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Incident energy dependence of blistering at tungsten irradiated by low energy high flux deuterium plasma beams

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Abstract

Polycrystalline tungsten samples have been irradiated at near room temperature by high flux (1×10^{22} D/m^2/s) deuterium plasma beams with incident ion energies ranging 7–98 eV/D. Surface blistering occurred at all energies as observed by means of scanning electron microscopy. At all energies, the blisters increased in their size and number with fluence within the corresponding low fluence ranges. The size increase tended to saturate at certain fluences within the experimental fluence ranges, which might be attributed to rupturing of blisters. The critical fluence for blistering Φ_{cr} was found to increase with decreasing the incident energy. At energies <20 eV/D, Φ_{cr} increased more rapidly. This energy dependence of Φ_{cr} may be explained by a proposed model dealing with the oxide barrier to deuterium uptake into and release from the bulk W. The presence of oxide layers of some monolayers on the surfaces was verified by means of X-ray photoelectron spectroscopy analysis and secondary ion mass spectroscopy before and after irradiation to different fluences.

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1. Introduction

A firm basis has been established for demonstration of the scientific and technological feasibility of controlled fusion through continuous efforts on magnetically confined fusion [1,2]. However, the choice of armor materials for the divertor and first wall is still a critical issue regarding a future reactor, including the International Thermonuclear Experimental Reactor (ITER), towards a long term steady state operation [3]. Tungsten and its alloys have been considered as candidate armor material for part of the ITER divertor and the whole first wall in the further DEMO reactors [4], due to their acceptable thermo-mechanical properties, possible advantage of very low or negligible erosion at low plasma temperatures, and a moderate uptake of tritium [5].

The ITER divertor may be operated in a scenario of semi-detached plasma condition in order to reduce particle and heat fluxes onto the divertor plate [1], and the incident particle energy will range from <1 eV till 100 eV, whereas the flux will be as high as 10^{22}/m^2/s on average. In the past, hydrogen–metal interactions were studied overwhelmingly in incident energy of keV or higher and flux of 10^{20}/m^2/s or lower by using ion beam accelerators, as reviewed by Myers et al. [6], and the radiation-induced defects in the bulk and at the surface played a key role in hydrogen reemission, retention, and permeation

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processes. Recently, investigations of irradiation of hydrogen isotope ions on tungsten under energies around 100 eV have been carried out by using both ion accelerators and plasma generators [7–11], showing clearly blistering at the W surfaces irradiated at temperatures from room temperature to ~900 K although the incident energies were too low to generate directly radiation defects in the bulk. So far, the circumstances under which blistering at W is favored, and especially how it may evolve and affect hydrogen retention in W and component lifetime has not yet been understood well.

In this paper, blistering behavior has been investigated at W irradiated using a newly established plasma generator [12], in which a deuterium plasma beam was produced and delivered onto sample. The incident energy was controlled from 100 eV down to some eV. The energy dependence of blistering has been studied systematically at near room temperature for the first time within this very low energy range, which should be beneficial to fundamental understanding on the mechanisms of blistering at W, and further hydrogen retention in and permeation through W.

2. Experimental

Tungsten plates (A.L.M.T. Corp.) were prepared by powder-metallurgy and hot-rolled reduction, and then heat treated at 1473 K for 30 min for stress relief as the standard final step before storing or cold working like grinding and polishing. The plates were subsequently cut and double-sided polished into samples of $10 \times 10 \times 2$ mm before shipping to our laboratory. The tungsten material has a purity of 99.99 wt% and the principal impurities in weight ppm are Mo and Fe ~10, C and O < 30. The as-shipped samples were irradiated without any more pretreatment prior to loading into chamber, except cleaning in an acetone ultrasonic bath.

The linear plasma generator [12] used in this study is capable of delivering plasma beams comparable to the practical edge plasma at ITER divertor. The generator consists of sections of vacuum chamber and pumping, cooling water, gas admittance, power supply, plasma generation, plasma delivery, sample holder, and plasma diagnosis. The achievable background pressure is lower than 5×10^{-6} Pa. The water-cooled sample holder is isolated from the grounded chamber wall so that the sample can be negatively biased to adjust the energy of ions impinging onto the sample. A single Langmuir probe is equipped about 3 cm upstream from the sample to obtain basic plasma parameters. The ion species in the plasma beam can be controlled via adjusting various parameters and measured using a differentially pumped quadrupole mass spectrometer (QMS) system as described in [13]. The species in the plasma beam generated in this work were determined to be predominantly D_2^+. And a fixed flux of 5×10^{21} $D_2^+/m^2/s$ (equivalent to 1×10^{22} $D/m^2/s$) was used in the experiments. The incident energy was varied via changing the bias voltage applied to the sample. The following bias voltages were employed, −200/−140/−80/−50/−30 V, resulting in the incident energies of 98/68/38/23/13 eV/D, respectively, taking simultaneously into account the plasma potential of ~−4 V measured by the Langmuir probe. An even lower energy of 7 eV/D was achieved by floating the sample, resulting in a floating potential of ~−18 V. The incident fluence was controlled by varying the irradiation time. The working pressure within the chamber was kept at ~1 Pa (D_2). The cooling water to sample holder was turned on to its full capacity of 2.6 l/min at 0.5 MPa and the temperature before irradiation was always about 293 K. The temperature rise of the sample being irradiated did not exceed 6 K during any irradiations, monitored by a type K thermocouple tightly pressing the rear of the sample.

The irradiated samples were observed at a tilt angle of 45° with a scanning electron microscope (SEM) in our laboratory (JEOL, JSM5410) to examine the occurrence and evolution of surface blistering. The judgment of blister appearance was limited by the resolution of the SEM employed that is not easy to distinguish blisters smaller than ~0.1 μm. Another SEM (Keyence, VE-7800) was used to observe surfaces of some of the samples and the cross-section of a sample prepared by means of focused ion beam (FIB) milling for better understanding the structure of blisters. X-ray photoelectron spectroscopy (XPS) in our laboratory (PERKIN ELMER PHI 1600) was employed to analyze the sample surfaces unirradiated and irradiated, with AlKα as the X-ray source, exposure area of 2×2 mm^2, and detected area of 0.8 mm^2. Depth profiles of the surface layers were obtained by means of secondary ion mass spectroscopy (SIMS) (*Φ* Physical Electronics, PHI ADEPT 1010) with the following beam parameters, Cs^+ beam energy of 5 keV, beam current of 10 nA, beam size of ~30 μm, incident angle of 60° from the surface normal, raster area of 400×400 μm^2. Negative secondary ions were detected from the central part of the raster area through an electronic aperture of 100×100 μm^2. The sputtering rate was estimated to be ~0.015 nm/s by means of a surface profiler (Veeco Corp., Dektak^{3}ST) that measures the height of step created by the sputtering at the surface.

3. Results and discussion

3.1. Surface morphology with varying the incident energy and fluence

At each incident energy, blisters were found at the irradiated surface of W samples after a corresponding