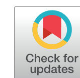
Journal of Nuclear Materials 502 (2018) 247–254

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Deuterium retention and release behaviours of tungsten and deuterium co-deposited layers



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ARTICLE INFO

Article history:
Received 14 August 2017
Received in revised form
6 February 2018
Accepted 6 February 2018
Available online 9 February 2018

Keywords:
Deuterium retention
Deuterium release
Tungsten co-deposited layers
Magnetron sputtering

ABSTRACT

Tungsten (W) layer deposited in argon and deuterium atmosphere by magnetron sputtering was used as a model system to study the deuterium (D) retention and release behavior in co-deposited W layer. After deposition several selected samples were exposed in deuterium plasma at 370 K with a flux of 4.0×10^{21} D/(m^2 s) up to a fluence of 1.1×10^{25} D/m^2. Structures of co-deposited W layers are investigated by field-emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD), and the corresponding D retention and release behaviors are studied as functions of deposition and exposure parameters using thermal desorption spectroscopy (TDS). Two main D release peaks were detected from TDS spectra located near 600 and 800 K in these W and D co-deposited layers, and total deuterium retention increased linearly as a function of W layer's thickness. After deuterium plasma exposure, the total D retention amount in W layer increases significantly and D release peak shifts to lower temperature. Clearly, despite the high density of defects expected in co-deposited W layers, the initial deuterium retention before exposure to the deuterium plasma is low even for the samples with a W&D layer. But due to the high densities of defects, during the deuterium plasma exposure the deuterium retention increases faster for co-deposited layer than for the bulk W sample.

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1. Introduction

Because retention of large quantities of tritium in first wall materials increases the fuel costs of a reactor and presents a safety concern, understanding of fuel retention properties of first wall materials is important for ITER and next-step fusion devices such as DEMO [1–5]. Due to its favourable physical properties, such as low erosion yield and high melting temperature, tungsten (W) will be used as plasma-facing material (PFM) in the ITER divertor and considered as the most promising PFM in future fusion devices [3,6,7]. As the first wall material in a future fusion device, W will be subjected to intense fluxes of incident particles and thermal loading [1,8], and it will inevitably be sputtered out from initial position and redeposited on nearby surfaces, then these redeposited W layers will partially cover the areas subjected to further intense fluxes of incident particles and thermal loading [2,3].

Since redeposited W layer can be considered as a defect-rich material, the microstructures and deuterium (D) retention behaviours of these films are definitely different compared with bulk W. Consequently, the deuterium retention in W layer should be taken into account. In this manuscript, we take the tungsten layers prepared in argon and deuterium atmosphere by magnetron sputtering as a model system to study the deuterium retention and release behaviors. The influences of deposition parameters, the layer storage time and deuterium plasma exposure on the deuterium retention and release behaviors were studied and compared with bulk W.

2. Experimental details

2.1. Sample preparation

W layers were deposited on single crystalline (100) silicon wafers using a direct current magnetron sputtering system. The Si substrates were cleaned twice ultrasonically in alcohol for 10 min before deposition, and then mounted on the sample holder. The

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https://doi.org/10.1016/j.jnucmat.2018.02.009
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system was pumped down to a base pressure of less than 7×10^{-4} Pa. The mixture gas of deuterium (D_2) and argon (Ar) was introduced via two mass flow controllers, respectively. The deposition was performed at a working pressure of 0.75 Pa. In this paper, the gas flow rate of Ar was kept as a constant of 20 sccm (standard-state cubic centimeter per minute), and the deuterium gas flow rate varied from 0 to 40 sccm, which corresponds to the increasing D_2 fraction in the mixture gas. Three groups of W layers were prepared by sputtering of a W target with 300 W dc power. In the first group, the gas flow rate of Ar was kept as a constant of 20 sccm, the D_2 flow rates were 0, 2, 4, 8, 10, 20 and 40 sccm respectively, and the deposition time was kept at 30 min for each deposition batch. The second group was deposited under the fixed fraction of D_2 (20 and 10 sccm flow rates for Ar and D_2 gases, respectively) with increasing deposition duration, consequently, W layers with different thicknesses varying from 0.5 to 10 μm were achieved. In the third deposition batch, a negative bias of 100 V was applied to the sample holder during W layer deposition in Ar and D_2 mixture atmosphere with 20 and 10 sccm flow rate, respectively.

Bulk W samples, cut from one batch of bulk material to the dimension of 10 mm × 10 mm × 1 mm, were mechanically polished with increasingly fine-grained SiC grinding papers and diamond suspension on the nap cloth to mirror-like finish. Then, to remove water soluble pollutants produced during the mechanical polishing process, samples were polished with deionized water on the nap cloth. Finally the bulk W samples were cleaned in an ultrasonic bath with acetone for 20 min at room temperature and cleaned in an ultrasonic bath with acetone [9,10]. Then bulk W samples were stress-relieved and degassed by heating at 1173 K in an ultra-high vacuum oven. Prior to this annealing treatment, the system was pumped down to a base pressure less than 1×10^{-5} Pa.

2.2. Sample characterization

The surface and cross-sectional microstructures of deposited W layers were investigated by field-emission scanning electron microscopy (FESEM) (JSM-6701F; JEOL, Japan). Cross-section images of the W co-deposited layers were obtained by breaking the silicon substrate after layer deposition. The thicknesses of these layers were measured from the cross-sectional images. Crystallographic phases and composition of samples were investigated by X-ray diffraction (XRD). XRD measurements were performed on a Rigaku RINT2400 X-ray diffractometer using Cu Kα radiation at $\lambda = 1.54056$ Å. Diffractograms were acquired from 30° to 90°.

2.3. Deuterium plasma exposure

The deuterium plasma exposure was performed in a linear experimental plasma system (LEPS) at Lanzhou Institute of Chemical Physics as shown in Fig. 1. A basic description of LEPS is given in short: a microwave (2.45 GHz) power is introduced into the vacuum chamber through a waveguide terminated by an alumina window. Then it further coupled with a remote electron cyclotron resonance in the magnetic field. To decouple plasma from substrate, the plasma is confined in a metallic mesh cage, so that particles can leave the cage in an axial direction through an aperture in the bottom plate with a diameter of 50 mm. At the edge position of the aperture two external magnetic coils were applied to confine the plasma beam. A confined plasma with a diameter about 50 mm impinges perpendicularly onto the substrates which are located 75 mm below the cage exit. More details for LEPS can be found in Ref. [9].

During deuterium plasma exposure the microwave power was set to 500 W with a constant D_2 gas pressure prior to plasma ignition of 0.3 Pa, and the maximum total ion flux and electron density were about 4.0×10^{21} D/(m^2 s) and 8.0×10^{16} m^{-3} detected by a Langmuir probe (ESPION) with a corresponding floating potential of 15 V. During exposure the substrate temperature was kept at 370 K controlled by an external open circuit thermostat using cooling water. After stopping plasma substrate temperature drops quickly within about three minutes and then slowly approaches room temperature.

Unfortunately, the mass distribution of impinging ions is currently unknown to LEPS due to experimental limitation. But because the external plasma operational parameters for LEPS are quite similar comparing to PlaQ (IPP, Garching) [11], we assume in our case the main ion is D_3^+. To calibrate the ions flux of D_2^+ and D^+ in impinging beam, tungsten film with a thickness of 800 nm deposited on silicon substrate was used as a reference, and −200 V, −300 V and −500 V bias were applied on tungsten film during deuterium plasma exposure, while the sputtered tungsten amount after exposure was measured by ion beam analysis. From the known sputtering yield (0.0002 at 300 eV/D and 0.0015 at 500 eV/D) of tungsten by deuterium, the absolute ion flux can be determined by measuring the removed tungsten amount during defined sputtering time. After 4 h, exposure at −200, −300 and −500 V bias the total quantity of removed tungsten determined by RBS are 15×10^{15}, 98×10^{15} and 600×10^{15} W/cm^2, respectively. Obviously, at −200 V bias the sputtering of tungsten should be attributed to impurities in plasma because the ion energy of deuterium at the bias is below the physical sputtering threshold of tungsten. In −300 and −500 V bias if only D^+ ion was assumed in two cases the corresponding ion flux would be 4.0×10^{20} and 4.1×10^{20} D/(m^2 s), that means the total ion flux for both D_2^+ and D^+ should be less than 10%. Accordingly in the following experiments, the calibrated D_3^+ flux of 3.6×10^{21} D/(m^2 s) was used.

Thermal desorption spectroscopy (TDS) is utilized to get information about D release behaviors and total deuterium retention in selected W layers after deposition or exposure. In all TDS measurements here the maximum temperature is set at 1173 K with an oven heating rate of 10 K/min and holding time at the maximum temperature is 10 min. Two independent experiments were carried out to calibrate temperature response of the samples to the linear oven temperature ramp, which was performed on a silicon or bulk W sample by a K-type thermocouple attached to target. The desorbed gases were measured by a quadrupole mass spectrometer (Pfeiffer QME220). Mass channels between 2 and 44 amu were selected to be recorded as functions of time using multiple ion detection mode (MID). The following 13 mass channels were recorded respectively, i.e. 2, 3, 4, 14, 16, 17, 18, 19, 20, 28, 32, 40 and 44 amu. Some of them, for example 32, 40, and 44 amu, are just measured to assess background emission and to check that no other species are released from the selected samples [6,7,12]. Release flux of molecules HD (mass 3) and D_2 (mass 4) were recorded to quantitatively analyze the deuterium release from the W layers during the temperature ramp [13]. For other deuterium-containing molecular compounds, such as HDO and D_2O, signals could not be quantified because of the absence of corresponding calibrated leaks [14]. However, in general, their contribution to the total retention is negligible [13,14].

In addition, quantification of mass 4 signal (corresponding to the release of D_2) was necessarily executed using a calibrated D_2 leak, and quantification of mass 3 signal (HD) was performed using the averaged value of signals of two calibrated leaks-H_2 and D_2 [15]. The calibration procedure was verified for D_2 and H_2 using a

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