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Compositional and morphological analysis of FeW films modified by sputtering and heating

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ABSTRACT

Surface compositional changes of iron-tungsten films by deuterium (D) ion bombardment were studied by means of medium energy ion scattering, elastic recoil detection analysis and Rutherford backscattering spectrometry. The energy of the bombarding ions was 200 eV/D and the fluence was varied from 10^{21} D/m^2 to 10^{24} D/m^2. A significant increase of the tungsten concentration within the 20 nm closest to the sample surface, caused by preferential sputtering of iron, was seen for the films exposed 10^{23} D/m^2 or more. In the sample exposed to the highest fluence, 10^{24} D/m^2, the concentration of tungsten was increased from an initial 1.7 at. % up to approximately 24 at. % averaged over the 5 nm closest to the surface. The analysis was complicated by the presence of oxygen on the sample surfaces. In order to study the thermal stability of the tungsten enriched layer, the sample initially exposed to 10^{23} D/m^2 at room temperature was heated to 400 °C in the measurement chamber for medium energy ion scattering and several spectra were recorded at intermediate temperatures. The obtained data showed that the layer was relatively stable below 200 °C whereas a drastic change in the film composition occurred between 200 °C and 250 °C due to interdiffusion of iron and silicon, the latter of which was the substrate material. The surface morphologies of the films were probed with atomic force microscopy showing that protrusions of 10–100 nm width appeared after deuterium bombardment at fluences higher than 10^{22} D/m^2.

1. Introduction

The reduced activation ferritic-martensitic (RAFM) steel Eurofer is being considered as a first wall material in the conceptual design of a European DEMO fusion power plant [1,2]. In present design suggestions and applicability studies, sandwich type plasma facing modules are often considered [3–5], with a pure tungsten armor on top of a Eurofer structure. However, fabrication difficulties and cost assessments suggest that at least parts of the first wall will consist of bare Eurofer [6,7]. To determine the feasibility of Eurofer as a plasma facing material it is relevant to investigate how its surface composition is modified when exposed to conditions similar to those at the fusion plasma edge. Preferential sputtering [8] by plasma ions, for example deuterium, leading to surface enrichment of tungsten is here of particular interest. An enriched tungsten layer at the plasma-facing surface could significantly reduce sputter erosion, thus protecting the underlying bulk material from additional modification by the plasma. For the effect to be relevant in a fusion machine, the enriched layer must not be lost by diffusion into the bulk material at the elevated temperatures experienced by the components of interest during operation. In the present study model films composed of an iron-tungsten alloy were investigated, providing a simpler system than real Eurofer. This permits to study the relevant effects and to draw conclusions about the onset and evolution of a tungsten-enriched layer in Eurofer exposed to deuterium bombardment. Time-of-flight medium energy ion scattering (ToF-MEIS) provides a finer depth resolution that what has been previously obtained with Rutherford backscattering spectrometry (RBS) [9]. Such improved resolution is necessary in order to resolve layer profiles with variations over a few tens of Å, which are expected from ref. [10].

2. Measurements and results

Five iron-tungsten model films containing 1.7 at. % tungsten, approximately 300 nm thick, were prepared by magnetron sput-

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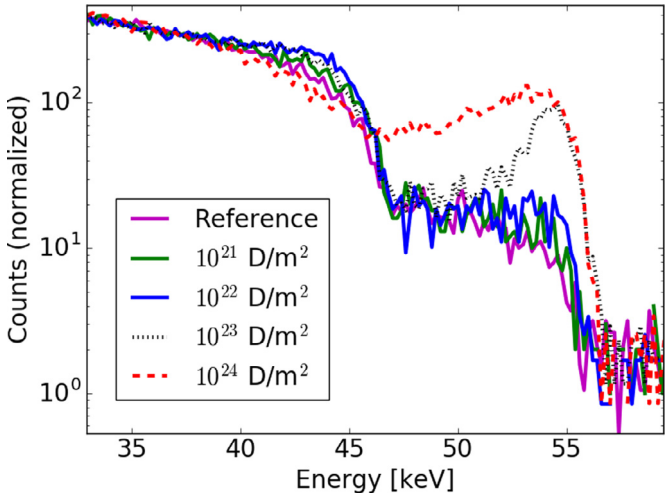

Fig. 1. Energy converted ToF-MEIS spectra from FeW model films exposed to the D_3^+ ion beam. All exposures were performed at room temperature for 200 eV/D and the figure legend gives the deuterium fluences. The surface energies for scattering from iron and tungsten are 45.6 keV and 55.2 keV respectively, averaged over all isotopes. These known points were used in the process of converting the initial uncalibrated time-of-flight data to obtain the energy scale on the x-axis.

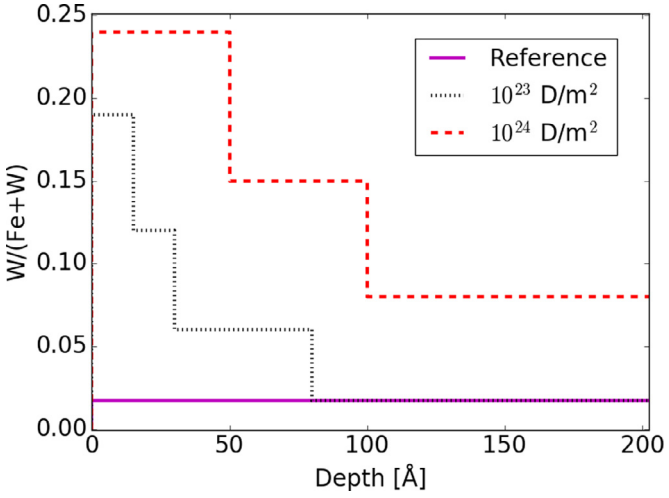

Fig. 2. Depth profiles of the tungsten concentration in the samples exposed to the highest deuterium ion fluences and the unexposed reference sample.

ter deposition on a silicon substrate. One sample was kept as a reference and the others were exposed to a mass separated D_3^+ ion beam at the high current ion source at IPP Garching, Germany, the basic design of which is described in ref. [11]. The exposure was performed at room temperature and the energy of the ions was 600 eV, meaning 200 eV per deuteron. The ion fluence was varied between the four exposed samples, from 10^{21} D/m^2 up to 10^{24} D/m^2. After exposure, the samples were first analyzed with ToF-MEIS at Uppsala University [12]. The technique has been previously tested for applicability to this type of analysis and has shown to be suitable [13]. A 60 keV $^4He^+$ beam was used and projectiles backscattered from the sample were detected at an angle of 155° ± 2° with respect to the forward beam direction. Fig. 1 displays energy converted ToF-MEIS spectra obtained from all five samples. The position in the spectrum where the tungsten signal intersects the iron signal corresponds to ions backscattered from tungsten at a depth of 20 nm in the sample. In the present ToF-MEIS configuration, we thus gain information from the top 20 nm layer without signal overlap. Normalization of the spectra was performed by dividing by the integral from 30 keV to 40 keV and multiplying by the same integral from the reference spectrum, placing the iron signal at the same level for all curves and thus providing a more suitable graph for direct comparison. This normalization was only used to allow for comparative figures. When analyzing atomic concentrations, the spectra were treated individually.

Simulations were performed with Biersack and Steinbauer's Monte Carlo program TRBS [14] to establish the concentration depth profiles that correspond to the ToF-MEIS spectra. Fig. 2 shows the atomic fraction of tungsten in the simulated layers that best fit the experimental data for the samples exposed to 10^{23} and 10^{24} D/m^2, i.e. the samples that were significantly modified by the deuterium bombardment. The constant tungsten concentration of 1.7 at. % for the unexposed sample is added as a solid line for comparison. The simulated layer thickness can be compared to the depth resolution of the measurement which is estimated by taking into account the stopping power of the probing ions in the sample matter. For 60 keV ^4He, the energy loss per unit length travelled in the sample is 281 eV/nm (from SRIM by Biersack and Ziegler). After scattering off one iron atom, reducing the energy of the projectile to 45.6 keV, the energy loss is 238 eV/nm. Since the scattered ions come out at an angle, the outgoing path length is increased accordingly. Thus, the change in energy loss of the projectile divided by the change in the depth at which a single scattering event takes place is 476 eV/nm, where the stopping power for the incoming ion has also been corrected by the kinematic factor. With a detector resolution on the order of 1 keV, this yields a surface depth resolution of approximately 2 nm. Note that the simulation that best fits the spectrum for the sample exposed to 10^{23} D/m^2 has two layers within the first 2 nm. With the above estimation of the depth resolution, we conclude that the ratio of the tungsten concentrations in these two layers and their thicknesses are not to be taken as exact values. The average amount of tungsten in the top 2 nm, however, is accurately measured. For the sample exposed to 10^{24} D/m^2 all simulated layers have a thickness larger than the surface depth resolution of the measurement. Materials other than tungsten and iron were ignored here, in particular oxygen which is present at the sample surfaces but not directly detectable with ToF-MEIS. Ignoring oxygen rules out the possibility for an accurate fit to the high energy edges in the spectra. This simplification has limited impact on the analysis in the present case, and the concentration of tungsten presented should simply be read as the ratio of tungsten to the total amount of iron and tungsten.

In order to measure the amount of oxygen at the sample surfaces and to probe the entire film thickness for any compositional changes caused by the deuterium bombardment, time-of-flight elastic recoil detection analysis (ToF-ERDA) was performed with a ^{127}I beam at 36 MeV. The angle between the beam and the sample surface was 23° and recoil ions were detected at 45° using the detection system described in ref. [15]. All samples were studied except the one previously exposed to 10^{23} D/m^2, which was reserved for the heating experiment described in the last section of this paper. Depth profiles of all elements above detectable concentration were generated from the raw data using the CONTES code by M. Janson. The tungsten enriched layer at the surface of each sample cannot be resolved with ToF-ERDA. As such the tungsten depth profile simply lies close to 1.7 at. % throughout the film, with a small unresolved bulge close to the surface, and has been omitted below. The depth profiles of the principal constituents of the samples (Fe, O and Si) were similar for all studied samples except the one exposed to 10^{24} D/m^2. The latter, along with the same profiles from the unexposed reference sample is displayed in Fig. 3.

The integrated amount of oxygen in the top 5e17 at/cm^2 is between 7.4e16 and 8.7e16 at/cm^2 in the samples exposed to less than 10^{23} D/m^2. In the sample exposed to 10^{24} D/m^2, it is 1.1e17 at/cm^2. The areal density of 1e15 at/cm^2 corresponds to a

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