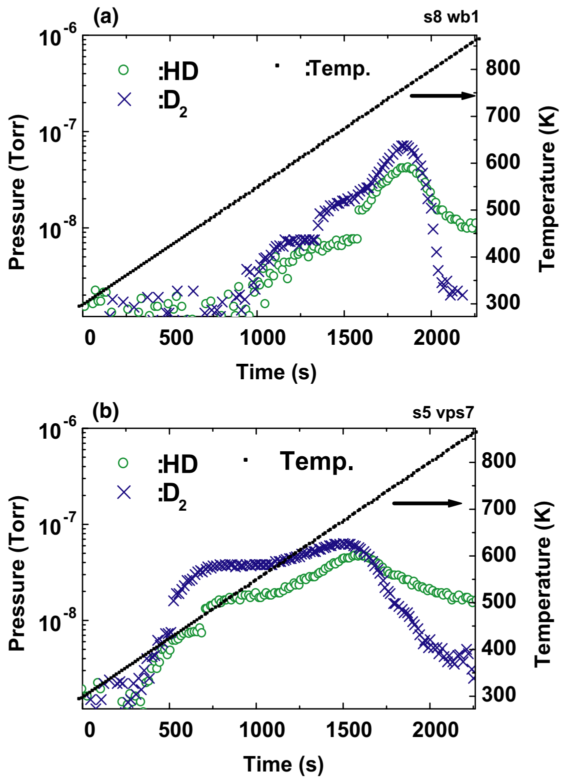

Fig. 4. Thermal desorption spectrum of HD and D_2 from PM-W (a) and VPS-W (b) irradiated at 333 K.

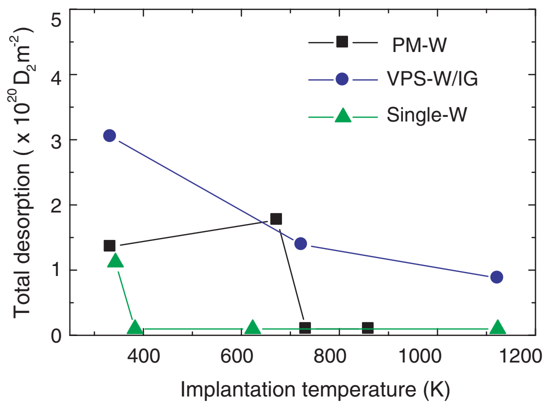

Fig. 5. Total amount of desorption of D as a function of irradiation temperature for PM-W, VPS-W and SC-W.

4. Discussion

A lot of research about blister formation due to gas ion irradiation was carried out, but only for energies high enough to cause elastic damage [7]. These studies have shown that one of the necessary conditions for blister formation in metals is agglomeration of implanted gas atoms and vacancies which are formed by displacement damage to form to gas bubbles in near-surface region. In the present experiment condition under parameters more relevant for the fusion devices, displacement damage is not formed. Therefore, the blister formation in the present experiment is different from that of the previous studies. Recent observation of modification of tungsten surfaces exposed to low energy, high flux plasmas and ion beam bombardment have shown the formation of blisters [1–6]. Experiments are underway to try to systematically investigate and identify the mechanisms responsible for this behavior.

In the case of PM-W used in the present experiment, structure such as grain boundary and dislocation distributes in parallel to rolling direction and surface of PM-W was parallel to the structure. Deuterium ions were injected to the surface normal. Therefore, one of the possible mechanisms is that deuterium implanted diffuses to deeper into PM-W, and agglomerates at grain boundaries with parallel direction for the surface, which were produced by rolling process during the fabrication of PM-W. This is agree with that blister was not formed on surface of SC-W, which grain boundaries were not exist, in the temperature range above 383 K. However, blister formation on surface of SC-W irradiated at 343 K indicates that other possible new kind of trap site exists except for gain boundary. One of the possible mechanism may be due to deuterium bubble formation by high density deuterium in tungsten surface layer, which is caused by low energy high flux deuterium implantation at relatively low temperature.

In the case of VPS-W, amount of impurity is expected to be larger than that of PM-W and SC-W because of the fabrication process. In addition, structure is different from that of PM-W because tungsten layer was formed by deposition and resolidfication of molten droplets which were produced by plasma spray. It is expected that the structure different between VPS-W and PM-W attribute to surface modification by the irradiation. Formation of blister may influence erosion because blistering may result in flaking by higher irradiation. In the present experiments, the manufacturing process and the sample history of tungsten is one of the key factors for the blister formation on the tungsten surface.

5. Conclusion

Three kinds of tungsten have been irradiated by low energy and high flux deuterium (100 eV, 1×10^{22} D^+ m^{-2} s^{-1}, 1×10^{26} D^+ m^{-2}) at a temperature in the range between 333 K and 1130 K using a plasma simulator. Surface modification and deuterium retention have been examined. Formation of blister depends on kind of tungsten, structure and irradiation temperature.

Thermal desorption spectroscopy shows that desorption of deuterium is seen from the samples, which blisters are formed on the surface. This means that deuterium retention is corresponding to the blister formation. These experimental results indicate that the manufacturing process of tungsten is one of the key factors for the blister formation and the retention of hydrogen isotope on tungsten.

Acknowledgment

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