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Thermal shock behaviour of blisters on W surface during combined steady-state/pulsed plasma loading

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Abstract

The thermal shock behaviour of blister-covered W surfaces during combined steady-state/pulsed plasma loading was studied by scanning electron microscopy and electron backscatter diffraction. The W samples were first exposed to steady-state D plasma to induce blisters on the surface, and then the blistered surfaces were exposed to steady-state/pulsed plasma. Growth and cracking of blisters were observed after the exposure to the steady-state/pulsed plasma, while no obvious damage occurred on the surface area not covered with blisters. The results confirm that blisters induced by D plasma might represent weak spots on the W surface when exposed to transient heat load of ELMs. The cracks on blisters were different from the cracks due to the transient heat loads reported before, and they were assumed to be caused by stress and strain due to the gas expansion inside the blisters during the plasma pulses. Moreover, most of cracks were found to appear on the blisters formed on grains with surface orientation near [1 1 1].

Keywords: blister, crack, tungsten, ELMs, pulsed plasma

(Some figures may appear in colour only in the online journal)

1. Introduction

Due to its favourable physical properties, such as low sputtering yield, high melting temperature and high thermal conductivity, tungsten (W) will be used as a plasma facing material (PFM) in the ITER divertor [1]. As a plasma facing material, W will be subjected to repetitive thermal shock caused by edge localized modes (ELMs), the number of which will be higher than 10^6 events during the divertor lifetime [2]. The maximum energy density of 0.5 MJ $\cdot$ m^{-2} has been adopted as the upper limit for controlled ELM energy loads in order to ensure an appropriate lifetime of the ITER divertor [3]. Previous studies have shown that transient heat loads will induce surface modifications, cracking and melting of tungsten surfaces [4, 5]. The occurrence of such ELM-induced damage is a significant threat to the lifetime of the divertor and its compatibility with high plasma performances. Besides the transient heat loads, tungsten will be subjected to steady state high fluxes and fluences of low energy plasma ions (D, T, He), which are known to lead to significant surface morphology changes such as blistering [6] and helium-induced nanostructures [7]. The recent observation of blistering at elevated temperatures (up to 1100 K) under ITER-relevant plasma conditions [8], calls for dedicated studies of the thermal shock resistance of blistered surfaces under ITER-like ELMs conditions.

The study of possible synergistic effects during simultaneous steady hydrogen plasma flux and transient heat loads is an area of active research on W materials [9–12]. The addition of plasma irradiation has been found to render tungsten more prone to damage during transient heat loads [9–12]. However, in those studies, the high heat flux induced severe surface damage on the surface whether it was covered by blisters or not, and the effect of blisters on the thermal shock behaviour of W was so far not investigated in details.

The goal of this study is to investigate the effect of a blistered surface on the thermal shock behaviour of W under ITER-like ELMs conditions. We first exposed W samples to high-flux D plasma to induce blistering on the surface. The pre-exposed surfaces were then exposed to combined steady-state/pulsed plasma loading. Evolution of the surface morphology was studied before and after each step to investigate the surface damage induced by the transient heat loads.

2. Experimental conditions

Polycrystalline tungsten samples with purity of 99.95 wt% were cut from a rolled sheet, which was supplied by Advanced Technology & Materials CO., Ltd. (China). The textures of the W sample are mainly {1 0 0} <1 1 0> and <1 1 1> // ND as reported in [8]. The samples were first exposed to a high flux D plasma beam in the Pilot-PSI linear plasma generator (step 1 exposure). The peak flux was about 1.5×10^{24} $m^{-2}s^{-1}$, and the ion energy was controlled by negatively biasing the sample and was fixed to ~38 eV. The plasma conditions were kept constant throughout this study. The samples were exposed to D plasma at two temperatures ~500 K and ~1000 K. 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. For each sample, the total fluence of D plasma was about 6–8 $\times 10^{26}$ m^{-2}. The detailed exposure conditions can be found in [8].

After the exposure to D plasma, all samples were exposed to combined steady-state/pulsed H plasma in Pilot-PSI using the pulsed plasma system described in [13, 14] (step 2 exposure). During the steady state, the ion temperature of the plasma was about 1–2 eV, and no bias was applied during this step exposure. During the plasma pulses, the ion density and temperature increased rapidly, increasing the heat flux to the target [13, 14]. During this step, each sample was exposed to one shot of steady-state/pulsed H plasma, during which the fluence of H particles was about 10^{25} m^{-2}. The time evolution of the surface temperature during the exposure was monitored by a fast infrared camera (FLIR SC7500MB) with a frame rate around 3 kHz. The evolution of the peak temperature during the exposure is shown in figure 1. The steady-state plasma duration was 10 s, and the plasma pulses were triggered for 5 s with a frequency 10 Hz (50 pulses in total). The temperature rise induced by the sub-ms (~ 0.7 ms) plasma pulse was ~700 K, after which it returned to the base temperature of ~800 K. The target heat fluxes were calculated using the THEODOR code [15], a 2D inverse heat transfer code. The heat flux of the steady state plasma was about 5 $MW \cdot m^{-2}$ for 10 s, while the energy density during the ELM-like pulse was about 0.3 $MJ \cdot m^{-2}$ (with a peak heat flux about 400 $MW \cdot m^{-2}$).

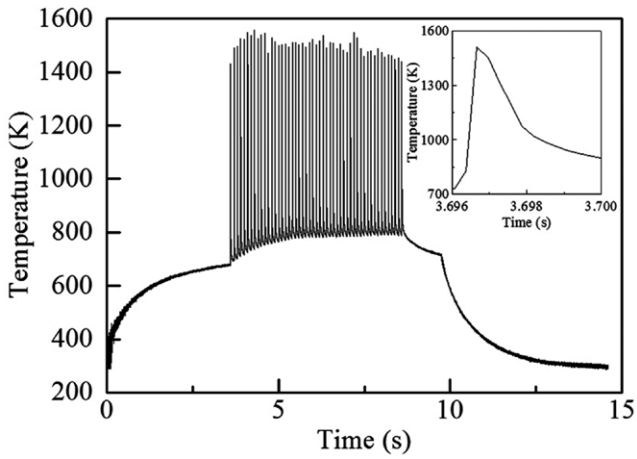

Figure 1. Surface temperature evolution during the combined steady-state/pulsed plasma loading.

The surface morphology changes of the samples after steps 1 and 2 were observed using a TESCAN MIRA 3 LMH high-resolution scanning electron microscope (SEM). The crystallographic grain orientation was analysed by Oxford instrument NordlysMax^2 electron backscatter diffraction technique (EBSD).

3. Results and discussion

Figure 2(*a*) shows the surface morphology of the sample exposed to D plasma (step 1) at ~500 K. The D plasma exposure caused blistering on the surface. Figure 2(*b*) shows morphology of the same area after the step 2 exposure to combined steady-state/pulsed H plasma. Cracking was not observed on most of the surface. The absence of cracks is consistent with the temperature during exposure being higher than the ductile–brittle transition temperature (DBTT) of W ~ 700 K [16], so after the exposure to the transient heat loads the material can endure the volumetric shrinkage and the correlated high tensile stresses [5]. However, as can be seen from the inset images in figure 2, some cracks were observed on the edge of the blister after the step 2 exposure. In addition, during the step 2 exposure the blister grew slightly, for instance by 100–200 nm in the position of the red arrow, and some cracks were observed on the growth front edge. Figures 3(*a*) and (*b*) show the surface morphology of another position before and after the step 2 exposure. The same position on the surface was located by marks made before the step 1 exposure, and the identical small blisters in figures 3(*a*) and (*b*) ensure that the pictures are indeed taken at the same location. After the exposure, a new blister structure appeared on the surface as indicated by the red arrows. As shown in the inset of figure 3(*b*), fine cracks were observed on the edge of the newly formed blister structure. Cross-section observation of this blister was obtained by focused ion beam (FIB) milling as shown in figure 3(*c*). The new blister structure on the surface

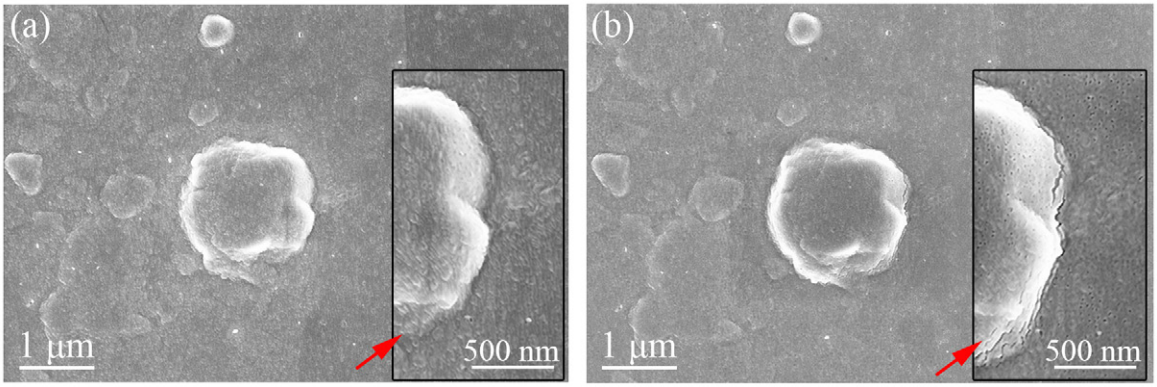

Figure 2. (*a*) SEM image of the surface after D plasma exposure at ~500 K; (*b*) same surface area after the exposure to steady-state/pulsed plasma, and growth and cracks occurred on the edge of the blister.

was formed due to the gas pressure inside the cavity along the grain boundary beneath the surface. However, the crack was invisible on the boundary of the blister from the cross-section morphology in figure 3(*c*), which indicates that the crack did not extend much.

The fatigue cracks on tungsten surface exposed to high heat loads were reported in many literatures [4, 5], but fatigue effects would be expected only at much higher cycle numbers compared with the pulse number of present study (only 50 and even 10 plasma pulses), so the cracks on blisters observed here should not be the fatigue cracks. Moreover, if the cracks on blisters were caused by fatigue, then the fact that cracks were only formed on blisters can only be explained by the mechanical properties degradation due to blister structures. However, on the W surface pre-exposed to D plasma at 1000 K, although blister structures were also observed on the surface after the step 1 exposure (as shown in figure 4(*a*), which is consistent with our previous observations [8]), no such cracks were formed on blisters after the exposure to steady-state/pulsed plasma, as shown in figure 4(*b*). Therefore, the crack appearance on blisters formed at 500 K are not fatigue cracks of blistered surface due to the thermal stresses under repetitive pulsed heat loads reported before [4, 5]. The cracks observed on blisters in this study are very fine and concentrated around blisters, and as such are a new type of surface damage due to transient heat loading.

To clarify the mechanisms of crack formation on blisters, a reference sample (also pre-exposed to D plasma at ~500 K) was exposed to similar steady-state plasma conditions in the absence of plasma pulses to isolate the effect of the combined exposure with that of possible morphology changes induced by the H plasma alone. On the surface of the reference sample, there were no cracks or growth features observed on the blisters after the exposure to steady-state H plasma. Therefore, the growth and cracks of blisters must be caused by the plasma pulses during the step 2 exposure. The plasma pulses may affect the blisters in two aspects. First, the temperature rise due to the transient heat load will make the gas inside the blisters expand rapidly. Secondly, during the pulses the ion temperature will increase to about 5–15 eV, and the particle density will increase as well by up to an order of magnitude

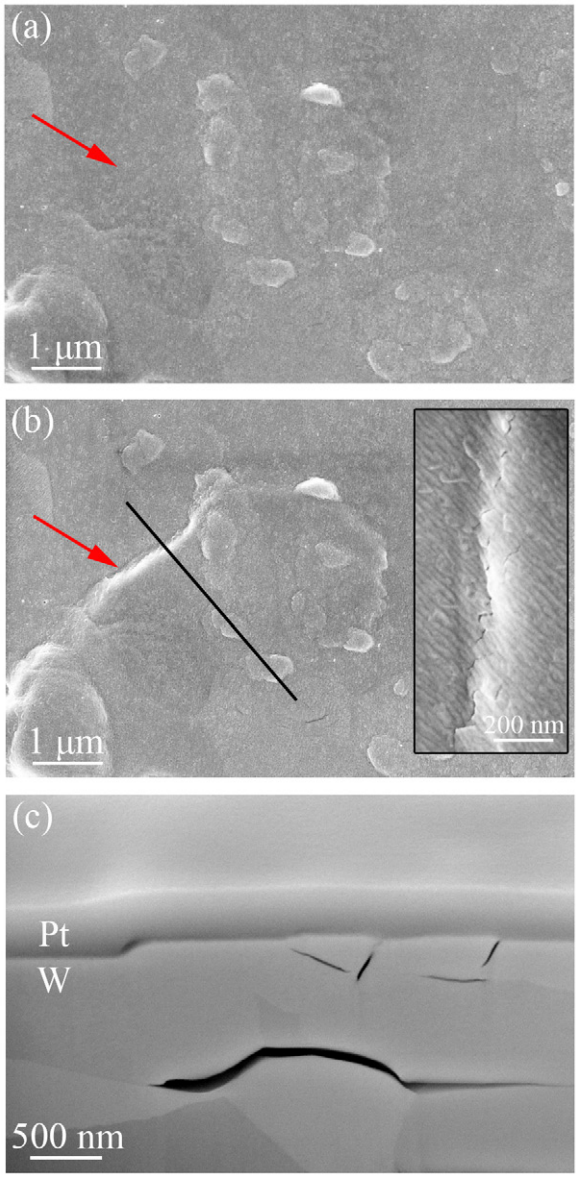

Figure 3. (*a*) SEM image of the surface after D plasma exposure at ~500 K; (*b*) same surface area after the exposure to steady-state/pulsed plasma, and a new blister appear with fine cracks on the edge shown in the inset; (*c*) the cross-section morphology along the black line in (*b*).

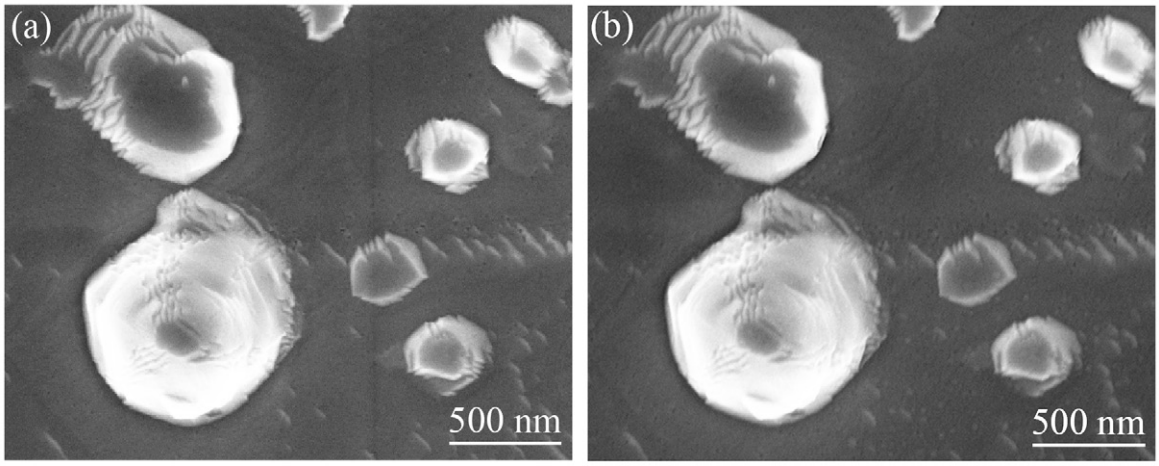

Figure 4. (*a*) SEM image of the surface after D plasma exposure at ~1000 K; (*b*) same surface area after the exposure to steady-state/pulsed plasma, and no obvious changes occurred to the blisters.

[13, 14], which may increase the H concentration and gas quantity inside the blisters. However, the same steady-state/pulsed plasma loading did not induce any blisters on another polished W sample surface without being pre-exposed to D plasma. This means the hydrogen retention in W induced by the pulsed plasma was little. In addition, the cracks were also observed on another pre-exposed sample after the step 2 exposure even with only 10 plasma pulses, and in this case the total duration of the ELM-like pulses was only about 10 ms, so the gas quantity increase in the sample due to the plasma pulses should be negligible. Therefore, the growth and cracks of the blisters were likely caused by the transient heat load of the plasma pulses. Many researchers estimated that the gas pressure inside a blister on W surface could be a few hundred MPa [17, 18]. In this study, the temperature rise induced by a plasma pulse will induce a strong increase of the gas pressure inside the blister. The gas pressure will severely deform the surface layer above, resulting in the growth and crack of blisters. In addition, the high temperature ramp rate during a plasma pulse (~1×10^6 K s^{-1}) will induce a high strain rate. W materials mechanical properties are very sensitive to the strain rate, and the high strain rate will decrease the ductility of W materials [19, 20]. Therefore, although the exposure temperature was above the DBTT of W materials, still cracks were formed on the blister surface. As shown is figure 3(*c*), the cracks did not penetrate deeply in the present exposure condition. However, the effect of larger pulse number on crack propagation and blister stability should be examined in the future—it is indeed known that significant surface damage can occur for large pulse numbers (~1E6) even for low energy densities [21].

Several studies reported that the addition of hydrogen plasma exposure could lead to an increased damage level caused by transient heat loads [9–12]. However, in those studies, the transient heat loadings were different from that of ELMs in many aspects, such as heat source, heat flux and pulse length. In addition, the transient heat loading induced severe damage to the surfaces whether blistered or not, so the effect of blisters on the thermal shock behaviour was not investigated. In the present study, the transient heat loading is achieved using plasma pulses superimposed on a high flux steady-state plasma very similar to that of controlled ELMs in ITER in terms of the heat source (pulsed plasma), the energy density (around 0.3 MJ·m^{-2}), and the heat pulse length (sub-ms). Under our experiment conditions, the transient heat loads of pulsed plasma induced cracks on the edge of blisters, while no obvious damage was observed on the non-blistered surface. This indicates that blisters are more sensitive to transient-induced damage.

For the blisters formed at ~1000 K, no cracks or growth features were observed after the same steady-state/pulsed H plasma loading, as shown in figure 4. Our previous study found that the deuterium retention was much lower than that at ~500 K [8], which may be because of desorption of D atoms during the cooling down period after the high temperature exposure. Therefore, although there were blister structures after the D plasma exposure at ~1000 K, the gas quantity inside the blister should be low. The low gas quantity and pressure inside the blister cannot induce enough stress to the surface layer, so the transient heat load is unable to cause growth or cracks on blisters.

As can be seen from figures 2(*b*) and 3(*b*), the cracks mainly occurred on the edge of the blisters, while in the blister centre cracks were not observed. This is mainly because the stress and strain fields due to the gas pressure are not spatially homogenous in the blister. In previous studies, theoretical model and finite element method both found the stress and strain reaches the maximum at the boundary of the blister [17, 22, 23]. During the exposure to the transient heat load although the gas pressure increased homogeneously inside the blister, the strain and stress will be concentrated in the region of blister boundary as a crack tip. Therefore, the deformation caused by the gas pressure was more severe on the edge than other positions of the blister, so cracks tended to occur on the edge of blisters.

However, on the surface exposed to D plasma at ~500 K, cracks did not appear uniformly on all blisters after the step 2 steady-state/pulsed plasma loading. As figure 5 shows, cracks were observed on the edge of the blister in figure 5(*a*), while the blisters in figure 5(*b*) did not show any crack features on the edge after the step 2 exposure. We found the crack formation on blisters had strong relationship with the lattice orientation of the surface normal (SN). The correlation between the

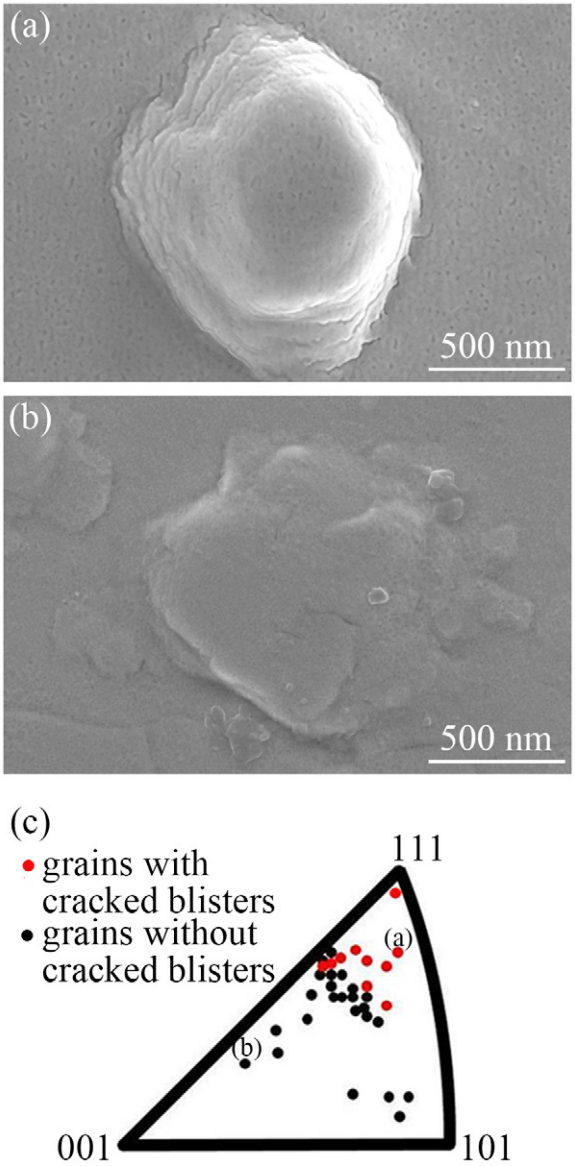

Figure 5. (*a*) A blister with cracks on the edge after the exposure to steady-state/pulsed plasma; (*b*) a blister without cracks on the edge after the exposure to steady-state/pulsed plasma; (*c*) the surface orientations of the grains with or without cracked blisters shown in the inverse pole figure.

crack formation and the orientation of the SN was analysed by SEM and EBSD. The surface orientations of the grains with or without cracked blisters were summarized in the inverse pole figure in figure 5(*c*) including the surface orientations of grains shown in figures 5(*a*) and (*b*). Blisters were observed on all grains shown in figure 5(*c*), but the cracks appeared mostly on the blisters formed on surfaces with a surface orientation near [1 1 1], while the blisters on surfaces with SNs lying elsewhere in the inverse pole figure did not show any cracks. In many studies, the blisters were form preferentially on the surface with [1 1 1] surface orientation [8, 24, 25]. In our previous study, the blister structure was found to have strong relationship with the deformation mechanism of the W material [8].

Because the slip direction of dislocations in BCC metals is [1 1 1], so on [1 1 1] surface, the surface layer is more easily deformed by the gas pressure inside the cavity beneath the surface, resulting in an enhanced susceptibility to blistering. Also, as can be seen from figure 3, the growth process of blister during the exposure to transient heat load was quite similar to that during the exposure to D plasma, and it is also the result of the surface layer deformation induced by the gas pressure inside the blister. Therefore, during the exposure to pulsed plasma, the gas pressure will deform the blisters on [1 1 1] surfaces more severely, resulting in more cracks. In summary, [1 1 1] surfaces are more prone to blister formation during D plasma exposure, and blisters formed on [1 1 1] surfaces are more sensitive to cracking during transient heat loads.

4. Conclusions

In this study, the blistered W surfaces were exposed to combined steady-state/pulsed plasma. The power density of the sub-ms (~0.7 ms) plasma pulses was around 0.3 MJ·m^{-2}, relevant for mitigated ELMs in ITER. The pulsed plasma used in this study induced cracks on the edge of blisters on W surface, but no obvious damage was observed in the surface area without blisters. Therefore, these results confirm that the blisters induced by D plasma have negative effect on the thermal shock behaviour and might represent weak spots on the surface of W materials under the ELMs condition of ITER divertor. These blister cracks were mainly caused by stress and strain due to the gas expansion inside the blisters during the pulsed plasma. Blister cracking during transient events represent a new type of surface damage induced by the high heat flux, which is different from the crack formation on W surface exposed to high heat flux reported in previous studies.

In addition, cracks due to the transient heat flux did not appear on all blisters, and most cracks were found to appear on the blisters formed on grains with surface orientation near [1 1 1]. This is mainly because the deformation of the surface layer caused by the gas expansion during transient heat loading was more severe on [1 1 1] surface.

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