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Influence of nitrogen pre-irradiation at different temperatures on surface blistering and deuterium retention in tungsten

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ABSTRACT

Nitrogen seeding is considered to be used to reduce the power load onto tungsten divertor armors in tokamaks. Consequently, plasma-material interactions will be affected by seeded nitrogen impurities. The influence of nitrogen pre-irradiation on deuterium induced blistering and deuterium retention in rolled tungsten has been investigated at different pre-irradiation temperatures in the linear plasma device STEP with ion flux > $10^{21}\,m^{-2}\,s^{-1}$. Commercially rolled W samples were sequentially exposed to nitrogen plasma (380 K and 470 K) and deuterium plasma (510 K). Surface observations show that nitrogen pre-irradiation suppresses deuterium-induced surface blistering. Thermal desorption spectroscopy measurements indicate that deuterium retention in the rolled tungsten is reduced due to nitrogen pre-irradiation, and it significantly changes with the pre-irradiation temperature. These phenomena are supposed to be related to the formation and change of WN_xs at different temperatures which is validated by X-ray photoelectron spectroscopy analyses.

1. Introduction

Tungsten (W) is a leading candidate for plasma facing materials (PFMs) in fusion devices because of its high melting point, high thermal conductivity and low sputtering yield for light elements [1]. From the material research perspective, one of the key challenges is to ensure that PFMs can survive under the extreme heat loads, both for steady-state and transient conditions [2,3]. Impurity seeding (noble gases or nitrogen) is applied to reduce the power load onto the divertor armors. Extrinsic gas species are injected into the plasma edge and dissipate a significant fraction of the incoming power by radiation [4]. However, impurity seeding results in complicated plasma-material interactions. One of the important issues is the interactions between seeded impurities and W-PFMs, and its influence on fuel retention in W-PFMs [5–7].

The influence of nitrogen (N) pre-irradiation on deuterium (D) retention in W has been studied in the electron cyclotron resonance plasma chamber 'PlaQ' with low-flux plasmas ($\sim 10^{19}\,m^{-2}\,s^{-1}$) [8]. The results indicate that N pre-irradiation at 500 K influences surface blistering and increases D retention in W, while few changes have been found when the pre-irradiation temperature reduces to 300 K. The formation of W-N layer is supposed to be responsible for these observations [8]. However, the change of WN_x components on the bulk W surface irradiated by high-flux N plasmas ($> 10^{21}\,m^{-2}\,s^{-1}$) at different temperatures is little known, and the underlying mechanism of how D behavior in W is affected by N pre-irradiation at different temperatures needs to be revealed.

In this work, W samples were sequentially exposed to N and D plasmas with a flux of $\sim 10^{22}\,m^{-2}\,s^{-1}$ and a high fluence of $\sim 10^{26}\,m^{-2}$ in the linear plasma generator STEP [9]. The effect of N pre-irradiation at different temperatures on surface blistering and D retention in W is studied, and the relevant mechanism is discussed.

2. Experimental details

The commercial polycrystalline tungsten (> 99.95 wt.% purity) with grain size of 2–5 μm was used in this work, which was produced by Advanced Technology and Materials Co., Ltd China. The specimens with dimensions of $10 \times 10 \times 1\,mm^3$ were cut from a rolled W sheet. They were electrolytic polished. Thereafter, the W samples were annealed at 1273 K for 1 h at a background pressure of 5×10^{-4} Pa.

N and D plasma exposures were carried out in the linear plasma generator STEP located at Beihang University [9]. The plasma was produced by a LaB_6 source and confined by an axial magnetic field of < 0.1 T. Electron density, temperature and the flux of the plasma beam were measured by a triple Langmuir probe. The exposure temperature was measured by a K-type thermocouple attached to the back of targets. The impinging ions were accelerated by applying a negative

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bias voltage of 80 V. According to [10], the N ions at this condition are easier to be ionized to N^+ and $N_2{}^+$ and D ions could be ionized to D^+, $D_2{}^+$ and $D_3{}^+$. We are not sensitive to the ion species and assume that the N ions are N^+ and the D ions are D^+. In the N irradiation we applied the arc voltage of 71 V, the arc current of 28 A and the working pressure is 0.1 Pa. In the D implantation experiment the arc voltage is 65 V; the arc current is 20 A and the working pressure is 0.3 Pa. The W samples were first exposed to N plasma at 380 K and 470 K with a flux of 1×10^{22} N m^{-2} s^{-1} and a fluence of 1×10^{26} N m^{-2}, and then to D plasma at 510 K with a flux of 1×10^{22} D m^{-2} s^{-1} and a fluence of 1×10^{26} D m^{-2}.

After plasma exposures, surface morphology was observed by a scanning electron microscope (SEM, MIRA3 TESCAN). D desorption behaviors in the irradiated W were analyzed by thermal desorption spectroscopy (TDS). The tungsten targets were heated up to 1273 K with a ramping rate of 1 K/s. The residual gases such as D_2 (mass 4), HD (mass 3) were monitored by a quadrupole mass spectrometer (MKS micro Vision). When calculating the total D retention, D from HD and D_2 was taken into account. The uncertainty in the measurement is mainly attributed to calibration process, which is about 5% in the present setup. To investigate the formation and change of nitrides during pre-irradiation, X-ray photoelectron spectroscopic analysis (XPS) were performed on N-irradiated W samples. The measurements were conducted in a ESCALAB 250Xi XPS system using Al Kα source (CAE mode: Pass Energy 50.0 eV). Sputtering depth profiling was achieved by argon (Ar) ions etching. The energy of Ar^+ was 3000 eV and the ion current density was 231.8 nA/mm^2. The tilt angle of 50° with respect to the surface normal was applied.

3. Results

3.1. Surface morphology

Fig. 1 shows surface morphology of the W samples before and after being exposed to different plasma species at different temperatures. In the N-only case as shown in Fig. 1(b) and (c), surface morphology does not change significantly. After D implantation, we find the surface with N pre-irradiation is easier to be sputtered by D as shown in Fig. 1(f). With only D implantation at 510 K, the surface is covered by a large number of small blisters with an irregular shape and a diameter of 0.1–5.0 μm as shown in Fig. 1(d). Most of the blisters size are 0.5–1.0 μm and just very few blisters are 3.0–5.0 μm. We have calculated the area of the blisters in a random area of 500 × 500 μm^2 and eventually find that blisters account for ~4.6% of the surface. For samples sequentially exposed to N plasma and D plasma, surface blistering is quite different compared to the D-only case. As shown in Fig. 1(e), with N pre-irradiation at 380 K, only a small number of blisters with a diameter of 0.1–5.0 μm are observed. Most of the blisters size are 0.2-0.5 μm and the blistering area ratio decreases to 3.7%. While with N pre-irradiation at 470 K (Fig. 1(f)), a tiny minority of 0.1–3.0 μm blisters are found on the exposed surface. Most of the blisters size is about 0.1–0.2 μm and the blistering area ratio dramatically reduces to about 0.4%. Therefore, the surface morphology observations indicate that N pre-irradiation can inhibit D-induced surface blistering, and it exhibits a strong temperature dependence.

3.2. Deuterium retention

The D_2 desorption spectra for the targets exposed to D-only and sequential N–D plasmas are shown in Fig. 2(a). The target only exposed to D plasma at 510 K has a dominant desorption peak at 735 K. The dominant desorption peak for 470 K N pre-irradiation shifts to 667 K, and the total desorption significantly decreases compared to the D-only case. The shape of the 380 K N pre-irradiation spectrum differs from the other two spectra. There are two peaks. The main peak is located at ~600 K, and a shoulder peak appears at 485 K.

3.3. Chemical states of W-N surface layer

Fig. 3 presents the XPS results of the W sample surface irradiated by N plasma at 380 K and 470 K, as well as the initial surface (labeled as "blank"). The spectra of N 1s is shown in Fig. 3(a). In "blank" W, the peak is located at 399.87 eV which corresponds to molecular nitrogen adsorbed on W surfaces[11]. The samples after N implantation at 380 K and 470 K show peaks at 396.82 eV and 397.67 eV respectively. According to literatures [12,13,14], the N 1s peak for tungsten nitrides

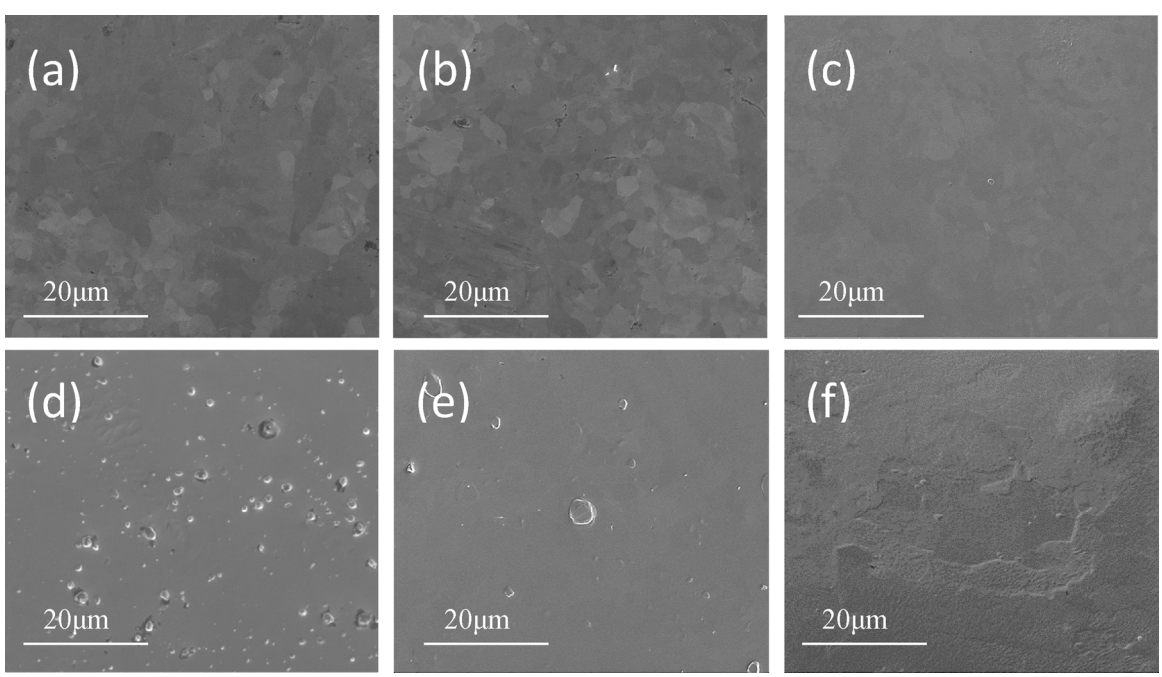

Fig. 1. Surface morphology of W samples with or without N pre-implantation at different temperatures: (a) initial W, (b) N380 K, (c) N470 K, (d) D510 K, (e) N380 K_D510 K, (f) N470 K_D510 K.