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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

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

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Abstract

Iron-tungsten (Fe-W) model system for EUROFER steel was exposed to the low flux deuterium plasma of GyM up to a fluence of $1.8E24 \; D^+\text{m}^{-2}$ in order to study the sputtering yield as a function of the ion fluence and sample temperature. From literature, it is known that an increase of these two parameters lowers the Fe-W sputtering yield. Due to the high sputtering threshold of tungsten in comparison with that of iron, for higher ion fluences, the concentration of W at the surface of Fe-W layers increases (the so-called W-enrichment) due to the preferential sputtering of Fe. At the same time, high temperature exposures promote W diffusion towards the sample surface leading to the W-rich surface layer. In this work, ad-hoc experiments were performed to evaluate separately the dependence of ion fluence and sample temperature on W-enrichment. After exposure, W enrichment was evaluated directly by Rutherford Backscattering (RBS) and indirectly by measuring the eroded sample thickness using RBS and profilometer. The Fe-W sputtering yield at low fluence is $2.9 \times 10^{-2} \; atoms/ D^+ \; ion$ while at high fluence it reduces to $1.1 \times 10^{-2} \; atoms/D^+ \; ion$. The main result in the range of the parameters investigated here is that the effect of ion fluence on the reduction of Fe-W sputtering yield is stronger than that of temperature, which can be neglected below 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 of the order of 200 eV [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 significantly higher than that of W, one expects that during plasma erosion, the former will be sputtered much faster, leaving behind a W-rich layer which would be hardly eroded from the sample surface. For controlled investigations 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 [1,3-5]. These experiments

clearly show the presence of a W-rich layer at the surface (few nm) of Fe-W samples whose W content increases with D ion fluence. Moreover, a role of the temperature has been demonstrated: from 500 °C the diffusion of W into Fe becomes effective, influencing the behavior of the sputtering yield [1].

In order to distinguish the effects on W enrichment of the selective Fe sputtering and of the W thermal diffusion, we performed appropriate exposures of Fe-W layers to the low flux (10^{20} D^+m^{-2}s^{-1}) deuterium plasma of GyM [6,7]. On one hand, some Fe-W films were exposed to high fluences at temperatures T much lower than 500 °C to study only the preferential Fe sputtering due to D ions bombardment. On the other hand, low fluences exposures at 500 °C were performed to bring up the effects of W thermal diffusion on the erosion of Fe-W films.

The full set of exposure parameters was: fluences of $8.0 \times 10^{22} \div 1.8 \times 10^{24} D^+$ m^{-2}, sample biases of -100 and -300 V (we refer to their absolute value in the following) and temperatures of RT $\div$ 500 °C . 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 characterizations. The details of the deposition conditions are described elsewhere [3].

In the following, the exposure conditions and the instrumentation 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 [6-9], which is capable of operating at steady state. Two designed stainless steel sample-holder manipulators were used whether the exposure was at RT or at the elevated temperatures (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 was measured by a thermocouple pressed behind the samples and by a pyrometer (figure 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 machine axis. Even though this angle is far away from 90°, sputtering of D ions is approximately normal to the sample surface due to the action of the magnetic pre-sheath which progressively reorients the incoming ion trajectories normally to the sample's surface [10].

Both sample holders can be biased. Since the deuterium sputtering of W is expected to be negligibly small below 200 eV [3], the bias voltages chosen for

these experiments were 100 V and 300 V, in order to investigate the effect of impact energies of deuterium species below and above the sputtering threshold of W on the dynamics of Fe-W surface W enrichment.

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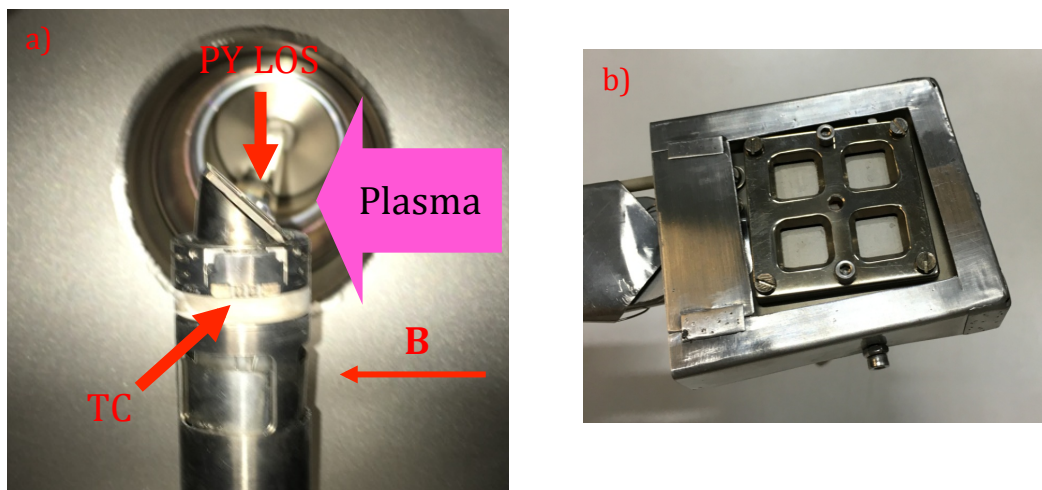

Fig.1: Scheme of the high temperature exposures at 45° with respect to the magnetic field **B** (a). The position of the thermocouple (TC) and the line-of-sight of the pyrometer (PY LOS) are also shown. Photograph of the cooled sample holder for exposures at RT (b).

Deuterium plasma properties at GyM axis were monitored by means of a Langmuir probe. At the working deuterium pressure of 5.0×10^{-5} mbar, typical ion fluxes in the range $4.0-5.0\times10^{20}\,ions\,m^{-2}s^{-1}$ were measured (electron density $n_e\sim 9.0\times10^{16}$ m^{-3} , electron temperature $T_e\sim 4-5$ eV and plasma potential $V_p\sim 14$ V). Since one of the objectives of the present work was to evaluate the sputtering yield of 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 $E_K = eV_b - eV_p$. 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. For this reason, a polished silicon (100) wafer was placed in the fourth available position of the sample holder during one of the deuterium plasma exposures at 100 V bias voltage (total fluence 5×10^{24} $ions$ m^{-2}, sample temperature 200°C). In these conditions $E_K \sim 86$ eV. Deuterons formed when D_2^+ and D_3^+ impinge on silicon have an energy close to the theoretical sputtering threshold of bulk silicon $E_{th} = 26.48$ eV [11] and their contribution to the sputtering can be neglected. Measuring the eroded thickness of the sample (s) after the exposure with profilometer and using the formula

$$F = s\rho N_A / YA \qquad (1)$$

where N_A is the Avogadro's number, ρ and A the silicon density and atomic number, the D^+ fluence F can be estimated using the theoretical sputtering yield Y_{Si} of Si at 86 eV [11]. This reasoning is clearly valid assuming negligible re-deposition and deposition of contaminants during the experiment (see subsection 3.4). The result of the analysis gives a fraction of D^+ of approximately 20% in GyM deuterium plasma at 5.0×10^{-5} mbar. This value is in agreement with literature [12]: at this working pressure the production of D_2^+ ions from D_2 molecules ionization is more effective than that of D^+ ions from the ionization of D atoms and the dissociation of D_2 molecules and also is more favorable than the formation of D_3^+ from $D_2 + D_2^+$ reactions.

In the present work, sample bias voltage of 100 V and 300 V results in D+ energy of 86 and 286 eV, respectively, due to the plasma potential of 14 V.

2.2 Post-exposure analyses

Erosion of the samples was evaluated using a KLA Tenchor mechanical profilometer which measures the eroded step height between the exposed and masked area of the sample. For each layer, twenty independent chords were taken at several positions and the mean value and standard deviation were finally calculated.

After the exposure, W enrichment of each Fe-W layer was evaluated directly by Rutherford Backscattering Spectroscopy (RBS) and indirectly by measuring the erosion of the samples with RBS and profilometer. RBS analyses were performed at the Ruđer Bošković Institute in Zagreb using 4 MeV Li[3+] ions with normal incidence to the sample surface. The RBS detector was placed at 165° angle toward the beam direction.

In order to evaluate possible deposition of contaminants mainly coming from the sample holder, the exposed surface of the samples was analyzed by Secondary Ions Mass Spectrometry (SIMS) and Time-of-Flight Elastic Recoil Detection Analysis (ToF-ERDA). SIMS was equipped with a HIDEN RF quadrupole spectrometer (EQS-300), a primary beam delivered by a SPECS 12/38 impact ionization O2 ion gun, and a Wien filter [13]. For surface wide scan mass spectrum, a 5 keV O[2+] beam was scanned over a 1500 × 1500 μm[2] surface area at 60° incidence angle and sputtered secondary ions were collected on a 500 × 500 μm[2] gating area. ToF-ERDA used 23 MeV [127]I[6+] beam.

3. Results and discussion

A survey of the results for exposures of Fe-W layers to the deuterium plasma of GyM is described in this section. Samples were exposed at different fluences, $10^{22} - 10^{24} D^+$ m^{-2}, bias voltages, 100 V ($E_K \sim 86$ eV) and 300 V ($E_K \sim 286$ eV), and temperatures, RT, 200 °C, 340 °C and 500 °C. Erosion yield Y of the samples was evaluated using Eq.1 from profilometer and RBS measurements (which gave the eroded thickness s). The density of Fe-W layers was taken to be 90% of Fe bulk density [14]. As F we considered the fluence of only D+ ions (20% of the

total value), even though at 300 V, the contribution of D_2^+ and D_3^+ ions to the sputtering of the Fe-W layers (iron $E_{th} = 44.32$ eV [11]) could be not negligible. As it is shown in the next subsection, this rough assumption does not prevent to have a good agreement with the sputtering yield of Fe-W ($Y_{Fe\text{-}W}$) as a function of fluence for different ion energies, obtained from Fe-W layers irradiation with a mass separated deuterium ion beam (D_3^+ beam of HSQ setup at IPP Garching) [3].

3.1 Fluence Dependence

The results of the behavior of the erosion yield $Y_{Fe\text{-}W}$ as a function of fluence ($8.0E22 - 1.8E24 \ D^+/m^2$) are shown in figure 2. In order to exclude possible thermal effects on $Y_{Fe\text{-}W}$, exposures were performed at the temperatures well below 500°C, from which diffusion of W into Fe would become significant [1]. Figures 2a and 2b refer to the experiments with a bias voltage of 100 V (at 200°C and 300 °C) and 300 V (at 340°C), respectively. The comparison between $Y_{Fe\text{-}W}$ estimated from profilometer and RBS measurements highlights a good qualitative and quantitative agreement between the two techniques. $Y_{Fe\text{-}W}$ decreases with the fluence for both the ion energies due to the progressive selective sputtering of iron and the consequent formation of a W-rich layer at the surface of the samples, as demonstrated by RBS spectra (see subsection 3.3). As expected, the values of $Y_{Fe\text{-}W}$ associated to the exposures at 300 V are higher than those of exposures at 100 eV because of the increase of iron and tungsten erosion yields with the energy of the deuterons impinging on the sample's surface.
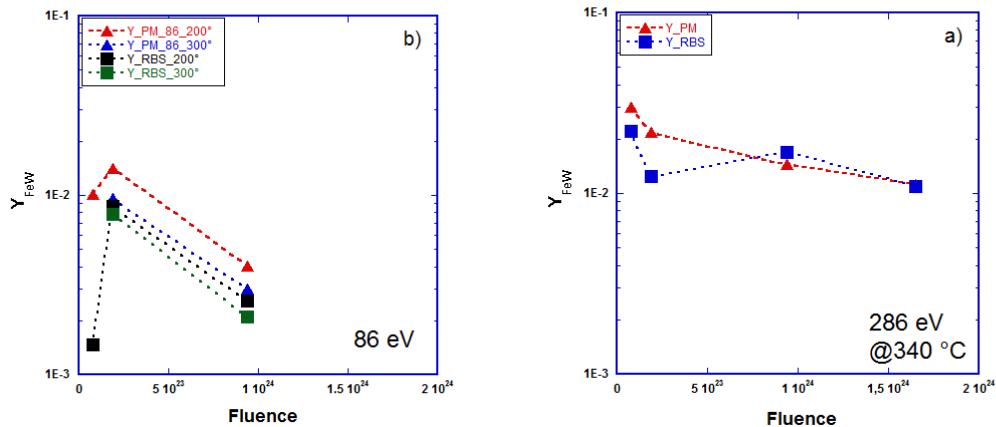

Fig.2: Fluence dependence of the sputtering yield of Fe-W layers, $Y_{Fe\text{-}W}$, estimated from profilometer (triangles) and RBS measurements (squares), at D$^+$ energy of 86 eV and 200°C and 300 °C (a) and 286 eV at 340°C (b).

The main qualitative difference between figures 2a and 2b is the slope of $Y_{Fe\text{-}W}$ within the range $2.0E23 - 8.0E23 \ D^+/m^2$. A reduction by a factor of 4.5 and 2.5 was obtained for exposures at 100 V and 300 V, respectively. This can be ascribed to the very low erosion yield of W for deuterium ions with the energy below 200 eV, which makes the selective sputtering of iron more effective at 86 eV. From figure 2a, the slight decrease of $Y_{Fe\text{-}W}$ with increasing temperature

(from 200 °C to 300 °C) was probably due to the growing importance of the thermal effects [1]. Since the highest fluence considered here is far away from $1.0E25\ D^+/m^2$, the saturation behavior of $Y_{Fe\text{-}W}$ was not observed [1].

In figure 3, sputtering yields estimated by the profilometer for Fe-W layers exposed in GyM (Y_{GyM}) at $E_K = 86$ eV and 286 eV as a function of the ion fluence are compared with the yields obtained by the exposure of Fe-W samples to HSQ D_3^+ ion beam (Y_{HSQ}) [3]. Both experiments show a decrease of Fe-W sputtering yield in the range of medium fluences ($1.0E23 - 1.0E24\ D^+/m^2$) for every deuterium energy. Y_{GyM} values at 286 eV are correctly placed between the points of Y_{HSQ} at 200 eV and 500 eV. The higher Y_{GyM} at 86 eV than Y_{HSQ} at 100 eV could be due to the fact that we were considering only D$^+$ fluence. Including also the fluence of deuterons from D_2^+ and D_3^+ in some way, Eq.1 leads to the lower values of the sputtering yield.
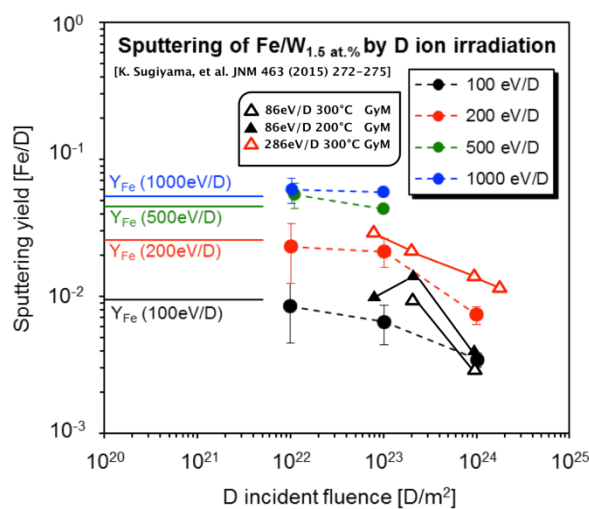

Fig.3: $Y_{Fe\text{-}W}$ as a function of D$^+$ fluence for different deuteron energy as estimated by profilometer from Fe-W layers exposed to the plasma of GyM (@200 °C, filled triangles; @300 °C open triangles) and HSQ setup at IPP Garching (filled circles) [3,15].

3.2 Temperature dependence

In order to investigate possible effects of the sample temperature on $Y_{Fe\text{-}W}$, a series of exposures was done at RT, 200°C, 300°C, 340°C and 500°C, keeping the fluence constant at $8.2E22\ D^+/m^2$. The erosion yield of Fe-W layers and pure Fe and W reference coatings (Y_{Fe} and Y_W), estimated from the profilometer measurements, as a function of the sample-holder's temperature, is shown in figure 4 for the bias voltage of 100 V (a) and 300 V (b).

Regardless the bias voltage, a reduction of $Y_{Fe\text{-}W}$ was found for temperatures between 200 °C and 500 °C (the scale of the erosion yield is log-10). This behavior was also observed after EUROFER irradiation with the deuterium plasma of PISCES-A [1]. It may be correlated to the growing importance of W diffusion into Fe toward the surface of the samples thus contributing to the W enrichment of Fe-W layers. At this purpose, the evaluation of W at the surface of these samples was made by RBS and the results are described in the next subsection.

Since W $E_{th} \approx 200\,eV$, erosion of W coatings for exposures at 100 V was accordingly below the depth resolution of the instrument (~10 nm). Although the physical sputtering of pure iron and tungsten should not depend on temperature, the scattering of Y of Fe and W reference samples in figure 4 can be explained considering the fact that they are not bulk materials but magnetron sputtering coatings whose morphology and structure (and hence the erosion) may vary with the temperature. As expected, Y_{Fe-W} always stays between Y_{Fe} and Y_W.

The general dependence of Y with ion energy was accordingly observed for all layers (Fe-W, Fe and W samples). The data in figure 4a are indeed all below the corresponding points in figure 4b.
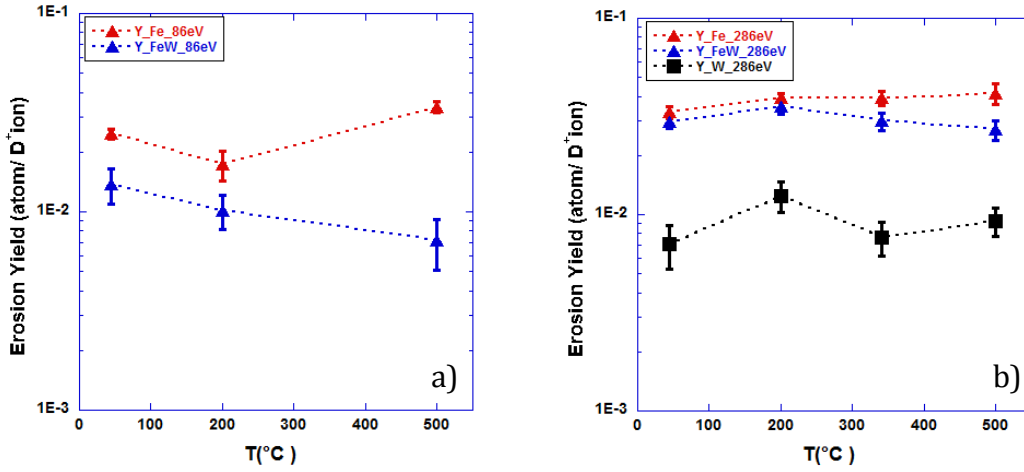

Fig.4: Temperature dependence of Y_{Fe-W} estimated from the profilometer measurements at D^+ energy of 86 eV (a) and 286 eV (b). The sputtering yield of reference Fe and W layers is also reported. At 86 eV the eroded thicknesses of W are below the depth resolution of the profilometer and the corresponding Y_W is not reported in figure (a).

3.3 W surface enrichment

We have seen so far that the behavior of Fe-W sputtering yield primarily depends on deuterium ion fluence and sample temperature. In order to understand the impact of these two parameters on Y_{Fe-W} separately, we had performed exposures at different fluences keeping the temperature at values much smaller than 500°C (subsection 3.1), and exposures at different temperatures keeping the fluence constant (subsection 3.2). The general trend is that both, fluence and temperature lead to a decrease of Y_{Fe-W} probably due to the higher concentration of W at the surface of Fe-W layers during the experiments. In order to prove this statement, the RBS measurements on Fe-W samples are described here. For each layer, the RBS measurement was performed on both, the exposed and unexposed side of the sample.

Figure 5 depicts the RBS spectra of Fe-W layers exposed at 340 °C and 300 V for different D^+ ion fluences. The RBS spectrum of the unexposed side of one of the samples is also shown for comparison. The increase of the W surface concentration with fluence is visible from the growing intensity of backscattering peak at 3450 keV associated to W (as also observed for Fe-W samples exposed in HSQ setup, figure 3 of [3]). This clearly explains the reduction of Y_{Fe-W} with fluence reported in figure 2b. The fact that diffusion of W into Fe was negligible

up to the temperature of 340 °C is confirmed by the absence of the W peak at 3450 keV in the spectrum of the unexposed side of the Fe-W layers. W enrichment of the surface of Fe-W samples was also observed after the exposures at 100 V of figure 1a.

The shift toward higher energies and the decrease of the intensity of the feature between 1500 keV and 2500 keV in figure 5, testify the progressive erosion of Fe-W coatings increasing the deuterium fluence.
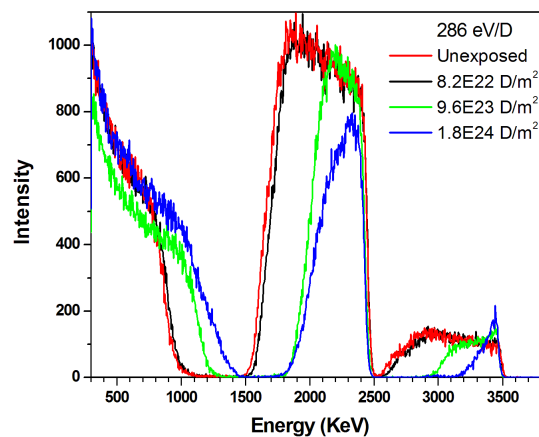

Fig.5: RBS spectra of Fe-W layers exposed to GyM deuterium plasma at 340°C and 286 eV/D for different fluences. The spectrum of the unexposed side of one of these samples is also reported.

From literature [1], it is known that diffusion of W into Fe becomes very effective from 500 °C. As a proof of this, the RBS spectrum of a Fe-W layer annealed at 500°C in the ultra-high vacuum ($5.0E - 10\ mbar$) chamber for 30 min (which is approximately the pre-heating time of Fe-W layers exposed at 500 °C) was acquired (figure 6a). A pronounced peak at 3450 keV fingerprints the presence of a W-rich surface layer. The same feature is visible in the RBS spectra (also shown in figure 6a) of the un-exposed side of the samples bombarded with deuterium ions ($8.2E22\ D^+/m^2$) at 500 °C. Since these experiments lasted 45 min on the whole (pre-heating and exposure times are 30 min and 15 min, respectively), the peak of the un-exposed side of the Fe-W layers is accordingly slightly higher than that of the annealed sample. The depth of the W-rich layer for these samples was however very thin, ~0.3 nm, as estimated by RBS. Recently, Low Energy Ion Scattering (LEIS) measurements of these samples have also been performed at IFP-CNR. They confirm the presence of a surface region with a high W concentration. Details of this analysis will be the subject of a future work.

Considering the exposed side of the samples at 500 °C, during the pre-heating phase (~30 min) a W-rich layer formed on their top. This, together with the W atoms diffused toward the surface during plasma exposure, is mainly responsible for the decrease of Y_{Fe-W} at higher temperatures of figure 4. It is interesting that, as shown in figure 6b, the W peak at about 3450 keV is not present in the RBS spectra of the exposed side of these samples. Although if the fluence was too low to lead to a W enrichment due to Fe preferential sputtering (see figure 5), one might expect that a peak could have been detected as a consequence of the

thermal effects shown in figure 6a. The reason for the absence of the peak is probably related to the very shallow (sub-nm) W surface layer established during the pre-heating phase. We speculate that the plasma species completely removed this W-rich layer and the diffusion of W atoms towards the surface was slower than W erosion rate by deuterium ions during the exposure.
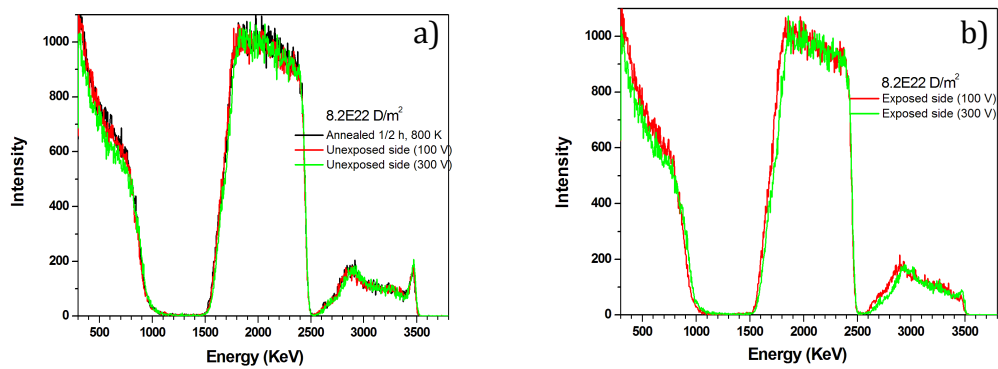

Fig.6: RBS spectra of Fe-W layers exposed at 500 °C and 100 V, 300 V as bias voltages. Measurements on the unexposed side (a) and the exposed side (b) are shown. In (a) also the spectrum of a Fe-W layer annealed at 500 °C for 30 min in vacuum is displayed.

3.4 Deposition and contaminants

Considering the parameters of GyM's plasma (n_e and T_e), no iron re-deposition after ionization of sputtered atoms was expected on Fe-W samples. The evaluation of the deposition of contaminants from the environment and the stainless steel sample's holder during the exposures is anyway important to find a possible spurious contribution to the erosion results described so far. For this reason, ToF-ERDA and SIMS were performed on Fe-W layers and silicon samples. The Fe-W layer exposed to GyM D plasma at 200 °C, 300 V and $2.0E23\ D^+/m^2$, was analyzed by means of ToF-ERDA. The ERDA spectra were acquired on the unexposed and exposed side of the sample as shown in figure 7. The comparison of the two spectra clearly shows that after deuterium bombardment, aluminum (Al) and nitrogen (N) species are present at the sample's surface. Moreover, as revealed by ToF-ERDA depth profiling (not shown here), a slight increase of the carbon (C) concentration is found in the exposed side of the sample (5% at.) compared to the unexposed side (3 % at.). The presence of these species can be explained by the contamination of the plasma chamber due to the previous experimental campaigns.

High percentage of oxygen, O (20% at.), was found both in the masked and unmasked side of the sample surface indicating that it was retained during the preparation of the sample via magnetron sputtering or immediately after its exposure to the air.
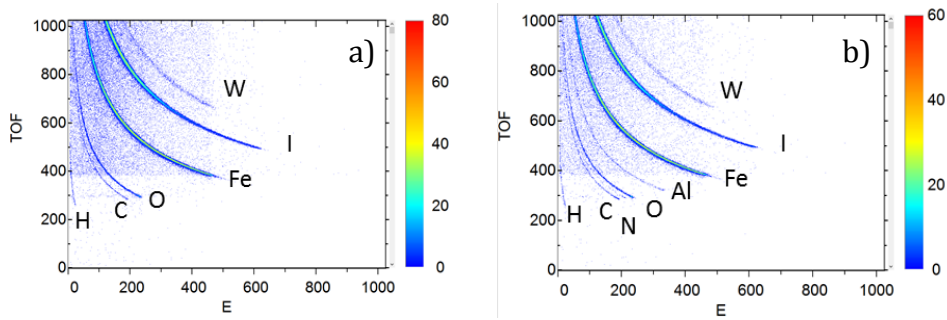
Fig.7: ToF-ERDA spectra on the unexposed (a) and exposed (b) side of the Fe-W sample exposed to GyM D plasma at 200 °C, 300 V and 2.0E23 D^+/m^2.

In order to evaluate the importance of iron deposition from the stainless steel sample holder, SIMS measurements were carried out on the polished pure (100) Si sample exposed to 286 eV/D ions at the fluence of 9.4E23 D^+/m^2. Wide scan positive ions mass spectrum on the exposed and unexposed side of the sample is reported in figure 8. The exposed side of the Si sample shows the presence of the signals associated to environmental contaminants such as sodium (Na), potassium (K) and calcium (Ca). Chromium (Cr) and Fe with some clusters of their oxides from sputtering and subsequent deposition of stainless steel of the sample holder are found as traces. Concentration of Fe and Cr at the surface layer is indeed very low (0.01 and 0.003 at.%, respectively, as obtained from the relative sensitivity factors). Following this result we can conclude that the deposition rate of Cr and Fe during the exposures is shallow and therefore is not significantly affecting the estimated erosion yield of the Fe-W exposed layers.
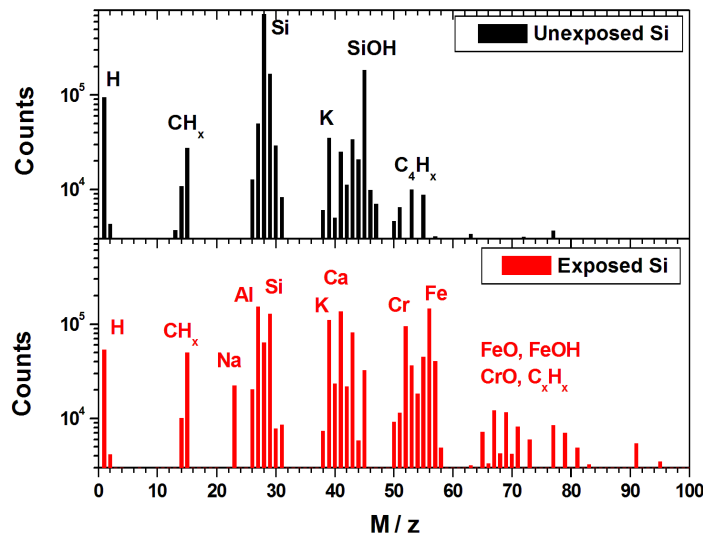

Fig.8: SIMS spectra of the unexposed (upper panel) and exposed (lower panel) of the (100) Si sample exposed to 286 eV/D+ ions at the fluence of 9.4E23 D/m².

4. Conclusions

FeW model systems of EUROFER steel were exposed to the GyM D plasma in the fluence range from $8.2E22\ D^+/m^2$ to $1.8E24\ D^+/m^2$ as a function of the applied voltage (-100 V, -300 V) and temperature (RT, 200 °C, 300 °C, 340 °C and 500 °C) of the samples. All the experiments were performed at the same pressure of 5.0×10^{-5} mbar and D^+ ions flux in the range $0.9 - 1\times10^{20}\ m^{-2}s^{-1}$.

An attempt was made to distinguish the effects on W enrichment of the selective Fe sputtering and of the W thermal diffusion, properly choosing the exposure conditions. Results show that for low exposure temperatures (< 500 °C) the erosion yield of Fe-W layers decreases with increasing fluence for both ion energies due to the progressive selective sputtering of iron and the consequent formation of a W-rich layer at the sample's surface. In this case W surface enrichment due to the W surface thermal diffusion can be neglected as demonstrated by the RBS measurements on the unexposed side of the samples.

The Fe-W sputtering yield decreases from $2.9 \times 10^{-2}\ atoms/D^+\ ion$ to $1.1 \times 10^{-2}\ atoms/D^+\ ion$ for 300 V exposures in the fluence interval reported above. In this range, the comparison with the erosion yield results from Fe-W samples exposed to HSQ D_3^+ ion beam shows a quite good agreement.

In order to investigate W enrichment contribution coming from the W thermal diffusion, experiments were performed at the low fluence of $8.2E22\ D^+/m^2$ where no W surface enrichment by Fe selective sputtering was found. Results show that Fe-W erosion yield decreases with the increasing sample exposure temperature due to the raising importance of the W thermal diffusion within the surface layer. A shallow W thin layer was found on the masked side of the sample exposed at 500°C and no W surface enrichment was detected on the exposed side. This could be explained by the fact that during the exposure, the D ions sputtering rate of W is still higher than the W diffusion velocity toward the surface.

We can conclude that the main result in the range of the investigated parameters is that the effect of the ion fluence on the reduction of Fe-W sputtering yield is stronger than that of temperature, which can be neglected below 500 °C.

In order to understand the synergic effect between W thermal surface diffusion and selective Fe sputtering, higher fluence and higher temperature experiments need to be performed on Fe-W layers.

Acknowledgement

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