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Influence of tungsten microstructure and ion flux on deuterium plasma-induced surface modifications and deuterium retention

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ABSTRACT

The influence of surface temperature, particle flux density and material microstructure on the surface morphology and deuterium retention was studied by exposing tungsten targets (20 μ m and 40 μ m grain size) to deuterium plasma at the same particle fluence (10^{26} m⁻²) and incident ion energy (40 eV) to two different ion fluxes (low flux: 10^{22} m⁻² s⁻¹, high flux: 10^{24} m⁻² s⁻¹). The maximum of deuterium retention was observed at ~630 K for low flux density and at ~870 K for high flux density, as indicated from the thermal desorption spectroscopy data (TDS). Scanning electron microscopy observations revealed the presence of blisters with a diameter of up to 1 μ m which were formed at high flux density and high temperature (1170 K) contrasting with previously reported surface modification results at such exposure conditions.

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1. Introduction

Tungsten will be used as plasma-facing material for the ITER divertor due to its favorable thermal properties, low erosion and fuel retention [1]. Bombardment of tungsten by low energy ions of hydrogen isotopes, the fluxes of which will vary by several orders of magnitude at various locations along the divertor, can lead to surface modifications and influence the fuel accumulation in the material [2]. In addition to the particle flux [3], these changes are strongly affected by the surface temperature during the exposure and by the material microstructure. Investigations on the role of grain boundaries and intrinsic defects in the material such as dislocations and vacancies have confirmed that they can trap deuterium and act as centers for the bubble nucleation [4–6].

In our previous work, a comparative study of the influence of the particle flux on plasma-induced surface modifications of tungsten and deuterium retention was performed by exposing Large Grain Tungsten samples (LGW-grain size ~40 μ m) at temperatures of 530–870 K to two different ranges of deuterium ion fluxes (~5 × 10²³ m⁻² s⁻¹ and 9 × 10²¹ m⁻² s⁻¹), using the

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http://dx.doi.org/10.1016/j.jnucmat.2014.12.006 0022-3115/© 2014 Elsevier B.V. All rights reserved. Magnum - PSI and PSI - 2 linear plasma devices, respectively [3]. At high particle flux density and temperature, small blisters of several tens of nanometres were detected as well as an increase of the total deuterium retention compared to the lower flux density exposure was observed.

This contribution extends the previous work and addresses the impact of material microstructure on the correlation between the particle flux, surface modifications and deuterium retention in tungsten. A second set of experiments was performed by exposing polished tungsten samples with smaller grain size (SGW - grain size ~20 μ m) to deuterium plasma in Pilot-PSI and PSI-2 at the same incident ion energy (40 eV, below the physical sputtering threshold) and fluence (10^{26} m⁻³), varying the particle flux density (10^{22} – 10^{24} m⁻² s⁻¹) and exposure temperature (510–1170 K).

2. Experiments

Targets with a diameter of 14 mm and 4 mm thickness were cut from the one tungsten rod material so that the elongated grains were oriented perpendicular to the plasma exposed surface. They were mechanically polished to a mirror finish (~40 nm RMS), ultrasonically cleaned in alcohol and acetone to remove the polishing impurities and subsequently outgassed at ~ 10^{-6} mbar at 1273 K for one hour. Electron Backscattering Diffraction (EBSD)

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analysis was performed on the sample surface to investigate the microstructure, namely the grain size, shape and orientation. In Fig. 1, an EBSD map for two types of samples is given. The difference in grain size originates from the production history of the material and the rolling process (material supplied by Negele Hartmetall GmbH [7]). The grains were stretched to a maximum width of about 40 μ m and 20 μ m respectively, and a length of 200–300 μ m. The elongated grains were produced by forging (hammering) the tungsten rods after sintering. The forging of the SGW samples, done at a higher hammering force, produced smaller grains which were distributed more homogeneously.

The high particle flux density experiments were carried out in the Pilot - PSI [8] linear plasma device where a deuterium plasma beam was generated by a cascaded arc source at an axial magnetic field of 0.8–1.6 T. Plasma parameters (n_e, T_e) were measured by applying Thomson scattering method. To minimize the effect of the radial distribution of plasma parameters, a surface area of \sim 10 mm in diameter was exposed on the target which is close to the full width at half maximum (FWHM) of plasma T_e and n_e profiles. Surface temperature variation on the tungsten samples was achieved by varying the magnetic field strength and the physical contact of the sample with the water-cooled holder. A fast infrared camera (FLIR SC5500 MWBB InSB) was used to measure the temperature. Pilot - PSI operates in a pulsed mode with pulse lengths limited by the capabilities to cool the magnets; therefore the total fluence was accumulated in 2-3 consecutive discharges of 20-30 s per target.

Three tungsten samples were exposed in the linear plasma device PSI - 2 [9] which generates deuterium plasma with flux densities up to 10^{23} m⁻² s⁻¹ using a LaB₆ heated cathode and a Mo hollow anode. The plasma column has a hollow density and temperature profile with a diameter of 6 cm confined by a 0.1 T axial magnetic field. The plasma parameters were measured via a Langmuir probe. Targets were clamped on a water cooled holder and aligned properly with the maximum of the plasma profile. A

fast infrared camera was used also in this case to measure the temperature and in addition a thermocouple was mounted on the back of the sample. PSI - 2 is operated under steady-state conditions therefore the required particle fluence was accumulated in a single plasma exposure. In both experiments, deuterium plasma consisted mainly of singly ionized ions (D+) and the ion energy was calculated from the empirical formula $E_{\rm ion} = U_{\rm bias} - 2kT_e$ [10]. An external biasing of the target to -40 V (Pilot-PSI) and -60 V (PSI-2) was applied, which yield the desired ion energy at 40 eV. The influence of the plasma potential was neglected in the calculations.

Post mortem analysis was done using scanning electron microscopy (SEM), thermal desorption spectroscopy (TDS) and secondary ion mass spectroscopy analysis (SIMS) to investigate the surface modifications, total deuterium retention and deuterium depth dis-



Fig. 3. Blister diameter (Φ) is given as a function of exposure temperature and incident flux density for two sets of experiments, Large and Small Grain Tungsten samples (LGW [3], SGW).



Fig. 1. Microstructure analysis of the targets used in the experiments performed with the Electron Backscattering Diffraction method (EBSD) to illustrate the grain size, shape and orientation of the grains of Large Grain Tungsten (LGW ~40 μm) and Small Grain Tungsten (SGW ~20 μm).



Fig. 2. SEM images taken on the SGW samples after exposure to D plasma at PSI-2 (a)-(c) and Pilot-PSI (a₁)-(d₁) at a fluence of 10²⁶ m⁻² and incident ion energy of 40 eV.

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