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Sputtering of polished EUROFER97 steel: Surface structure modification and enrichment with tungsten and tantalum

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HIGHLIGHTS

- Surface enrichment with W and Ta in EUROFER97 exposed to 600 eV D_3^+ ion beam.
- D particle flux approximately 8×10^{18} $m^{-2}s^{-1}$; fluence between 10^{21} and 10^{24} D/m^2.
- Different exposure temperatures up to 1050 K gave similar enrichment profiles.
- Morphology changes included grain dependent erosion, cracking and recrystallization.

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ABSTRACT

Surface structure modification and enrichment with tungsten and tantalum were measured for polished EUROFER97 samples after exposure to a deuterium ion beam. Time-of-flight medium energy ion scattering and time-of-flight elastic recoil detection analysis were implemented for measuring atomic composition profiles. Atomic force microscopy and optical microscopy were used to investigate surface morphology. The deuterium particle fluence was varied between 10^{21} D/m^2 and 10^{24} D/m^2, projectile energy was 200 eV/D and exposure temperatures up to 1050 K were applied. The average fraction of tungsten plus tantalum to total metal content in the 2 nm closest to the sample surface was increased from an initial 0.0046 to 0.12 for the sample exposed to the highest fluence at room temperature. The enrichment was accompanied by an increase in surface roughness of one order of magnitude and grain dependent erosion of the material. The appearance of protrusions with heights up to approximately 40 nm after ion beam exposure at room temperature was observed on individual grains. Samples exposed to 10^{23} D/m^2 at temperatures of 900 K and 1050 K displayed recrystallization and cracking while changes to the total surface fraction of tungsten and tantalum were limited to less than a factor of two compared to the sample exposed to the same fluence at room temperature.

1. Introduction

EUROFER97 was developed in the European Union as a reduced activation ferritic martensitic (RAFM) steel to be used in blanket modules for the ITER experiment [1,2]. Presently it is also considered for a future DEMO reactor [3,4]. Consequently, characteristics of EUROFER97 such as tensile strength, thermal creep properties, electrical, thermal and magnetic properties and behavior under neutron and proton irradiation have been investigated in previous works [5–8]. The nominal composition of EUROFER97 is 9.5 at.% Cr, 0.5 at.% C, 0.48 at.% Mn, 0.33 at.% W, 0.22 at.% V and 0.043 at.% Ta, balanced by Fe (atomic fractions converted from the weight fractions as reported in Ref. [7], different sources give slightly different numbers). It has been shown that both EUROFER and the similar steel F82H display reduced erosion rates after exposure to light ion bombardment [9,10]. This property can be understood from the presence of heavy elements with low sputtering yields, i.e. tungsten

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and tantalum, which are enriched at the surface by preferential sputtering [11] and thereby lower the overall sputter rate. Quantification of the thickness of the enriched layer as well as the fraction of tungsten in it has previously been attempted with model samples in the form of iron-tungsten films deposited on carbon or silicon. Methods employed to this end include Rutherford backscattering spectrometry (RBS) [12] and, to obtain a higher surface depth resolution, time-of-flight medium energy ion scattering (ToF-MEIS) [13,14]. In the latter case, studies of the properties of the enriched layer at elevated temperatures have so far been complicated by diffusion of substrate material into the film [14]. Measurements of the enriched layer thickness and depth profiling of the tungsten and tantalum fraction in EUROFER97 after deuterium ion bombardment, with depth resolution of a few nanometers, have not been performed previously. The purpose of the present study is to provide such measurements and correlate their results to changes in surface morphology. The information can help improve the understanding of the material's response to plasma erosion. It can thereby help to guide design decisions on using RAFM steels in plasma exposed regions of future fusion machines, as suggested for example in Ref. [15]. We have employed ToF-MEIS and time-of-flight elastic recoil detection analysis (ToF-ERDA) to analyze the enriched layer of tungsten and tantalum produced in polished samples of EUROFER97 after exposure to a 600 eV D_3^+ ion beam. Deuterium particle fluences from 10^{21} D/m^2 to 10^{24} D/m^2 were applied at room temperature and for the fluence of 10^{23} D/m^2, several samples were exposed at different temperatures up to a maximum of 1050 K. Surface structure modification has been observed through optical microscopy and atomic force microscopy (AFM).

2. Experimental

2.1. Sample preparation and exposure

12 samples, 8×10 mm^2 and 1 mm thick were machined from a slab of EUROFER97 which was manufactured at Forschungszentrum Karlsruhe GmbH, Germany and delivered via the European Union's undertaking Fusion for Energy. Polishing was performed with 600 and 1200 grit sandpaper followed by diamond lapping films on a rotating disc. The grain sizes for the lapping films were successively 30 μm, 15 μm, 9 μm, 6 μm and 3 μm. In the next step, an oil based 1.5 μm diamond lapping paste was applied to a microfiber cloth which was used to polish the samples by hand. Finally, a 50 nm water based aluminum oxide suspension from Tedpella was used, also on a microfiber cloth. The samples were cleaned with water and ethanol after every polishing step. Following the final polishing step, washing was performed by first wiping the samples with a microfiber cloth soaked in ethanol and then submerging them in acetone for 20 min. Once removed from the acetone, the samples were wiped dry with non-abrasive optical lens tissue and baked at approximately 10^{-2} mbar and 130 °C for 48 h.

The exposure of the samples to a deuterium ion beam was performed with the recently assembled Second Ion Experiment for Sputtering and Thermal desorption Analysis (SIESTA) at IPP Garching, Germany. A discharge was set up in a deuterium plasma source which was placed at 3.6 kV for samples numbered 1 and 2, and 4.7 kV for the remaining samples. The increase of the source voltage was performed in order to increase the beam current and thereby reduce the exposure time for the samples. D_3^+ ions were selected by means of a bending magnet and the resulting beam was impinging at normal incidence on the sample which was set at a potential 600 V lower than the source. Consequently the energy of the D_3^+ ions when hitting the sample was 600 eV, or 200 eV/D. The total ion fluence was measured via the time integrated current received by the sample and secondary electron emission was taken into account using biased Faraday shields surrounding the target platform. Beam currents between 9 μA and 13 μA onto the sample were reached, corresponding to a deuterium particle flux between 6.7×10^{18} D/m^{2}s and 9.7×10^{18} D/m^{2}s over a beam footprint area of 0.25 cm^2. Heating via electron impact was applied using a tungsten filament placed behind the sample and temperature was measured both with a thermocouple pressed onto the back of the sample and with an infrared pyrometer monitoring the irradiated surface. The temperature was controlled within ±70 K. Table 1 gives the exposure conditions for the samples.

2.2. Ion beam analysis

After exposure, the samples were sealed in an argon atmosphere and shipped to Uppsala University where ToF-MEIS measurements were performed to quantify the surface enrichment with tungsten and tantalum, and obtain depth-profiles of the enriched layer. A description of the ToF-MEIS setup can be found in Ref. [16]. The primary beam of 60 keV $^4He^+$ was impinging on the samples parallel to the surface normal and backscattered ions were detected at a scattering angle of 155°. The beam current was less than 10 pA and the measurement time was shorter than 1000 s. With a beam spot area of approximately 1 mm^2, the corresponding maximum $^4He^+$ ion fluence is 6.2×10^{16}/m^2, i.e. little enough not to have had any measurable effect on the composition of the samples. For some samples, several measurements were taken, varying the position of the ToF-MEIS beam spot within the SIESTA beam footprint. By this approach it was verified that the surface enrichment is homogeneous within the beam footprint for independent measurements averaged over the size of the ToF-MEIS beam spot (~1 mm^2).

Measurements with ToF-ERDA were carried out primarily to quantify the amount of oxygen on the sample surfaces. A 36 MeV beam of $^{127}I^{8+}$ was employed at 67° incidence with respect to the sample normal and recoils were detected at 45° from the forward beam direction using the detection system described in Ref. [17].

2.3. Microscopy

To investigate surface morphology changes resulting from the ion beam exposure, the samples were photographed through an optical microscope. The appearance of the surface was similar everywhere within the SIESTA beam footprint and images of 150×150 μm^2 were recorded at points close to the center of the footprint.

AFM scans were performed on all samples both before and after

Table 1
Exposure parameters for polished EUROFER97 samples at the SIESTA setup. Samples 1–5 constitute a fluence series at room temperature while samples 3, 9, 11 and 12 constitute a temperature series. Samples 6 and 8 were excluded from additional evaluation due to a misalignment of sample 6 in the setup causing the beam to partially miss the surface, and a carbon rich contamination layer on sample 8 that interfered with the intended sputtering experiment.

Sample number	Fluence [D/m^2]	Temperature [K]
1	10^{21}	300
2	10^{22}	300
3	10^{23}	300
4	10^{24}	300
5	5×10^{23}	300
7	0	300
9	10^{23}	750
10	0	300
11	10^{23}	900
12	10^{23}	1050