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Deuterium-induced nanostructure formation on tungsten exposed to high-flux plasma

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ABSTRACT

Surface topography of polycrystalline tungsten (W) have been examined after exposure to a low-energy (38 eV/D), high-flux ($\sim 1.1–1.5 \times 10^{24}\ m^{-2}\ s^{-1}$) deuterium plasma in the Pilot-PSI linear plasma device. The methods used were scanning electron microscopy (SEM), transmission electron microscopy (TEM), positron annihilation Doppler broadening (PADB) and grazing incident X-ray diffraction (GI-XRD). After exposure to high flux D plasma, blisters and nanostructures are formed on the W surface. Generation of defects was evidenced by PADB, while high stress and mixture of phases were detected in depth of 50 nm by GI-XRD. TEM observation revealed fluctuations and disordered microstructure on the outmost surface layer. Based on these results, surface reconstruction is considered as a possible mechanism for the formation of defects and nanostructures.

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1. Introduction

Tungsten (W) is currently the plasma-facing material for the ITER divertor and a candidate for future fusion devices owing to its good thermal properties and low erosion by incoming plasma particles. Blister formation has been widely observed on W surfaces for bombardment by deuterium (D) plasma with flux lower than $10^{23}\ m^{-2}\ s^{-1}$, and depends on ion fluence, energy and surface temperature [1–6]. Recently, the surface modification of W exposed to high flux D plasma ($\sim 10^{24}\ m^{-2}\ s^{-1}$) similar to that expected in the ITER divertor region during operations was investigated by the authors [7]. Much smaller sized blisters are observed on tungsten surface with insisting appearance at elevated temperature (up to 873 K). A new type of surface nanostructure was also identified on the surface under those conditions suggesting that the ion flux plays an important role on the enhancement of the surface modification. The importance of ion flux was already identified in case of helium ion irradiation, where the development of "nano-fuzz" depends on the incoming He ion flux, and that for fluxes higher than $7 \times 10^{21}\ m^{-2}\ s^{-1}$, it only depends on the exposure time [8]. With that in mind, the significance of such morphology changes for the behavior of tungsten under fusion-relevant conditions is far from clear, in particular in the divertor region where the particle flux is the highest ($>10^{24}\ m^{-2}\ s^{-1}$).

In this paper, efforts are made to explore the mechanism of surface modification by high flux D plasma exposure. The surface morphology is observed by high resolution scanning microscope (SEM). Positron Annihilation Doppler broadening (PADB) is used to monitor the defect evolution by plasma exposure. Grazing incident X-ray diffraction (GI-XRD) and transmission electron microscopy (TEM) are applied to analyze the microstructure of exposed surface.

2. Experimental procedures

The preparation of the tungsten samples used in this study has been described in more details in [9]. Briefly, polycrystalline tungsten samples with diameter of 30 mm and thickness of 3 mm were cut from a rolled sheet and electro-polished, and then outgassed at 1273 K for 1 h at a background pressure of 5×10^{-4} Pa before implantation. The purity of the samples is 99.95 wt% with main impurities Mo, Fe, C around 50 ppm, O about 2500 ppm.

The plasma exposure is performed in the Pilot-PSI linear plasma device, which is uniquely capable of producing low energy plasmas with a high flux ranging from $10^{23}–10^{25}\ D\ m^{-2}\ s^{-1}$ and with energy

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fluxes up to 50 MW m^{-2} [10]. The ion energy is fixed at 38 eV by negatively biasing the target. The electron density and temperature are measured by Thomson scattering, and are used to calculate the ion flux. The plasma beam with a Gaussian profile is obtained with the peak flux of about 1–2 × 10^{24} D m^{-2} s^{-1} in the center and a beam diameter of ~1 cm. Several consecutive shots are carried out to reach the required fluence, with effective heating-up and cooling down of 1–2 s. The surface temperature is determined by the heat flux of the plasma and active cooling of the target holder, and is monitored by a fast infrared camera (FLIR SC7500-MB) during experiment.

Surface morphology is observed by secondary electron (SE) mode and inlens mode [11] in a high resolution SEM. PADB was used to investigate the formation of defects in the crystal lattice caused by plasma exposure, the details of the set-up of experiments can be found in [7]. GI-XRD and FIB–TEM are carried out to characterize the surface structure. The samples are analyzed with 50 KV Cu Kα XRD analysis, the step width is 0.04°, and time per step is 7.0 s.

3. Results and discussions

Fig. 1 shows a typical view of the sample surface after exposure in Pilot-PSI to a plasma fluence of 7 × 10^{26} D m^{-2} at a surface temperature of 423 K. In Fig. 1(a), blisters with irregular shapes and relatively small size are observed widespread on the exposed surfaces. The most popular blister size is measured to be ~0.3–0.4 μm, while the maximum diameter d_{max} is found to be lower than 2 μm. Fig. 1(b) shows a high magnification image of the highlighted rectangle in Fig. 1(a). Some nano-structures are present homogeneously on the surface, even on the top of blisters, but appear in different shapes within different cells/grains. In total, three types of nanostructures are found on the surface, which can be described as triangles, ripples and sponge-like structures. Detailed information of the evolution of nanostructures with fluence and surface temperature and its dependence on grain orientations has been reported in previous investigations, which can be found in [7,12].

To obtain insights on the effect of high flux deuterium plasma exposure, the sample was then analyzed by PADB with the Variable Energy Positron (VEP) beam at Delft Technology University, as has been reported in [7]. Fig. 2 shows the measured *S* parameter as a function of positron annihilation depth in Fig. 2(a) and *S* vs. W data in Fig. 2(b). The Doppler broadening spectrum of a virgin rolled-W sample is also included for comparison (Fig. 2, ●). Taking the *S* parameter of 0.46 from annealed ‘defect-free’ tungsten as the reference bulk *S* parameter, the *S* parameter of the rolled-W sample is increased by 4.3% (Fig. 2(a)), suggesting that the non-exposed material readily contains defects such as dislocations and small vacancies, which is typical for a rolled polycrystalline material. After analysis by VEPFIT program [13], a positron diffusion length of 20 nm is obtained, which is much smaller than the value of 100 nm for the defect-free tungsten. This also evidences the presence of defects in rolled-W before plasma exposure. After plasma exposure, a significant increase of *S* profile in tungsten (Fig. 3, 🟥) is observed. The increase of the *S* parameters is found to be ~10% above the defect free reference value (indicated by the red dash line in Fig. 2(a)). The affected area by D plasma is extended to a depth of ~250 nm, and the significantly affected depth is ~100 nm, as depicted in Fig. 2(b). In general, for a positron trapped in an open volume defect (such as a dislocation, a vacancy or vacancy cluster or voids) the probability of annihilations with core electrons is reduced compared with that for valence electrons resulting in a higher *S* parameter [14]. Therefore, the generation of defects, mainly dislocations, vacancy/vacancy clusters by high flux D plasma is suggested by the PADB measurements. And the narrow distribution of created defects can be related to the low mobility of defects and D-defect complexes at ~423 K.

The question rises as how could defects be created by D ions with energy well below the displacement threshold. Since several hypotheses have been proposed based on the high stress caused

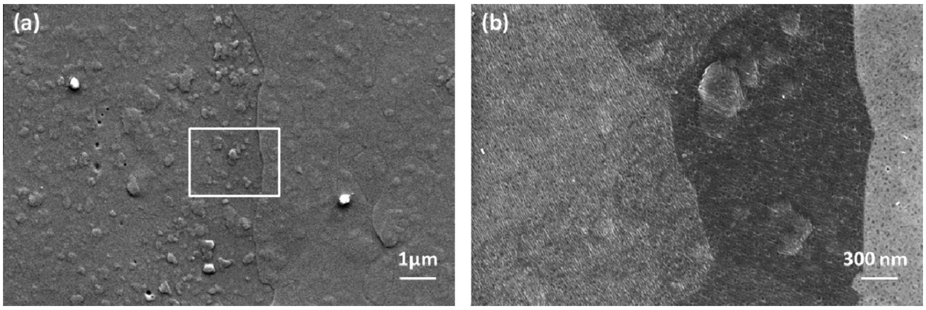

Fig. 1. Surface morphology of tungsten after exposure to high flux D plasma with fluence of 7 × 10^{26} D m^{-2} at ~423 K, (a) blisters shown by SE image and (b) nano-structures shown by inlens image.

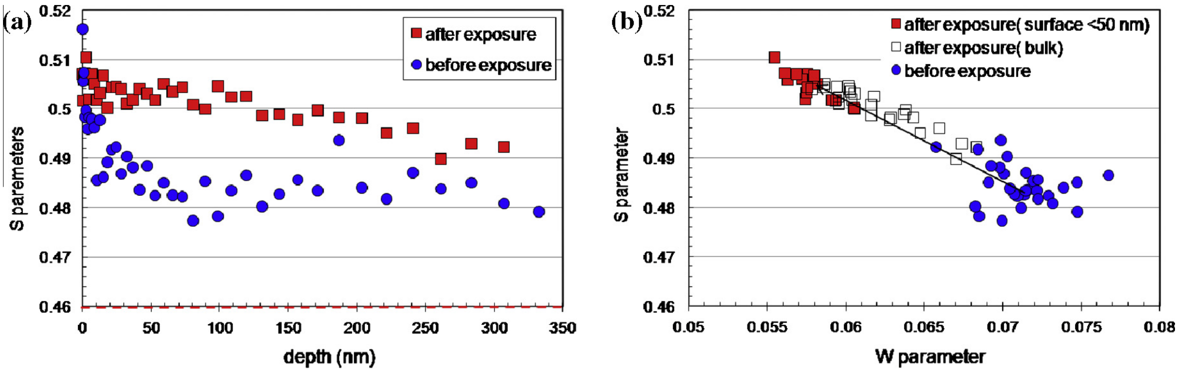

Fig. 2. PADB results of W before and after exposure to high flux D plasma with fluence of 7 × 10^{26} D m^{-2} at ~423 K, with S parameter versus depth in (a), and SW values in (b).

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