LIBS detection of erosion/deposition and deuterium retention resulting from exposure to Pilot-PSI plasmas

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Samples with tungsten (W) and tungsten–yttrium (Y) coatings on molybdenum were exposed to plasmas simulating the divertor conditions of ITER. The exposed surfaces were studied using in-situ laser induced breakdown spectroscopy diagnostics (LIBS) and the results were compared to those obtained by other surface characterization methods. Our results show that LIBS is a reliable technique for in situ monitoring of erosion, deposition, and fuel retention processes under reactor-relevant conditions. In the regions of the highest gross erosion the thickness of the remaining tungsten layer is thinnest and at the central part of samples both mixing and deposition of components took place. LIBS also allowed reliable recording of D retention. D accumulation is influenced by the surface temperature as well as by the surface irregularities.

1. Introduction

Plasma-surface interactions largely determine the life-time of wall components in fusion reactors. To minimize the damage that high power and particle fluxes will induce on them, operational plasma scenarios need to be optimized but, in addition, efficient in situ control of erosion, deposition, and fuel retention characteristics of wall structures are required. Laser-based methods are promising ones for remote in-situ monitoring of such processes [1,2]. In the case of laser induced breakdown spectroscopy (LIBS), the laser radiation evaporates a small amount of the tested material and by analyzing the spectrum of the formed plasma plume, the composition of the studied sample can be determined. The elemental depth profiles can then be reconstructed by evaluating the integrated intensity of selected spectral lines as a function of the number of laser pulses. A necessary requirement is that the amount of material ablated per pulse (ablation rate) has been determined beforehand.

Up to now, most of the studies related to the development of LIBS are carried out under ex situ conditions and using samples with a known composition and thickness. Only few [3–5] LIBS measurements have been carried out in conditions which correspond to a reactor relevant in situ setup. Carrying out in situ LIBS testing allows eliminating the influence of different ageing effects (oxidation, degassing, etc) which can mask plasma-induced effects at the sample surface.

Our post mortem LIBS experiments with plasma-exposed samples have shown [6] that the exposure of samples to high-energy ion fluxes changes the surface crystallinity and, as a result, the ablation rate is drastically reduced. At the same time, the surface morphology of the material can also change considerably [7]. This is an issue especially for W which has been selected as the divertor material of ITER. The result will be even larger changes in the ablation rate and, moreover, the more modified the surface becomes, the larger will be the trapping of hydrogen isotopes in it [8]. Thus, the development of LIBS as an in situ technique requires setting up an extensive database on the influence of various parameters on the LIBS spectra and cross comparison with other surface characterization methods like electron scanning microscopy (SEM), secondary ion mass spectrometry (SIMS) and nuclear reaction analysis (NRA).

The experiments reported here have been carried out in the
linear plasma machine Pilot-PSI, where plasma fluxes could reach values comparable of those of the ITER divertor [9–14]. The energy deposited on the target by charged particles, ions and/or electrons can be flexibly controlled in Pilot-PSI and also the surface temperature and the fluence can be varied within large intervals.

The study had three main tasks:

- In situ recording of LIBS profiles of samples immediately after their exposure to plasma.
- Determining the influence of surface morphology and roughness on the LIBS profiles by comparing LIBS results with those from ex situ analyses.
- Clarifying the effect of surface conditions on fuel retention.

2. Experimental

2.1. Samples

In the experiments different test samples produced by DIARC-Technology Ltd (Finland) were studied. The vacuum arc-discharge technique [15] was used for coating molybdenum (Mo) substrates with 2-μm thick tungsten (W) or mixed tungsten-yttrium (W/Y, Y content ~5 at. %) layers. In the coatings Y is likely in the form of an oxide [16]. The use of W/Y was triggered by the results of [17,18] which demonstrated that the inclusion of a few weight % of Y in W changes remarkably the material properties. Our task was to test how the Y addition influences the plasma-induced erosion. For determining net erosion of the exposed samples, also a reference coating with a 2 μm thick W layer on silicon was used to obtain the original thickness of the marker coatings by SIMS.

2.2. Plasma characteristics

Altogether four samples were exposed to deuterium or to deuterium-neon (D/Ne) plasmas. Neon was used to simulate the effect of heavy seeding gases and sputtered impurities from wall structures on plasma-surface interaction processes: according to [19], at low (=20 eV) electron temperatures the sputtering yield for Ne ions is approximately by an order lower than that of W ions. As the used Ne flux (Table 1) considerably exceeds W flux expected in reactors and is comparable to the one expected for light particles [20], we assumed that the sputtering effects induced by these ions are comparable.

Neon will also produce cracks and other imperfections at the surface, thus enhancing the deuterium retention. The electron density $n_e$ and temperature $T_e$ of the beam had bell-like distributions with maxima at the center where $n_e = 1.4 \times 10^{20} - 3.7 \times 10^{20}$ m$^{-3}$ and $T_e = 1–2$ eV. The other exposure parameters have been collected in Table 1. The flow rates in the table are volumetric flow rates in the plasma source inlet. It should be pointed out that for enhancing erosion the used Ne flows were very large compared with those of heavy particles in fusion reactors.

Samples 1 and 2 were exposed only to D/Ne plasma, while samples 3 and 4 passed a two-step plasma treatment. The aim of the first step was to modify the sample surface to favor retention of deuterium during the second exposure step. According to [21] the retention drastically decreases with increasing surface temperature. This is the reason why during the second step the surface temperature was kept below 600 °C. For more homogeneous D distribution along the sample surface, during this loading phase the plasma beam half-width, FWHM, was two times larger. Besides, the D flow rate was almost four times larger than in the D/Ne exposure.

In the case of D/Ne plasmas the particle flux was approximately $10^{24}$ m$^{-2}$ s$^{-1}$ while in the case of D plasmas it was nearly 20 times lower. In all cases the value of the bias voltage was ~40 V, corresponding to ion temperatures of around 38 eV on the target.

During the plasma exposure a spectral pyrometer and an IR camera were used to record the surface temperature of the samples.

To estimate gross erosion, a fast visible light camera with an interference filter for the neutral atom spectral line of W at 400.9 nm was used. The frame rate and the exposure time for the camera were 100 Hz and 9.9 ms, respectively. To estimate the contribution of the continuous background radiation to the signal recorded within the transmission band of the filter, a spectrometer with a 300-lines/mm grating was used.

2.3. LIBS setup

Fig. 1 gives a sketch of LIBS setup used for in situ measurements.

An Nd:YAG laser ($\lambda = 1064$ nm, pulse duration 8 ns) was applied. The laser was at ~20 m distance from the Pilot PSI device. The laser beam was first expanded to have a beam diameter of 3 cm and then guided via mirrors and finally focused on the sample using lenses with an effective focus length of 1.5 m. The laser pulse energy at the sample surface was 160 mJ and the area of the laser spot was approximately 1 mm$^2$ corresponding to a fluence of 16 J/cm$^2$. Using a plane mirror and a lens with a 30-cm focal length, the radiation of the laser-produced plasma was directed into a 0.8-mm-diameter fiber. The magnification of the plasma image at the fiber end was close to unity. The other end of the fiber was coupled to a fibre bundle consisting of 50 fibers, each of them having a diameter of 120 μm. At the spectrometer entrance the fibers in the bundle were arranged in a linear array. This array was used as an entrance slit of a Czerny-Turner spectrometer (focal length 1 m) coupled with an image intensifier and Manta G-145 CCD camera. The signal at a certain wavelength was formed by a number of vertically binned CCD pixels. In the case of LIBS both the signal-to-background ratio and the width of spectral lines change with time [22]. This is the reason why the time-resolved spectra were recorded at values of the delay time $t_\Delta$ between the laser pulse and the beginning of data acquisition and the recording time-gate $\Delta t$, where the lines were sufficiently narrow and were still clearly distinguishable from noise.

Two different gratings were used: a 300 lines/mm grating for recording the spectra in the 420–490 nm range containing mainly W and Mo lines and a 1200 lines/mm line grating for higher resolution measurements close to Balmer $\alpha$-lines of hydrogen isotopes.

### Table 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>Coating</th>
<th>Plasma &amp; exposure</th>
<th>Field T</th>
<th>FWHM mm</th>
<th>Plasma current, A</th>
<th>Peak temp, °C</th>
<th>D$_2$ flow rate, slm</th>
<th>Ne flow rate, slm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>W</td>
<td>D/Ne; 600 s</td>
<td>0.8</td>
<td>12</td>
<td>50–60</td>
<td>1300</td>
<td>0.7</td>
<td>2.22</td>
</tr>
<tr>
<td>2</td>
<td>W/Y</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>W</td>
<td>D/Ne; 300 s + D; 300 s</td>
<td>0.8</td>
<td>12</td>
<td>50–60</td>
<td>1300</td>
<td>0.7</td>
<td>2.22</td>
</tr>
<tr>
<td>4</td>
<td>W/Y</td>
<td></td>
<td>0.4</td>
<td>25</td>
<td>10–15</td>
<td>&lt;600</td>
<td>2.5</td>
<td>0</td>
</tr>
</tbody>
</table>
The W/Mo spectra were recorded at the Pilot PSI background pressure of \(10^{-3}\) mbar while the hydrogen isotope lines were reliably recorded only by using an argon background pressure of 1.2 mbar. Because of different background pressures, also the expansion rates of the laser-produced plasma plume were different and thus for the W/Mo spectra the optimum \(t_d\) value was 100 ns while for D/H spectra \(t_d = 200\) ns. In both cases, the width of the time-gate \(\Delta t\) was 3 \(\mu\)s.

The in situ LIBS measurements were carried out immediately after plasma exposure. Spectra as a function of the number of laser shots were recorded from selected regions on the sample, differently modified by the plasma. Samples 1 and 2 were used for recording of spectra in the 420–490 nm range, while samples 3 and 4 were used for recording of lines of hydrogen isotopes.

2.4. Ex situ diagnostics

There were long time intervals between Pilot-PSI measurements and ex situ tests, from one (SEM) to 10 (NRA) months. Between different analyses the samples were kept in ambient air which could cause uncontrolled changes like outgassing of samples.

SEM pictures were taken by a Helios\textsuperscript{TM} NanoLab 600 (FEI) device. A magnetic sector SIMS VG Ionex IX-70S with an \(O_2^+\) primary ion beam was applied for recording elemental depth profiles in the coatings. The primary beam was rastered across a \(0.3 \times 0.4\) mm\(^2\) area and by using electronic gating, only signal from the central part (10\%) of the resulting crater was recorded. The beam current was 200–500 nA and the ion energy 5 keV.
For analysis of deuterium content in the sample No. 4 micro-beam NRA analysis were performed employing \textit{d}({\textit{^3}\text{He},p})\textit{a} nuclear reaction. For this purpose a proton detector with thickness of 1000 \textmu m was added to the standard detector configuration on the micro-beam end station at Institute Jozef Stefan [23]. The NRA detector has a solid angle of 0.14 sr and is positioned at an angle of 135° with respect to the beam direction. In case of microbeam nuclear reaction analysis (\textmu NRA) the \textit{^3}\text{He}^{2+} ions with energies of 3.3 MeV where focussed onto 10 \times 10 \textmu m^2 area with beam current of 300 pA. At a fixed sample position, the beam scanned over 2.2 \times 2.2 mm^2 sample area. Carrying out the scanning at different sample positions, the information about the species distribution along the sample surface was obtained. NRA gave information about the total D concentration (at cm$^{-2}$) in the layer down to a thickness of $\approx 5$ \textmu m. Scanning with the focused \textit{^3}\text{He} beam over an area of 2.2 \times 2.2 mm^2 gave information of the lateral distribution also of other species. Simultaneously to \textmu NRA measurements, the detection of particle induced X-ray emission was performed and yields for W and Mo were obtained.

3. Results and discussion

The flux of the D/Ne plasma caused intense erosion of the sample material accompanied by noticeable radiation for the W 400.9 nm line (Fig. 2A). As at the D-ion energies $\leq$20 eV the sputtering yield is $< 10^{-5}$ [19], in the case of D plasma flux the radiation originated from the sample surface was not detectable. Thus, the erosion of samples is mainly caused by Ne ions.

The emission pattern of W from the sample surface recorded using the 400.9 nm filter exhibits a ring-like profile with the radial maximum at $X \approx 10$ mm from the center (Figs. 2A and 3A). Actually, at $X \approx 0$ mm the main contribution to the total radiation comes from the continuous background radiation (Fig. 3B) and the intensity of the W line is low. Thus at the center erosion is practically missing. From here it follows that the distribution of Ne ions, the main sputtering species, in the plasma column shows a hollow profile. On the other hand, Fig. 3A indicates that the highest power load occurs at the center where the surface is the hottest. These features are typical to strongly magnetized plasma in linear plasma machines. As measured in Ref. [10], both the electron density and temperature had their maxima at the center of the plasma beam, while the radial emission profile of the plasma column is similar to what was discussed above. Further analyses of the Pilot-PSI plasma demonstrated the dependence of its structure on the bias voltage [12]. Recent probe studies [15] showed that depending on the bias voltage of the sample, in linear plasma machines the plasma flux can bombard the sample with electrons in the central region and with ions in the remaining part.

SEM pictures (Fig. 4) demonstrate that the ring-like emission pattern on the surface (Fig. 2B) reflects the differences in surface morphology.

In case of all four tested samples, the main surface features were the same. At the central part of samples the surface has a grain-like structure which in the peripheral zones is replaced by domains of lamellar structure (Fig. 4). It also seems that at the center the surface is more porous and at distances $8 \leq X \leq 11$ mm the surface shows an increased roughness.

Net erosion was studied by LIBS immediately after the exposures. The LIBS spectra were recorded from different regions on the surface and each crater was ablated by 100 laser shots. Fig. 2B
shows an example of the resulting surface for sample 1. From the recorded spectra, lines which had an acceptable signal-to-noise ratio in the 420–490 nm spectral range were selected and are shown in Table 2.

To construct elemental depth profiles we chose spectral lines belonging to transitions, whose upper state energies $E_k$ do not differ much from each other: for W 468.761 nm and Mo 473.144 nm lines the $E_k$ values are 5.03 and 5.22 eV, respectively. As a rough estimate, we assumed that the populations of these states $x_k = I_{ki}/g_k A_{ki}$ are proportional to the concentration of different elements in the studied layers. Here $g_k$ is the statistical weight of the upper state and $A_{ki}$ is the coefficient of spontaneous emission. Thus, the relative concentrations $r_{Mo}$ and $r_W$ could be found as $r_{W/Mo} = x_{W}/x_{Mo}$.

Fig. 5 shows the Mo and W LIBS profiles for sample 2 for different positions on the sample, marked by distance from the center in mm. The sequence of $X \geq 5$ mm profiles could be related to the thickness of W coatings. Indeed, according to Fig. 2 the intensity of the neutral W emission (i.e. the gross erosion) is the highest at $X \approx 10$ mm and it gradually decreases towards the sample center. At the same time the SEM images in Fig. 4 show that the sample surface at distances $X \geq 5$ mm have almost the same lamellar structure which leads to the assumption that the ablation range remains unchanged in this region. Thus, at $X = 9$ mm the surface layer is the thinnest and the thickness gradually increases towards the center. Closer to the center ($X = 2$ mm in Fig. 5), the shape of LIBS profiles drastically differs from that at larger distances, $r_W$ remains practically unchanged during 50 laser shots. In this region the Ne-caused sputtering is not efficient (Fig. 2), the coating is formed due to electron flux and a high power load. Besides, a large number of ablated particles could be re-deposited in the central part. All these factors could influence both the coating thickness and the ablation rate thus making the comparison of coating thickness with that at the outer part doubtful.

At the central part of samples ($X = 2$ mm) we see the presence of Mo starting from the first laser shot. This is the reason why the value of the relative concentration $r_W$ at the center is lower than at $X = 5$ mm.

Besides, at the center both $r_W$ and $r_{Mo}$ remain practically unchanged during 50 laser shots. We think that Mo originates either from the plasma source (cathode material is Mo) or from Mo clamps for fastening the samples. In addition, the high temperature and electron bombardment at the center of the sample will lead to mixing of W and the substrate Mo. Indeed, if the grains at $X = 2$ mm (Fig. 3) have more-or-less spherical form, the gaps between the grains could be micrometer deep and thus the plasma flux could reach the substrate and mixing is initiated.

From the comparison the depth profiles for W/Y and W coatings at $X = 9$ mm (Fig. 5) it is evident that for removal of the W/Y coating more laser shots are needed i.e. Y increases the durability of the coating. Nevertheless, the mechanism behind this effect is not clear and needs further research. One possible explanation is that the Y content inhibits the grain growth in the W samples [16]. Also the higher hardness of Y-containing samples is reported in Ref. [16].

SIMS depth profiles as function of the distance X were recorded in the direction what was perpendicular to the craters row in Fig. 2A. Assuming that SIMS results are not influenced by surface morphology, the profiles in Fig. 6 show that at $X = 9$ mm the surface layer is the thinnest and the thickness gradually increases towards the center. Thus, the trends in the LIBS profiles are mainly related to surface layer thickness and possible changes of the laser ablation rate seems to be of smaller importance. Comparison of W profiles with that of the reference one (R in Fig. 6B) indicates that the coating thickness of unexposed sample is the largest. Thus it is possible to confirm that at distances $X > 5$ mm in the erosion/re-deposition interplay erosion dominates. Profiles belonging to $X = 4, 5, 6$ mm in Fig. 6A show that Mo at the top of the sample (<1000 mm) is separated from the substrate Mo. Thus, at least for these X values the presence of the top Mo is caused by deposition.

Near the sample center ($X = 2, 3$ mm) where the surface temperature is the highest, the W-Mo ratio remains unchanged at least within first 1000 nm. Whether mixing takes place with the substrate Mo or with the Mo from some another source is still unclear.

Comparing SIMS and LIBS profiles gives for the W/Y coating the laser ablation rate ~10 nm per shot. Dashed lines in Fig. 6 demonstrate the effect of plasma exposure time. According to Table 1, for sample 4 the exposure time to Ne/D plasma is two times shorter and the thickness of the remained layer at $X = 5$ mm

<table>
<thead>
<tr>
<th>Element</th>
<th>Wavelength, nm</th>
<th>$g_{Mo}A_{Mo}$, s$^{-1}$</th>
<th>$E_k$, eV</th>
</tr>
</thead>
<tbody>
<tr>
<td>W I</td>
<td>465.987</td>
<td>3.00E+06</td>
<td>2.66</td>
</tr>
<tr>
<td>W I</td>
<td>468.761</td>
<td>1.33E+07</td>
<td>5.03</td>
</tr>
<tr>
<td>W I</td>
<td>484.381</td>
<td>9.50E+06</td>
<td>2.97</td>
</tr>
<tr>
<td>Mo I</td>
<td>470.726</td>
<td>3.27E+08</td>
<td>5.12</td>
</tr>
<tr>
<td>Mo I</td>
<td>473.144</td>
<td>4.98E+08</td>
<td>5.22</td>
</tr>
<tr>
<td>Mo I</td>
<td>476.019</td>
<td>6.07E+08</td>
<td>5.25</td>
</tr>
<tr>
<td>Mo I</td>
<td>481.925</td>
<td>2.44E+08</td>
<td>5.2</td>
</tr>
<tr>
<td>Mo I</td>
<td>483.051</td>
<td>2.85E+08</td>
<td>5.2</td>
</tr>
<tr>
<td>Mo I</td>
<td>486.800</td>
<td>1.56E+08</td>
<td>5.14</td>
</tr>
</tbody>
</table>

Fig. 5. LIBS depth profiles for Mo (A) and W (B), recorded from sample 2 (W/Y coating, D/Ne exposure), as a function of the number of laser pulses. The distances are in mm from the sample center. Thinner lines (for $X < 7$ mm) show the results without smoothing, while thicker lines present moving average over 4 successive points. Profiles from sample 1 (W coating, D/Ne exposure) at $X = 9$ mm are marked using dashed lines.
is \( \approx 1.5 \) times larger.

W and Mo distributions recorded by \(^{3}\text{He}\)-beam induced X-ray emission (PIXE) are shown in Fig. 7, the yields are not related to each other. Recordings from nine sample positions along the radial line of the sample, just above the SIMS measurement line were used for analysis. Data from the \( 2.2 \times 2.2 \) mm\(^2 \) area belonging to a certain sample position are divided into 8 subsections. It should be pointed out that for a fixed \( X \), the PIXE recordings have been integrated over \( z \approx 5 \) mm depth.

At the central part of the sample (\( X = \pm 3 \) mm) Mo signal is the strongest. The Rutherford backscattering spectra also show a mixture of Mo and W layer on top surface what is not observed further away from the center. Likely because of the porosity at the sample center the W coating is more transparent to the X-ray radiation originating from the substrate. The explanation for increasing W and Mo yields within \( 4 < X < 10 \) mm region matches with the above-presented analyses of the relationship between the gross erosion and the thickness of the remaining W layer.

The LIBS results for H and D content of the exposed samples are shown in Fig. 8. The \( D_a \) and \( H_a \) lines were fitted with Lorentzian contours (FWHM = 0.14 nm). At \( X = 9 \) mm the deuterium signal can be reliably detected during the first five laser shots (Fig. 8A). At a fixed laser shot the \( D_a \) intensity decreases gradually towards the center (not shown in figures). Near the center (Fig. 8B) where the surface temperature was the highest, the intensity of the \( D_a \) line of the 3rd shot is already at the noise level. It is natural to relate the D retention dependence on \( X \) to the surface temperature. Indeed, the study [17] demonstrated that the temperature growth from 500 to 800 K causes the fall of D retention by three orders of magnitude.

The intensity of the \( H_a \) line remains practically independent of the laser shot number, likely it is caused by the presence of water vapor in the background argon gas.

Fig. 9A presents D profiles recorded by SIMS. The common trends of SIMS results match with those of LIBS: at \( \approx 30 \) nm depth (corresponding to the 3rd laser shot) the \( D_a \) intensity at the sample center was more by an order of magnitude lower than at \( X = 9 \) mm distance (Fig. 9A). A remarkable discrepancy with LIBS (Fig. 8A) is that at \( X = 9 \) mm the intensity of H signal always is larger than that of D signal. The likely reason of this difference is the contamination of the sample surface by water vapor during a long break between LIBS and SIMS measurements.

Like LIBS and SIMS, \( \mu \text{NRA} \) (Fig. 9B) shows a low level of deuterium signal at \( |X| < 2 \) mm. At \( X > 2 \) mm the D concentration fluctuates remarkably and reaches its peak value at \( X = 4 \) mm. The obtained D amounts is the integral of signal over the analysed depth of 5 \( \mu \)m where as in the case of SIMS and LIBS measurements the deuterium depth profile is shown. The D amounts for each point were obtained over an area of \( 2.2 \times 0.3 \) mm\(^2 \), providing information...
along the whole radial axis. On the other hand the LIBS and SIMS made analysis on individual spots integrating over the analysing area. The fluctuations can be explained by a much smaller spot area in μNRA meaning that μNRA gives more local information about the conditions at the sample surface. Large-scale fluctuations obtained by μNRA seem to confirm that the D distribution at the surface is uneven, largely localized to irregular spots [9] and inside the outermost erosion ring thus being co-deposited with impurities.

4. Conclusion

The most important findings of the paper are.

- During Ne/D plasma beam action, the distribution of the erosion-related radiation of tungsten at the sample surface has a hollow structure. At the same time the sample temperature is the highest at the center. Plasma-induced morphology also changes remarkably with the distance from the sample center. These findings confirm once again that electrons dominate in the central part of the plasma beam while the peak of ions is at the outer part of the beam.
- In situ LIBS gives a reliable set of elemental depth profiles showing that in the regions of the highest gross erosion the thickness of the remaining tungsten layer is the thinnest. The similarity of LIBS profiles with the SIMS data allows to estimate the value of the ablation rate. Elemental profiles at the central part of samples indicate that both mixing of elements and co-deposition took place. The main trends obtained for W and Mo by μNRA-PIXE match with LIBS and SIMS results.
- Both LIBS and SIMS recordings show that the efficiency of deuterium retention is the highest at the outer part of the sample surface while at the sample center deuterium accumulates only in a very thin layer. From the μNRA results could be concluded that the deuterium is mainly trapped in irregularities at the sample surface.

Acknowledgments

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