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The effect of ion flux on plasma-induced modification and deuterium retention in tungsten and tungsten–tantalum alloys

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ABSTRACT

The paper presents the results of an experimental study of deuterium retention in W and W–Ta alloy that were exposed to first-wall relevant low flux ($\sim 10^{20}\ \mathrm{m^{-2}\ s^{-1}}$) deuterium plasma in the ECR plasma generator PlaQ. Subsequent analysis included surface imaging by optical microscopy, deuterium depth profiling by nuclear reaction analysis (NRA) and measurements of deuterium content by thermal desorption spectroscopy (TDS). It was found that under investigated exposure conditions the deuterium content was higher in W–Ta alloy than in W. Combined with the previously reported results showing that under high-flux ($\sim 10^{24}\ \mathrm{m^{-2}\ s^{-1}}$) retention is higher in W instead, this gives rise to a peculiar flux effect – dependence of relative retention between different materials on exposure flux. We interpret this effect as evidence that at different flux ranges different populations of trapping sites determine the retention, namely pre-existing microstructural traps at low-flux exposure and plasma-induced ones at high-flux exposure.

1. Introduction

Tungsten is suggested to be used as a divertor material in ITER and possibly as a first wall material in future fusion reactors. Tungsten is known to have certain unfavorable properties hindering its use as a plasma-facing material (PFM), such as susceptibility to surface cracking under ELM-like transient heat loads [1] due to the high brittle-to-ductile transition temperature [2]. Several alloys were proposed with advantageous thermo-mechanical properties, one of such alloys being W–Ta [3], which was demonstrated to have better resistance to degradation under repetitive ELM-like heat loads [4].

It is known that exposure of tungsten-based materials to deuterium plasma leads to modification of these materials. It can manifest itself by the formation of subsurface cavities associated with surface blisters [5,6], as well as by the generation of atomic defects [7]. Plasma-induced material modification was also found to significantly influence deuterium retention, leading to the emergence of a history effect – the enhancement of the deuterium retention after the pre-exposure to low-energy, high-flux deuterium plasma [8].

Reports concerning deuterium retention in W–Ta available in the literature were initially contradictory (compare, for example, [9,10]). These contradictory results were pointed out to originate in the different ion fluxes where the studies were performed. In our previous publication [9], we suggested that the discrepancy might be explained by the difference in dynamics of plasma-induced defect formation that depends on ion flux.

These considerations were the motivation for the present work. In this contribution we present the results of the measurements of retention in W and W–Ta exposed to first-wall relevant low-flux plasma. We compare this to the earlier published results on retention in the same material grades performed at divertor-relevant high-flux exposure conditions. In addition, the interaction of low-flux plasma with materials pre-exposed to high-flux plasma is investigated, providing an indication of the difference between processes of deuterium uptake at low and high plasma fluxes.

2. Experiment

Powder metallurgical W and W–Ta alloy, containing 5 mass% of Ta, both provided by Plansee, were investigated. All investigated

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polycrystalline samples were mechanically polished to mirror finish with 0.05 μm alumina polishing suspension, and then annealed in vacuum at 1300 K for 1 h. During annealing the samples were not recrystallized. The samples were disks with a diameter of 20 mm and a thickness of 1 mm.

The low-flux exposures were performed at the electron cyclotron resonance (ECR) plasma generator PlaQ at the Max Planck Institute for Plasma Physics (Garching, Germany). Details of the design and operation of the device can be found in [11]. The energy of the ions arriving at the sample's surface is determined by a floating potential (no additional bias was applied), established at ~15 V. The majority of ion flux is carried by D^{3+} ions, which means that the energy per deuteron is equal to one third of the ion energy, ~5 eV. The exposure conditions were the following: ion flux $\sim 5*10^{19}\ m^{-2}\ s^{-1}$, surface temperature 300 K (RT), and duration of exposure 48 h, corresponding to a fluence of $\sim 10^{25}\ m^{-2}$.

Two sets of samples were exposed. The first consisted of W and W–Ta samples which were polished and thermally treated but not previously exposed to plasma (referred to as "reference samples"). The second set consisted of W and W–Ta samples which were previously exposed to high fluences ($\sim 10^{27}\ m^{-2}$) of high-flux ($\sim 10^{24}\ m^{-2}\ s^{-1}$) deuterium plasma at surface temperature of ~450 K, and subsequently degassed (referred to as "pre-exposed samples").

Imaging of the surfaces after plasma exposure was performed using optical microscopy.

For the deuterium depth profiling we implemented the commonly used $D(He^3; p)\alpha$ reaction. The use of this reaction for the investigation of D in tungsten is described in [12]. The energies used for the detailed depth profiling were 500 keV, 690 keV, 1.2 MeV, 1.8 MeV, 2.4 MeV, and 3.2 MeV. The reconstruction of depth profiles from raw NRA data (alpha and proton energy spectra) was performed with the use of the software packages SimNRA [13] and NRADC [14].

Total retention was measured with TDS. The maximum temperature during the run was 1273 K, the temperature ramp 0.5 K/s, and the holding time at the maximum temperature 5 min. The release fluxes of molecular HD and D_2 were measured by a quadrupole mass spectrometer.

The experimental sequence was the following. The samples – reference and pre-exposed ones – were exposed to low-flux deuterium plasma in PlaQ. After the exposures the surfaces of the samples were imaged in order to investigate whether any surface structures had formed. Subsequently, the deuterium depth profiles were reconstructed using NRA. Finally, the samples were degassed in TDS runs while recording deuterium release spectra.

3. Experimental results

3.1. Surface imaging

Fig. 1 shows the surface images of the reference W and W–Ta samples after exposure to low-flux plasma in PlaQ. It can be seen that neither material exhibits characteristic plasma-induced features such as blisters: only polishing morphology is visible.

The pre-exposed samples feature surface blistering, with W having higher number and larger size of blisters as compared to W–Ta (see [9]). Exposure of these pre-exposed samples to low-flux plasma did not lead to any noticeable changes of their surfaces (Fig. 2; compare to [Fig. 3 in 9]).

3.2. Results of NRA depth profiling

Fig. 3 shows the results of NRA deuterium depth profiling performed on the samples exposed only to low-flux plasma in PlaQ.

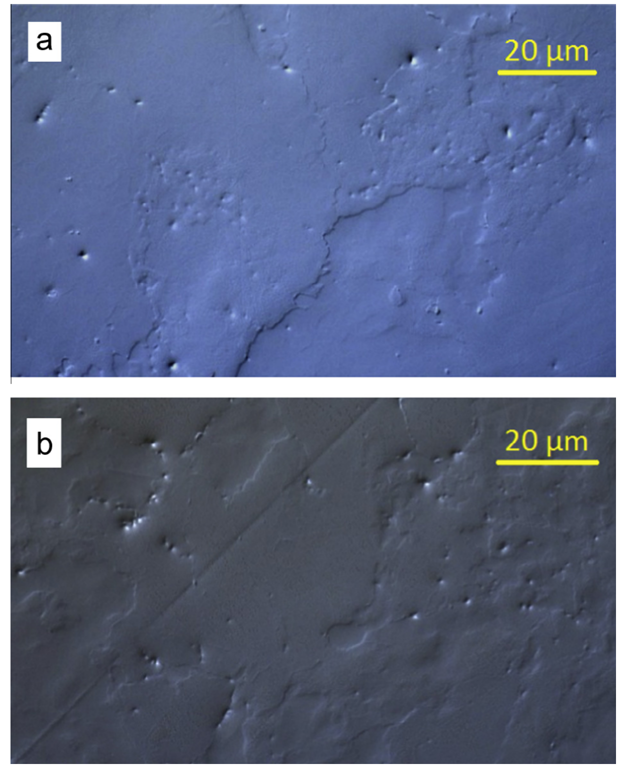

Fig. 1. Optical images of the surfaces of the reference samples: (a) W and (b) W–Ta.

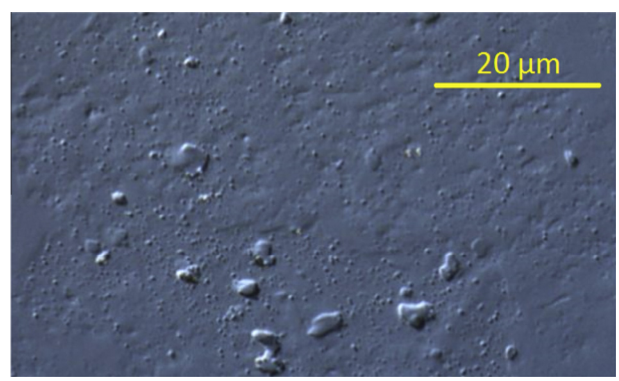

Fig. 2. Optical image of the surface of the pre-exposed W sample.

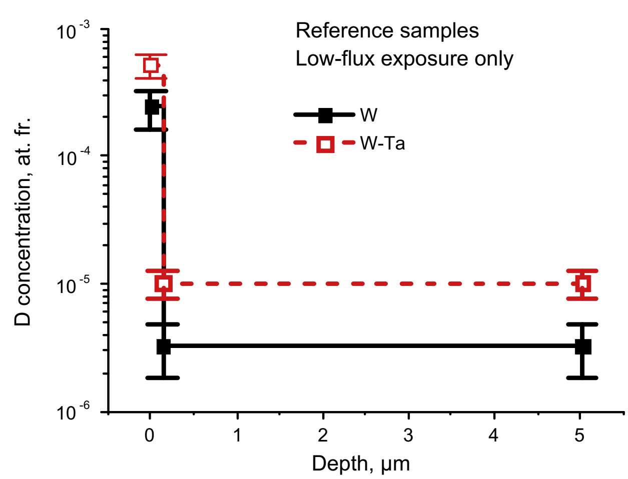

Fig. 3. D depth profiles in the reference W and W–Ta samples.