Surface morphology and deuterium retention in tungsten exposed

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Surface morphology and deuterium retention in tungsten exposed to high flux D plasma at high temperatures

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ABSTRACT

Surface modifications and deuterium retention induced in tungsten by high fluxes ($10^{24} \text{ m}^{-2} \text{ s}^{-1}$) low energy (38 eV) deuterium ions were studied as a function of surface temperature. Blister formation was studied by scanning electron microscopy and electron backscatter diffraction, while deuterium retention was measured by thermal desorption spectroscopy. Blisters are observed on the surface exposed at different temperatures, ranging from 493 K to 1273 K. The blister density and D retention decrease with the increasing exposure temperature. The formation of blisters at high temperatures is attributed to the high flux of D plasma. At 943 K, with the increasing fluence, there is trend to the saturation of D retention and blister density. The defects caused by plasma exposure have an important effect on the D trapping and blistering behavior. The formation of blisters has a strong relationship with slipping system of tungsten.

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1. Introduction

Due to its favorable physical properties, such as low sputtering yield, high melting temperature and high thermal conductivity, tungsten (W) will be used as a plasma facing material in the ITER divertor [1]. As a plasma facing material, tungsten will be subjected to high fluxes and fluences of hydrogen isotope ions, helium ions and energetic neutrals. Strong surface modifications such as blistering can occur on tungsten surfaces exposed to hydrogen isotope particles [2–6], even when the incident energy is below the threshold energy for displacement damage. Blistering could be an important issue for the application of tungsten materials as plasma facing material in ITER, as it may result in an increased tritium inventory and decreased thermal conductivity [7]. The exposure temperature plays an important role in the blistering behavior, because it affects the diffusion process of hydrogen atoms and the physical properties of W materials [4–6]. According to simulations [8], the temperature in the ITER divertor region can range from 400 K to 1400 K. In previous studies [5,6], blister formation was never observed after plasma exposure for surface temperatures higher than 750 K. However, most of these studies were carried out with D plasma fluxes about $10^{22} \text{ m}^{-2} \text{ s}^{-1}$, orders

of magnitude lower than expected in the divertor region of ITER, where the particle flux can reach up to $10^{24} \text{ m}^{-2} \text{ s}^{-1}$ during operations [9]. The ion flux can affect the D transport and retention in W materials [10,11], which can affect the blistering behavior on the surface. In order to understand the significance of blisters induced by hydrogen isotopes for ITER, it is necessary to investigate the occurrence of blisters at ITER relevant temperatures and fluxes.

In this study, experiments were carried out using low energy (38 eV) D plasma with a flux of $10^{24} \text{ m}^{-2} \text{ s}^{-1}$ at high temperatures (up to 1273 K). The evolution of the surface morphology and D retention in the material have been studied to get a better understanding of blistering behavior under divertor relevant plasma condition.

2. Experimental

Polycrystalline tungsten samples with purity of 99.95 wt% were cut from a rolled sheet, in the form of discs with diameter 30 mm and thickness 3 mm, which was supplied by Advanced Technology & Materials Co., Ltd. (China). The main impurities are Mo, Fe, O with concentrations below 15 wppm, and C with a concentration around 50 wppm. All the samples were stress relieved at 1273 K at a background pressure of $5 \times 10^{-4} \text{ Pa}$ for 1 h after polishing and before implantation.

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The samples were exposed to a high flux D plasma beam in the Pilot-PSI linear plasma generator [12], which is uniquely capable of producing low temperature plasmas with a high flux ranging from 10^{23} to $10^{25} \text{ m}^{-2} \text{ s}^{-1}$. Electron density and temperature of the plasma beam were determined by the Thomson scattering (TS) technique [13]. The D flux to the surface was determined from the TS-measured T_e and n_e , assuming the ions were accelerated to the sound speed at the sheath entrance (Bohm criterion) [14]. The peak flux was about $1.5 \times 10^{24} \text{ m}^{-2} \text{ s}^{-1}$ and the full-width at half-maximum (FWHM) of the plasma beam was $\sim 12 \text{ mm}$. The ion energy was controlled by negatively biasing the sample and was fixed to $\sim 38 \text{ eV}$. The plasma conditions were kept constant throughout this study. Because of the heating of the copper coils at high magnetic field, Pilot-PSI operates in a pulsed mode. For all samples we used identical discharge durations of 70 s. During such a discharge, the peak fluence was about $8 \times 10^{25} - 1.1 \times 10^{26} \text{ m}^{-2}$. In order to reach higher accumulated fluence, the same discharge was repeated several times with a time lag between two shots of about 10 min, during which the sample was not exposed to any plasma flux.

The surface temperature was controlled by the water cooling system from the back side of the samples and was a balance between the incoming power flux from the plasma and the cooling efficiency. The peak surface temperature was measured by a spectral pyrometer (FMPI SpectroPyrometer, FAR Associates) during the experiments, while the 2D surface temperature profile was measured by a fast infrared camera (FLIR A645 sc). The spectral pyrometer measurements allowed the unambiguous determination of the surface temperature and emissivity, and were used to cross-calibrate the infrared camera. The measurement spot of pyrometer had a diameter of 2 mm, and the error of the temperature was smaller than 30 K. It took about 10 s for the surface temperature to rise from room temperature to the stable temperature and after the exposure the temperature of the samples was back to room temperature typically within 5 s, as shown in Fig. 1(a). Fig. 1(b) shows the stable temperature profile over the exposed surface. In the center area (radius $< 5 \text{ mm}$), the surface temperature was found to be rather homogeneous spatially, and the value in that region was taken as the exposure temperature. Because of the sample size, the cooling was more efficient for the center of the sample, so the temperature profile was not exactly the same as the particle flux profile.

After exposure, surface morphology changes of the center area (radius $< 5 \text{ mm}$) were observed using TESCAN MIRA 3 LMH high-resolution scanning electron microscopes (SEM). The orientation of the surface grains has been detected by Oxford instrument NordlysMax² electron backscatter diffraction technique (EBSD). The EBSD data was analyzed by the software Channel 5. To reduce the noise of EBSD data, the 'Zero Solutions' slider was set to five, and the wild spikes were eliminated by extrapolation.

The targets were analyzed with thermal desorption spectroscopy (TDS) at DIFFER, and the time lags between the plasma exposure and the TDS measurement for each sample were 1–2 weeks. The tungsten target was clamped to a ceramic heater and heated with a linear temperature ramp of 1 K/s to 1273 K. A Balzers QMA125 quadrupole mass spectrometer (QMS) monitored the mass 4 (D_2) and mass 3 (HD) signals in the residual gas in the chamber, to determine the total amount of deuterium released from the target during the temperature ramp. The absolute sensitivity was determined using calibrated leaks of H_2 and D_2 . For the sensitivity of the mass 3 signal, the average of the sensitivities of mass 2 and mass 4 was taken. Before every TDS measurement, calibration with a calibrated deuterium leak was performed. The error in the determination of the total amount of deuterium was estimated to be about 5%.

3. Results

3.1. Blistering

Fig. 2 shows the SEM images of the surface morphology of the W samples exposed to D plasma at different temperatures and with one single shot (the fluence was about $1 \times 10^{26} \text{ m}^{-2}$). It is evident from these images that the plasma exposure caused blistering on the W surface for all exposure temperatures. Blisters were observed even after exposure at temperatures as high as 1273 K. Fig. 3 shows the average size and density of blisters formed at different temperatures. The blister size is defined as the equivalent circle diameter of the surface area covered by the blister. For all exposure temperatures, the surface areas used to analyze were about $3000 \mu\text{m}^2$. With the higher exposure temperature, fewer blisters were observed on the surfaces, and the blister density decreased significantly, from $0.41 \mu\text{m}^{-2}$ for 493 K to $0.17 \mu\text{m}^{-2}$ for 943 K. With the increasing temperature, the average blister size decreased from 470 nm (493 K) to about 390 nm (from 943 K to 1273 K).

Fig. 4 shows the blistering behavior of surfaces irradiated up to different fluences at 943 K. Compared with Fig. 2(b), the blister density first increased strongly with increasing fluence, and then stopped increasing after the surfaces were exposed with 4 shots. Fig. 4(d) shows the size distribution of blisters formed on surfaces exposed with different fluences at 943 K. The number of blisters analyzed was about 600 for each case, and blister density was different for each fluence as labeled in Fig. 4(d). Most of the blisters had sizes smaller than 500 nm, and the most probable blister size appeared relatively unaffected by the ion fluence and stayed constant at around 200–300 nm. The increasing fluence did not lead to a further growth of blisters, and instead, a large number of small blisters ($< 500 \text{ nm}$) appeared on the surface.

Fig. 1. (a) Typical temperature evolution during the shots at different exposure temperatures, and (b) radial dependence of surface temperature when the temperature was stable.

Fig. 2. SEM images of the surfaces of W samples exposed to 38 eV D plasma at different temperatures after one shot (fluence $\sim 1 \times 10^{26} \text{ m}^{-2}$): (a) 493 K, (b) 943 K, and (c) 1073 K, (d) 1273 K.

Fig. 3. Blister density and average blister size of different temperatures after one shot.

Blisters did not appear uniformly over the whole surface, but showed strong dependence on the grain orientation, as shown on the surface exposed to plasma with fluence of $7.1 \times 10^{26} \text{ m}^{-2}$ at 943 K in Fig. 5. The inverse pole figure (IPF) coloring image shows that blisters mainly appeared on the grains with nearly (111) surface orientation. On the contrary, when the surface orientation is near (100), the surface modification was essentially absent. The textures of the W material we used were mainly {100} {110} and {111}//ND, and the grains with surface orientation near (110) were rarely found.

In many studies, high dome blisters with ratio of height to width around 0.3–0.5, were observed on W surface after D plasma exposure at 500–600 K [15–17]. In this study, high dome blisters were observed on plasma-exposed surface at higher temperatures as well. Fig. 6 shows top-view and cross-section images corresponding to typical high dome blisters formed at 943 K. Some of the blisters showed step-like structures. In addition, there were large cavities under the lids of the high dome blisters, and some cavities showed step-like structures consistent with the surface structure.

Fig. 7(a) shows the image of blisters (1)–(4) formed on a (111) surface oriented grain of the surface exposed to plasma with

fluence of $7.1 \times 10^{26} \text{ m}^{-2}$ at 943 K (8 shots). The blisters (1)–(3) had similar morphology with smooth top surfaces and faceted sides, and it is speculated that they were in different evolution stages of the large high dome blisters. The edges of the top surfaces were perpendicular to [110] or [112] direction. The edges were speculated to be left by the deformation of the surface layer in the direction normal to the surface. In order to observe the inner structure of the blisters by removing their lids, the plasma-exposed surface and a polished surface of another sample were put face to face, and then squeezed by a bench vise. The blisters lids were then broken due to the force applied vertically to the plasma-exposed surface. The force applied was not high compared to the strength of W, as not all lids were removed as visible in Fig. 7. Fig. 7(b) and (c) shows that there were some traces inside the blisters which were perpendicular to the [110] direction. All the directions were detected by EBSD technique.

3.2. Deuterium retention

Fig. 8(a) shows TDS spectra of D_2 release from the W samples exposed with one shot (the peak fluence was about $1 \times 10^{26} \text{ m}^{-2}$) at different exposure temperatures. At low exposure temperature (493 K), the desorption temperature ranged from 500 K to 900 K. At high exposure temperatures (943 K and 1073 K), there were obvious desorption signal from 700 K to 1200 K. The desorption temperature range was similar to the exposure temperature range across the surface of 943 K and 1073 K experiments as shown in Fig. 1(b). Fig. 8(b) shows the total D retention in the samples exposed to D plasma at different surface temperatures. The D retention shown is the average retention over the exposure surface, but the exposure temperature was not the same across the surface, so the D retention may be higher in the low temperature region away from the center. A strong decrease of the amount of retained deuterium was observed with a 2 order of magnitude decrease between 493 K and 1273 K. At 493 K, the total D retention was higher than 10^{20} D/m^2 , which is similar to the data reported in the literature, from $5 \times 10^{19} \text{ D/m}^2$ to 10^{21} D/m^2 with the exposure temperature around 500 K [5,16,18].

Fig. 4. SEM images of the surfaces of W samples exposed to 38 eV D plasma to different fluences at 943 K: (a) 2 shots (fluence $\sim 1.6 \times 10^{26} \text{ m}^{-2}$), (b) 4 shots (fluence $\sim 4.2 \times 10^{26} \text{ m}^{-2}$), (c) 8 shots (fluence $\sim 7.1 \times 10^{26} \text{ m}^{-2}$), (d) size distribution of blisters with various numbers of shots.

Fig. 5. (a) SEM surface morphology, (b) surface orientation IPF map of the same surface area exposed with 8 shots at 943 K.

Fig. 9(a) shows the evolution of the TDS spectrum measured on W samples exposed at 943 K to varying number of plasma shots i.e. to increasing fluences. Though the intensity of the desorption peaks increased with the increasing fluence, the position of the desorption peaks did not change. The temperature of maximum desorption appeared independent of the ion fluence and was 900 K for all samples. Fig. 9(b) shows the total D retention and accumulated fluences with various number of shots at 943 K. The fluences in this figure were the peak fluences over the exposure surfaces. At 943 K, with increasing fluence, the D retention increased rapidly at first. When the fluence increased by nearly a factor of two (from $9 \times 10^{25} \text{ m}^{-2}$ to $1.6 \times 10^{26} \text{ m}^{-2}$), the D retention increased by a factor of thirty (from $1 \times 10^{18} \text{ D/m}^2$ to $3 \times 10^{19} \text{ D/m}^2$). For higher fluence, the amount of D retained in the material approached saturation at $\sim 8 \times 10^{19} \text{ D/m}^2$ for incident fluences $\geq 4.2 \times 10^{26} \text{ m}^{-2}$.

4. Discussion

When W is exposed to low-energy D plasma, D atoms will agglomerate around the defects in the subsurface region, such as vacancies, dislocations, and grain boundaries. When the D concentration around the defects is high enough, the D atoms will recombine to form gas molecules, leading to the bubble formation in the near surface region [15,19]. In many studies, blisters with high dome are observed on the surface after D plasma exposure at 500–600 K, some of which are with step-like structures [15–17]. This kind of blisters is different from the ideal shape of blisters, whose dome is much lower, with the ratio of height to width around 0.05 [15,20]. Shu et al. assumed the high dome blisters were formed because there was an interconnected network of voids and bubbles in the surroundings of the blisters [15]. Other studies speculated that these blisters were caused by the plastic

Fig. 6. Surface morphology and cross-section image of blisters with high dome.

deformation of W [6,16,17]. Lindig et al. found the lateral elongation direction of the blisters coincided with the projection of the $\{110\}$ planes of W onto the surface, and speculated that this can be explained by the gliding of the BCC slip system [17]. In addition, Alimov et al. found at room temperature there were only low dome blisters formed on W surface, and they speculated that this was because below brittle-to-ductile-transition temperature the stress caused by D_2 gas cannot be relaxed by dislocations movement [16]. In the literature, it is also found that blisters tend to be formed on grains with (111) surface orientation, which is probably because $[111]$ is the most open direction of BCC lattice, so D ions may have a deeper penetration range, leading to more blistering [21,22].

In this study, we observed high dome blisters at high temperatures as well and more evidence for the relationship of blisters with the plastic deformation of BCC metals. For BCC metals, the slip direction is $[111]$, and the most ordinary slip planes are $\{110\}$ and $\{112\}$ planes [23]. At 943 K, when the surface orientation is near (111) , the edges of the blisters top-surface were perpendicular to the $[110]$ or $[112]$ directions and the traces at the bottom of the blisters were perpendicular to the $[110]$ direction, so both of them are quite likely caused by the sliding of the dislocations, as shown in Fig. 10. Sliding of dislocations is speculated to cause the growth of blisters in the direction normal to the surface. In the process of the growth of the high dome blisters, the surface layer may deform vertically many times, which may be the cause of the step-like

Fig. 7. (a) Surface morphology of blisters, (b and c) inner structure of blisters of the same grain as shown in (a).

Fig. 8. (a) TDS spectra of D_2 release, (b) total D retention in W samples exposed to 38 eV D plasma to exposed with one shot (fluence $\sim 1.0 \times 10^{26} \text{ m}^{-2}$) at different exposure temperatures. Note that the spectra of 943 K, 1073 K and 1273 K are magnified by 100 times.

Fig. 9. (a) TDS spectra of D_2 release, (b) total D retention in W samples exposed to 38 eV D plasma to exposed with various numbers of shots at 943 K. The fluences in this figure are the peak fluence over the exposure surfaces.

Fig. 10. Schematic diagram of high dome blisters.

structure of the high dome blisters. In this study, the surface orientation dependence of the blister formation is also observed at high temperatures, which is similar to low temperature results reported in [21,22]. It is speculated that blisters are more likely to be formed on (111) surfaces not only because [111] is the most open direction of BCC lattice, but also because the surface layer is easier to be deformed vertically when the surface orientation is near (111), since the slip direction of dislocations in BCC metals is [111] direction.

With increasing temperature, the deuterium retention was found to strongly decrease. This is mainly because at high exposure temperature, the low energy trapping defects cannot act as trapping sites for D atoms anymore. Since the amount of retained D atoms decreases, the probability for D_2 molecules to form and precipitate is strongly reduced, and consequently fewer blisters are observed at higher temperature. In previous studies, blisters were observed not to form on the surface irradiated by 38 eV D plasma at high temperature ($>750 \text{ K}$) [5,6]. The reason is probably that at such high temperature, D atoms desorption rate is quite high, so D atoms retention in the near surface region is no longer

possible [5,19,24]. However, in this study, blisters were formed on the surfaces exposed to D plasma at 943–1073 K, and there are D atoms retained in W after the exposure at high temperatures. The main difference between the previous studies and this study is the high plasma flux. In previous studies, the plasma flux used was usually lower than $10^{22} \text{ m}^{-2} \text{ s}^{-1}$, while in this study the flux is about two orders of magnitude higher, about $10^{24} \text{ m}^{-2} \text{ s}^{-1}$. It is speculated that the flux used in this study may exceed desorption rate of D atoms, so there were D atoms retained in W material after the exposure at 943 K and 1073 K. In many literatures, the desorption peak around 900 K was also observed, and this peak was attributed to the D atoms trapped by vacancies and bubbles [18,25]. In addition, Yang and Hassanein simulated the formation of D bubbles in tungsten by molecular dynamics, and bubbles were formed with high D particle flux at 900 K and 1200 K, and they also speculated the particle flux can play an important role in the formation of D bubbles, since if the D particle flux is high enough, then the D atoms will accumulate in the near surface region even at high temperatures, leading to super saturation of D atoms [26].

At low exposure temperatures (lower than 600 K), the saturation of D retention with the fluence was reported in literatures, and the saturation level varies from 10^{20} D/m^2 to 10^{21} D/m^2 for different grades of W materials [18,24]. In this study, the D retention also tends to saturate with increasing fluence at high temperature (at 943 K), as the D retentions of 4 shots and 8 shots are almost the same considering the error of about 5%. Zayachuk et al. found the D retention has strong relationship with the defects caused by the plasma exposure [18]. The formation of blisters will cause many defects to be formed simultaneously, such as voids, vacancies, and dislocations. Therefore after one shot, there must be some newly created defects left in the material, and these defects could act as trapping sites for D atoms during the next shot as demonstrated in [18], so there would be more sites for D atoms to accumulate and precipitate. Therefore, with increasing fluence,

there would be more blisters formed on the surface. However, the plasma-induced defect creation does not continue indefinitely, but saturates at a certain level. As a result, the D retention and blister density will saturate with increasing fluence.

5. Summary

In this study, the blistering behavior and deuterium retention in W exposed to high-flux D plasma at high temperatures with different fluences were investigated. The results are summarized as follows:

Blisters are formed on W surface exposed at different temperatures, ranging from 493 K to 1273 K. As the exposure temperature increases, blister density and D retention decrease. The formation of blisters at high temperatures is attributed to the high flux of D plasma. At 943 K, with the increasing fluence, there is trend to the saturation of D retention and blister density. The defects caused by plasma exposure have an important effect on the D trapping and blistering behavior. There are many high dome blisters formed on the surface, and the formation of high dome blisters has a strong relationship with slipping system of BCC metals.

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