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Erosion yield and W surface enrichment of Fe-W model system exposed to low flux deuterium plasma in the linear device GyM

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Iron-tungsten (Fe-W) mixed films were exposed to the low flux deuterium plasma of GyM in order to study the behavior of the sputtering yield with the ion fluence and temperature of the samples. From literature, it is known that an increase of the former lowers the Fe-W layers' sputtering yield as a consequence of the preferential sputtering of Fe leading to an enrichment in W of the outermost layers. An opposite trend was instead found for the latter probably due to the inter-diffusion of Fe and W (effective from 200 °C) resulting in the suppression of the W enrichment. Moreover, from 500 °C, also W segregation to the surface occurs. What is missing from literature is a systematic investigation of the role of temperature on W enrichment. In this work, dedicated experiments in GyM were carried out to fill this gap. After exposure, W enrichment was evaluated by Rutherford Backscattering Spectrometry (RBS) and inferred from measuring the eroded thickness of the samples using RBS and profilometer. Concerning the Fe-W sputtering yield as a function of fluence, it decreases by a factor of ~3 between the lowest ($3.0 \times 10^{22} D^{+}m^{-2}$) and the highest fluence ($9.0 \times 10^{23} D^{+}m^{-2}$) values considered. The other main result is that, at the lowest fluence, the exposure at room temperature leads to an erosion of the Fe-W samples more pronounced than that associated to the exposure at 500 °C.

1. Introduction

In future magnetically confined thermonuclear fusion reactors like DEMO, the erosion of the components in the recessed areas of the first wall due to the impact of energetic particles, mainly charge-exchange neutrals (with the largest fluxes at energies in the order of 200 eV and below [1]), would be a serious issue. Bare Reduced Activation Ferritic Martensitic (RAFM) steels, such as EUROFER, are a valuable alternative to tungsten (W) coatings, or armor [2], both from the economical and technological point of view. RAFM steels are iron (Fe) based alloys containing mid-Z steel elements (chromium, etc.) and small amounts of high-Z elements such as W (~0.45% for EUROFER) [3]. Since the sputtering yield, Y, of Fe and other mid-Z elements is higher than that of W, one expects that during plasma erosion, the former will be sputtered much faster, leading to a W enrichment of the sample's surface. For a controlled investigation of W enrichment dynamics, Fe-W mixed layers as a model system of RAFM steels have been recently exposed to deuterium (D) ions and plasmas of linear machines [1,3–6]. These experiments clearly show the presence of a W enrichment at the surface (~nm) of the Fe-W samples whose W content increases with D ion fluence. Moreover, a role of the temperature has been demonstrated. First, from 200 °C the inter-diffusion of Fe and W becomes effective [1]. Second, at 500 °C, W segregation to the surface also occurs [6]. The two have opposite effects on Fe-W sputtering yield since the former clearly counteracts the W enrichment instead the latter enhances the W concentration in the outermost layers of the samples.

As far as we know, the literature that deals with the reduction of the sputtering yield of the Fe-W model system increasing the deuterium fluence is almost complete [1,3–6]. There is however a lack of data relative to the behavior of the Fe-W films with temperature [6]. The investigation was indeed carried out only till 300 °C for a single couple of fluence and ions energy. Moreover, at the fluence value considered in those experiments (1.0×10^{24} $D^{+}m^{-2}$)

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the preferential sputtering of Fe was also important. Therefore, the impacts of the fluence and temperature on the Fe-W erosion yield were thus not discernible.

In this work we present the results of the exposures of the Fe-W layers to the low flux ($\sim 10^{20}$ $D^{+}m^{-2}s^{-1}$) deuterium plasma of GyM [7,8]. On one hand, some Fe-W films were bombarded at a given temperature T and different fluences, from $3.0 \times 10^{22} D^{+}m^{-2}$ to $9.0 \times 10^{23} D^{+}m^{-2}$, to confirm the results found in the literature [1,3,6]. On the other hand, a series of exposures were done at the lowest fluence (thus minimizing the impact of Fe selective sputtering), from Room Temperature (RT) to 500 °C, as an attempt to study the possible thermal effects on the erosion of the Fe-W films.

On completion of these two studies, the experiments were carried out at two different D ions energy, the first (~100 eV) below and the second (~300 eV) above the W sputtering threshold (~200 eV).

Samples were properly characterized before and after the exposures in order to evaluate their erosion and W enrichment. Possible contaminants deposition on samples' surface during exposures, which could introduce spurious effects on the erosion yield estimate, was also evaluated and the analysis is here described.

2. Materials and method

The Fe-W layers were produced by magnetron sputtering at IPP-Garching with W concentration of 1.5 at.%. Silicon wafers were used as a substrate to ease the characterization. The details of the deposition conditions are described elsewhere [3]. The "as deposited" Fe-W films were extensively characterized by the other EUROfusion partners. An oxygen concentration of 1–2 at.% was found. However, the ageing of the samples dramatically increased this value to 20 at.% (see Section 3.3). Atomic Force Microscope (AFM) measurements showed that the samples' roughness is of the order of few nanometers. As expected, the density of the Fe-W films is slightly lower than that of the bulk material (90% [9]) and the actual value was considered for a proper evaluation of the erosion yield from profilometry.

In the following, the exposure conditions and the techniques used to measure the sputtering yield of the samples and their W-surface enrichment are described.

2.1. Exposure conditions

The Fe-W films were exposed to a deuterium plasma within the GyM linear device at IFP-CNR, Milan [7,8,10,11], which is capable of operating at steady state. The intensity of the magnetic field at the samples' location is ~80 mT and the ion temperature T_i is of the order of 0.1 eV. Deuterium plasma properties at the same radial position of the samples were monitored by means of a Langmuir probe (a steel cylinder: 1.5 mm Ø and 10 mm length, insulated with an alumina tube; its axis was perpendicular to the magnetic field lines). Since the deuterium ions' gyroradius was well below the length of the Langmuir probe, its projected area as viewed along the magnetic field (i.e. twice the product between diameter and length) was used to extract the plasma parameters from the Langmuir probe characteristic. At the working pressure of deuterium of 5.7×10^{-4} mbar, typical electron temperature and density of $T_e \sim 4$ eV and $n_e \sim 3.0 \times 10^{16}$ m^{-3}, plasma potential $V_p \sim 17$ V and a ion flux of 3.0×10^{20}ions m^{-2}s^{-1} were measured. Two designed stainless steel sample-holder manipulators were used whether the exposure was at RT or at elevated temperature (200 °C, 300 °C, 340 °C and 500 °C). The sample holder for exposures at RT requires a water-cooling system behind the samples, which allows part removal of the heating power coming from the plasma. The sample holder for exposures at the higher temperatures has a lamp, which heats the rear side of the samples. The temperature of the samples

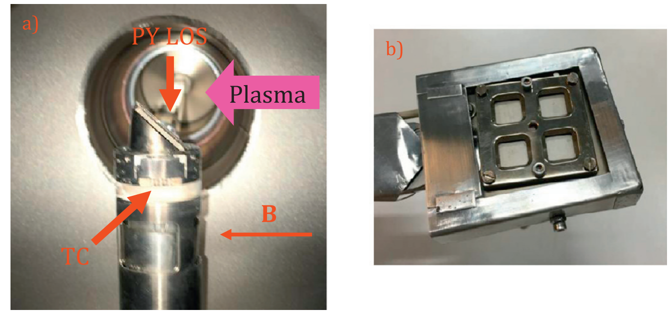

Fig. 1. Scheme of the high temperature exposures at 45° with respect to the magnetic field **B** (a). The position of the thermocouple (TC) and the line-of-sight of the pyrometer (PY LOS) are also shown. Photograph of the cooled sample holder for exposures at RT (b).

was measured by a thermocouple pressed behind the samples and by a pyrometer (Fig. 1a). Since the line of sight of the pyrometer was perpendicular to the GyM axis, exposures were performed at an angle of 45° between the sample holder normal and the magnetic field direction. Even though this angle is far away from 90°, the sputtering of D ions is still approximately normal to the sample's surface due to the low value of the magnetic field [12]. This statement was also supported by the fact that AFM topographic pictures of the Fe-W exposed samples (not shown here for brevity) do not exhibit any particular feature along 45°.

Both sample holders can be biased. Since the deuterium sputtering of W is expected to be negligible below 200 eV [3], the bias voltages chosen for these experiments were −100 V and −300 V (we consider their absolute value in the following), in order to investigate the effect of the impact energies of deuterium species below and above the sputtering threshold of W on the dynamics of the W enrichment of the Fe-W films.

The two sample holders accommodate up to four samples each (Fig. 1b). Small molybdenum (Mo) foils were used to mask half of each sample in order to allow mechanical profilometer measurements (see Section 2.2). In the case of the exposures at the higher temperatures, the masked side of the sample also permits the evaluation of the W thermal segregation toward the surface of the Fe-W films. Together with a Fe-W sample, also pure iron and tungsten magnetron sputtered films from IPP-Garching were exposed as reference.

Since one of the objectives of the present work was to evaluate the sputtering yield of the Fe-W layers which clearly depends on the energy of the incoming deuterium ions, an estimate of the deuterium species mix of the plasma was required. For a certain sample bias V_b (-100 V and -300 V, here) in fact, only D^{+} ions impinge on the samples at an energy $E_K = eV_b - eV_p$ (since T_i~0.1 eV, the ions thermal energy is negligible). D_2^{+} and D_3^{+} ions break up at the sample's surface and each deuteron takes 1/2 and 1/3 of E_K, respectively. To this regard it is worth to notice that a preliminary survey about equipping GyM with a commercial plasma mass spectrometer was not encouraging because of the technical difficulties and cost associated. The configuration of the magnetic field of GyM is such that it would require to design and develop a proper solution which is out of the scope of the present work [13,14]. For this reason, we made an attempt of estimating the fraction of D^{+} ions starting from the erosion data relative to the reference magnetron sputtered iron samples which were exposed in GyM together with the Fe-W films. First of all, the sputtering yield values of these Fe samples as experimentally determined by Sugiyama et al. [15] were used in the analysis (they bombarded the same Fe films with a D_3^{+} ion beam in HSQ). We considered one of the deuterium plasma exposures at -100 V bias voltage (total fluence 2.3×10^{24} ions m^{-2}, sample temperature 200 °C). In these