Journal of Nuclear Materials xxx (2014) xxx–xxx

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Dynamic measurement of the helium concentration of evolving tungsten nanostructures using Elastic Recoil Detection during plasma exposure

K.B. Woller *,[1], D.G. Whyte, G.M. Wright

MIT Plasma Science and Fusion Center, 175 Albany St., Cambridge, MA 02139, USA

ARTICLE INFO

Article history:
Available online xxxx

ABSTRACT

Helium (He) concentration depth profiles of evolving tungsten (W) nanostructures have been measured for the first time using in situ Elastic Recoil Detection (ERD) throughout plasma irradiation. Exposures resulting in fuzzy and non-fuzzy surfaces were analyzed in order to illuminate the role of He during the development of these surface morphologies. ERD was performed on samples with surface temperatures from T_s = 530–1100 K and irradiated by He flux densities of $\Gamma_{He} \sim 10^{20}$–10^{22} m^{-2} s^{-1}. He concentration profiles in samples that developed either non-fuzzy or fuzzy surfaces are uniformly shaped with concentrations of 1.5–7 at.%, which is presumed to be too low for pressure driven growth models. Therefore, surface morphology changes are not perpetuated by continuous bubble bursting deformation. Also, a threshold in He flux density above 10^{20} m^{-2} s^{-1} is suggested by using in situ ERD to monitor the depth profile evolution of the He-rich layer while changing the flux during exposure.

1. Introduction

Tungsten (W) nanostructures are of interest in many areas of materials research due to their drastic effects on the properties of the W surface. Arborescent nanostructures first occurred unexpectedly [1] and thus became a topic of alarm and intrigue because of their development under similar conditions as are expected in the ITER divertor and DEMO walls. Despite extensive study of the surface changes and performance, we still have limited understanding of how W nano-tendrils, or "fuzz," grow. If W fuzz is a desired surface modification from an engineering point-of-view, understanding the growth mechanisms might serve to optimize and accelerate specific W fuzz development, or, if not, suppress it. There are two basic premises for modeling W fuzz growth: pressure driven deformation [2] and surface diffusion around deformities [3–5]. If bubbles were to burst to deform the surface, pressure would have to build large enough to overcome the yield strength of W. However, many experiments performed on W fuzz after growth have shown that the He bubbles do not retain these required high pressures once the plasma exposure is ceased [6,7]. In this work, we measure the He concentration during fuzz development to show that the He concentration, even during growth, is less than what is thought necessary for bubble bursting models. In situ ERD has been developed and performed on samples grown under various conditions within the growth parameter space for W fuzz. First, an overview of the measurement technique and considerations for implementation near such an extreme environment are given. Then, the He concentrations as well as dynamic parameters are presented and discussed.

2. Materials and methods

2.1. Simultaneous ERD and RBS setup

He concentration measurements were carried out using the Dynamics of ION Implantation and Sputtering Of Surfaces (DIONISOS) experiment [8]. Fig. 1 shows a top–down schematic of the measurement set up. Essentially, DIONISOS has the capabilities of ordinary Ion Beam Analysis (IBA) techniques with the added ability to irradiate the sample at the same time.

A 7 MeV oxygen-16 (O) ion beam was directed at an angle of 75° normal to the surface of the samples. A 1 mm wide slit aperture in front of the detector defined the nominal scattering angle of 30° from the incident beam path. The solid angle subtended by the detector aperture was measured by performing ERD on a Kapton target while monitoring the beam current periodically with a faraday cup. The yield of the hydrogen (H) from the Kapton was simulated using SimNRA [9]. The solid angle determined in this manner was 3.22 ± 0.1 msr. The thickness and roughness of Diamond-like Carbon (DLC) foils acquired from MICROMATTER™ measured using Rutherford Backscattering Spectroscopy (RBS)

* Corresponding author at: MIT Plasma Science and Fusion Center, 175 Albany St., NW17-210, Cambridge, MA 02139, USA
E-mail address: kbwoller@mit.edu (K.B. Woller).
[1] Presenting author.

http://dx.doi.org/10.1016/j.jnucmat.2014.11.126
0022-3115/

were 5012×10^{15} atoms/cm^2 and 989×10^{15} atoms/cm^2, respectively, using SimNRA to fit the foil spectrum. To overcome the bane of scattered ion beam leakage through stopper foil pinholes, a stack of 9 of these DLC foils was placed after the aperture to stop the O scattered off of W from reaching the detector. An Ortec ULTRA charged particle detector was positioned behind the stopping foil stack to measure the final energy of the recoiled He. The recoiled energy scale of the detection set up was calibrated using separate proton and alpha ion beams of various known energies scattered off of a quartz target at a scattering angle of 45°. The overall depth resolution of the detection set up during irradiation was determined from this calibration and by an electronic pulser signal supplied to the detector preamplifier. The depth resolution was approximately 6 nm for He at the surface of W with a bulk density, or 40×10^{15} W atoms/cm^2.

The probing beam current, at approximately 1 μA, could not be measured directly during irradiation due to the several hundred mA of ion saturation current supplied to the sample by the plasma. Thus, the beam dose was calculated by measuring the RBS yield of O particles scattered from W at an angle of 90° from the incident beam path with a second detector simultaneous with the ERD detector. A single DLC foil carefully selected to minimize any pinholes was used in front of this detector to make it light-tight during plasma operation. The solid angle of the aperture in front of this detector was calibrated prior to irradiation experiments by measuring the beam current to a W sample that had secondary electrons suppressed. As before, the yield from this RBS signal was fit using SimNRA and the solid angle was measured to be $9.6 \pm 0.1 \times 10^{-6}$ sr.

2.2. In situ ERD and RBS considerations

Now, we present considerations taken to implement these in situ IBA techniques in such a dynamic environment. Particle trajectories deviate from the linear geometry of the measurement set up due to the magnetic field used for the plasma source and confinement. The magnetic field that the whole experiment is bathed in is very uniform and ranges from 230 to 675 G depending on the desired plasma mode. The trajectory deviations were calculated using the Lorentz force in a uniform magnetic field to be near 1° and were taken into account during the data analysis in SimNRA. This geometrical change results in a slight shift in the detected energy scale. However, the shift is much smaller than the detection resolution and does not result in any significant source of error.

The background neutral He gas in the chamber, with a pressure of 3–4 Pa, also interacts with the probing O beam creating an artifact at higher recoiled energies due to the lower recoil angle [8]. However, in this case, this background signal does not interfere with the He signal from the W surface because the geometry of the interaction requires He particles detected with the surface recoil energy or less to come from the sample. There is also a low energy background signal that is constant throughout the exposure. As Hydrogen (H) has long thermal desorption times even at these temperatures [10], the low energy background is likely due to multiple scattering of H and thus was subtracted away when necessary. These artifacts are discussed further below.

The ERD detector was in close proximity to an actively heated sample and a radiofrequency (RF) generated plasma column. If the detectors are not maintained at room temperature or below, then the resolution of the measurement is degraded. Also, the RF plasma was found to cause electromagnetic interference (EMI) in the ERD detector signal. To maintain low leakage current in the solid state detectors, the detectors were mounted in water-cooled housing. In the most extreme case, the leakage current in the ERD detector increased by a factor of 2 indicating that the detector temperature increase was only about 5 °C. The decrease in resolution was monitored by a pulser signal supplied to the detector preamplifier during irradiation measurements and the previously mentioned depth resolution was taken as the predominant case. The detectors were also insulated from the detector holders so that any EM waves generated by the plasma would be grounded to the vacuum vessel promptly without influencing the detector signal.

2.3. Sample growth conditions

The W used for these measurements were 25.4 mm diameter and 1.5 mm thick PLANSEE 99.95 wt% tungsten coins. They were mechanically polished to a mirror finish. Samples were then either actively heated with a Heat Wave Labs UHV heater or mounted on a water cooled target holder. The heater controller uses a type K thermocouple in contact with the alumina base of the heater stage just 1 mm from the sample for feedback. It has been observed that the emissivity of W surfaces increases with the development of fuzz [11]. Since the heater had feedback independent of the sample, the sample surface temperature is more easily regulated by the stage and not by the plasma heat flux. Thus, an increase in the emissivity due to nano-tendril development results in increased heat loss due to radiation and decreased plasma heat conduction to the sample, but this is compensated for by the stage, holding the temperature nearly constant. The samples without active feedback for temperature control did not develop fuzz. The surface temperatures of these samples were monitored using an

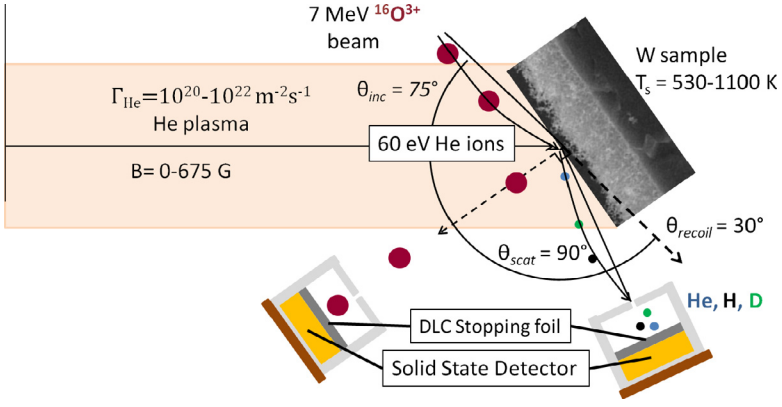

Fig. 1. Schematic looking down on the ion beam analysis set up within the DIONISOS irradiation chamber. The probing ion beam species was ^{16}O accelerated to 7 MeV by a tandem electrostatic accelerator. The sample was tilted 30° with respect to the magnetic field and 75° from the probing beam. A detector at 30° from the incident probing beam path collected recoiled species from the surface of the irradiated samples. A detector at a scattering angle of 90° collected scattered O from the surface as a measure of the beam dose.

Please cite this article in press as: K.B. Woller et al., J. Nucl. Mater. (2014), http://dx.doi.org/10.1016/j.jnucmat.2014.11.126