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Dimple optimization for XPS characterization of TEXTOR tile depositions

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ABSTRACT

The plasma wall-interaction in ITER is facing several challenges including surface erosion, material redeposition and tritium retention. In order to assess some of the possible issues, samples from TEXTOR (Forschungszentrum Jülich Tokamak, Germany) have been studied in this work. A smooth gradient in the form of a dimple should be established to enlarge the view of the depositions in depth before analyzing them with X-ray photoelectron spectroscopy (XPS). The dimple depth was optimized in order to cover the deposition thickness. Profilometer measurements with a confocal microscope were carried out to retrieve information on the profile and to ensure the optimum size/form of the dimples. Scanning electron microscopy measurements were carried out with a JSM 6310 microscope in order to obtain the morphology of the dimple area to verify if the deposition was not put under stress. The chemical composition and distribution of the elements along the dimples and on the surface of the samples was detected with an energy dispersive X-ray spectroscopy (EDS).

1. Introduction

The fusion plasma, with a typical temperature of 10 keV, has to be brought into contact with a physical wall in order to remove the helium produced and drain the excess energy in the fusion plasma. The fusion plasma is far too hot to be brought into direct contact with a physical wall. It would degrade the wall and the debris from the wall would extinguish the plasma. Therefore, schemes are developed to cool down the plasma locally before it impacts on a physical surface. The resulting plasma wall-interaction in ITER is facing several challenges including surface erosion, material redeposition and tritium retention. Modelling studies on ITER have raised issues of material erosion and deposition, and the related tritium retention, as critical for its success, especially if it operates with carbon targets as is currently planned [1]. These concerns have been highlighted by the detailed studies of plasma facing components (PFC) after each operational campaign of the JET tokamak [1–8]. In the same way, erosion/deposition studies have been carried out in the TEXTOR tokamak (FZJ, Germany) [9].

Non-contaminated TEXTOR samples have been analyzed in order to obtain information about the surface morphology and composition. Besides that, a grinding technique to perform suitable dimples on the sample surface have been developed and tested.

TEXTOR is a medium size tokamak (major radius 1.75 m, minor radius 0.47 m) with a circular poloidal plasma cross section [10]. The position of the last closed flux surface (LCFS) is defined by the toroidal belt limiter ALT – II (Advanced limiter Test II) [11]. ALT – II consists of eight blades, each carrying 28 tiles ordered in two rows, with a total surface area of 3.4 m^2. The tiles are made of fine–grain isotropic graphite.

2. Experimental procedure

Several fine–grain isotropic graphite samples from TEXTOR have been studied. These samples correspond to tile 20 (thick deposition ~10–20 μm) and tile 21 (thin deposition ~100 nm until 1 μm), which were located in ALT – II (Advanced Limiter Test II). Specimens with thick deposition present a rough surface, whereas samples with thin deposition have polished surface. Plasma discharges were performed between 2005 and 2006, with a total pulse duration of 9365 s and an area averaged fluence of 2.9×10^{25} cm^{-2}.

The first part of this work was focussed on the adjustment of a Precision Dimpling Instrument MODEL 515 by South Bay Technology Inc., in order to obtain craters of suitable size on the sample surface. This crater gives a large slope and therefore indirectly a depth profile study of the deposition layer on the specimens. To measure the size of those dimples a STIL MICROMESURE 3D

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measuring system, whose main part is a CHR-150 contactless optical sensor, with accuracy of 0.03 μm in *z* axis (depth) and 1 μm in *x* and *y* axis (spatial resolution), was used.

SEM analysis of the specimen surfaces, performed with a JSM 6310 SEM by Jeol, gave the morphology of the surface and craters. The EDS ability on the SEM gave an idea about the chemical composition present in the bulk of the different kinds of depositions. Finally, XPS analysis were carried out with a ThermoVG Scientific XPS Escalab 220i-XL which allows an spot of 200 μm by 200 μm to obtain the chemical bonding of the elements at the surface layers. Depth profile analysis were also carried out with this machine, using an Argon ion of 3 kV ion energy and 1 μA of current

3. Results

In Fig. 1 three craters grinded on tile 21 sample are shown. Dimples *a*, *b* and *c* correspond to different adjustments of the grinding machine. After watching the different spots with SEM, it was found that the best performance of the machine was achieved in crater *c*, which has no residual spot in the centre of the dimple, and no signals of stress over the grinded region are found. In addition, this configuration allowed a spotless crater of 12 μm depth and 1.2 mm wide with a gradual smooth slope which is needed to have the best depth profile.

EDS analysis of tile 20 as received sample surface is shown in Fig. 2. Carbon is the main component, although Oxygen and Silicon can be found in noticeable amount as well. Besides that, other elements, like Al, S, K, Cr, Cl, Ni, Cu and Zn appear in a very low concentration (<1%).

The XPS survey spectrum corresponding to tile 20 is shown in Fig. 3. The three different curves represent analysis performed on the sample surface (outside), in the middle of the crater slope (crater) and the deepest level of a depth profile study equivalent to 3000 s. of Argon ion etching (depth profile). The main peaks found are Cl ~ 200 eV, Carbon ~ 285 eV, Oxygen ~ 534 eV, Cu ~ 934 eV and Zn ~ 1022 and 1045 eV. All these elements were found in previous EDX analysis carried out on these samples as well.

In Fig. 4 the Carbon 1s peak (285 eV) is shown for the sample surface (outside), crater slope (crater) and after 3000 s of etching (Depth profile) of the same thick deposition specimen taken from tile 20. The depth profile peak is the sharpest, whereas surface

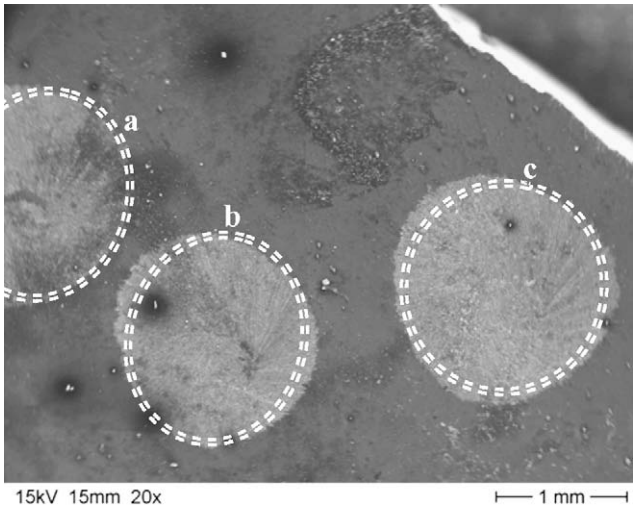

Fig. 1. SEM image of craters *a*, *b* and *c* performed with the grinding machine on tile 21.

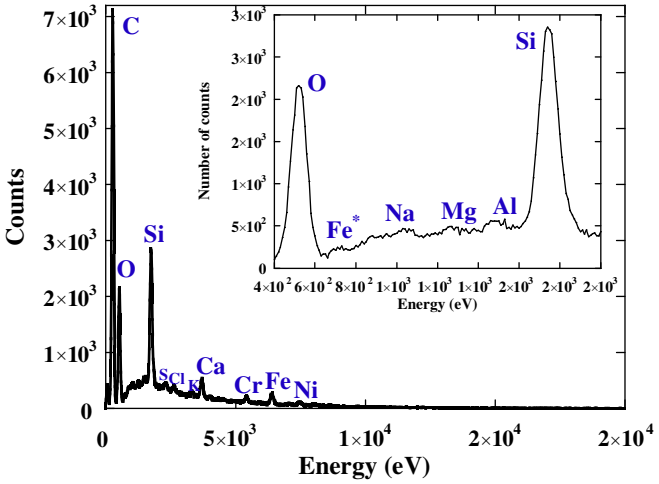

Fig. 2. EDS analysis of tile 20 as received sample surface.

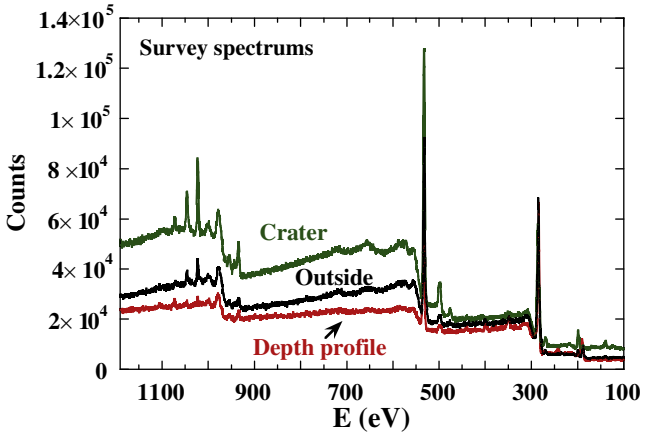

Fig. 3. XPS survey spectrum performed on the sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile) of thick deposition specimen (tile 20).

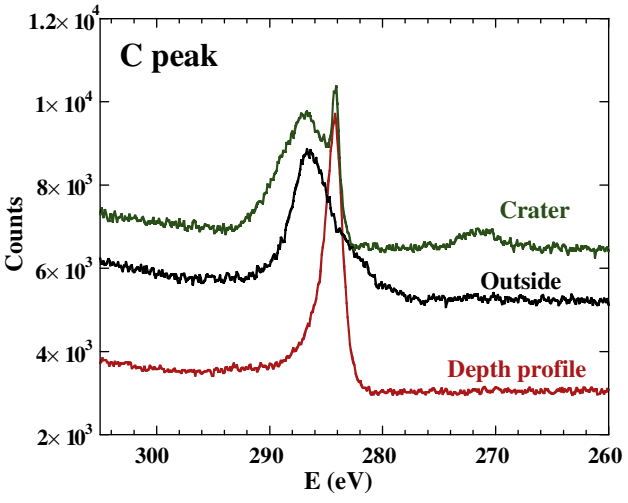

Fig. 4. XPS spectrum corresponding to Carbon 1s peak performed on the sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile) of thick deposition specimen (tile 20).

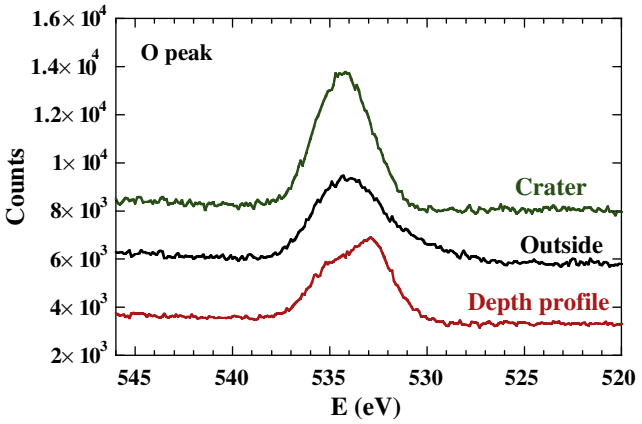

Fig. 5. XPS spectrum corresponding to Oxygen 1s peak performed on the sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile) of thick deposition specimen from tile 20.

sample one is wider. In the case of the crater peak, there is a mixture of both cases, therefore a double peak appears. Surface deposition components are linked to the peak centres in 288 eV, and bulk carbon is related to the original C1s peak. It can also be seen a small peak around 271 eV in the case of the crater curve is, due to Argon that originates from the ion etch cleaning of the surface sample.

XPS Oxygen 1s peak from tile 20 sample, i.e., thick deposition specimen, is shown in Fig. 5 in more detail. The three different curves represent analysis of sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile). The three peaks are centred at 534 eV binding energy, which is compatible with the O 1s. The depth profile peak is wider than the surface and crater peak; and on the top its shape exhibits a slope. This bump is related to a complex structure of bonds, which may be due to formation of some kind of Si–O and C–O bonds. The expected D/C ratio is indeed around ~10% [12].

A narrow scan of Copper $2p^3$ (934 eV) and Zinc $2p^1$ (1045 eV) and $2p^3$ (1022 eV) peaks are shown in Fig. 6. The three different curves represent XPS analysis of sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile). One can see how those peaks are clearly present in the case of crater slope, i.e., into the dimple, whereas there is no Cu and Zn in the depth profile, and only a little amount on the sample surface. The left spectrum shows the analysis of tile 20 and the right belongs to tile 21. It is clear that the same features are present.

4. Discussion

The results shown in Fig. 1 state how it is possible to achieve a spotless crater of around ten microns depth using a grinding machine on graphite samples. This means that, in principal, this technique can be used to study deposition profiles tens of microns thick on both non-contaminated and contaminated (Beryllium, tritium) carbon specimens.

An XPS depth profile and a crater slope analysis was carried out in samples from tile 20 and have been compared in order to be able to find common patterns. Both methods provide analysis of the chemical composition into the deposition layer, although at different depths. The main difference between them is the way to remove the deposited material form the sample surface. Either through sputtering with the Ar ion gun in the case of the depth profile or by using a grinding machine for the mechanical creation of a crater. The Carbon peak curve in the slope of the crater (Fig. 4) showed a superposition of peaks, whereas Oxygen peaks (Fig. 5) exhibit almost the same shape for both outside and crater curves. These two facts point out that during the grinding process small amount of material coming from the deposited layer goes into the dimple. However the material is not smeared out into the crater, because the Carbon crater peak would not show the combination of the surface and depth profile.

Other important issue is the presence of Cu and Zn in the crater in both tile 20 and 21 samples (Fig. 6a and b). Although Cu and Zn are present on the sample surface in very small amounts (Fig. 2), this contamination comes mainly from erosion of the grinding machine wheel, and in lower rate from the deposited layer. Therefore it is possible to find Cu and Zn in XPS and EDS analysis of sample surface and into the crater, but not in the deepest layer of the depth profile.

5. Conclusions

The surface preparation technique for XPS analysis in depth with the grinding machine allows studying deposition profiles. But some side effects have to be taken into account and therefore, care should be taken in the analysis. It will be expected to have some mixing of the true depth profile and material removal that falls into the crater coming from the surface. However, the major advantage of this technique is that ones it has been developed and tested on non-contaminated specimens, Beryllium and Tritium contaminated JET samples can be studied in the future. In this case, the dimple grinding process must be carried out into a fume hood, and XPS/AES surface analysis can be made in order to determine

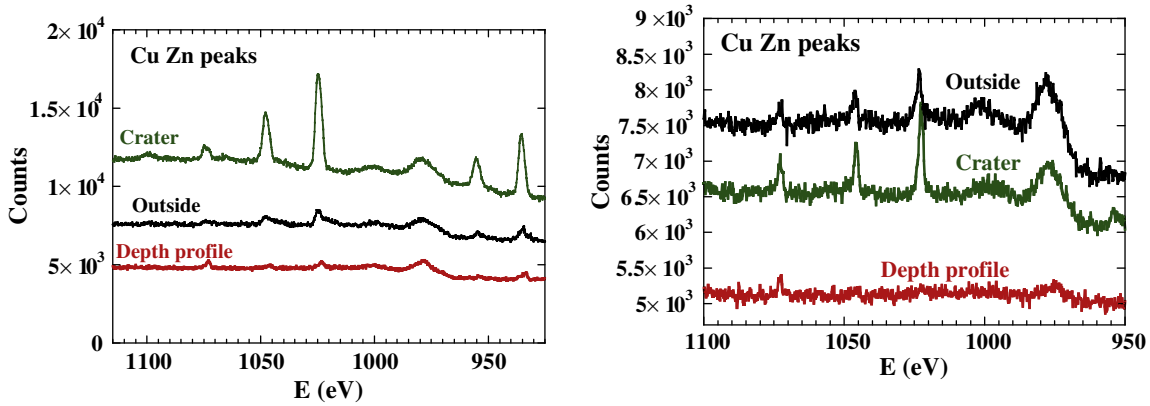

Fig. 6. XPS spectrum corresponding to Copper $2p^3$ (934 eV) and Zinc $2p^1$ (1045 eV) and $2p^3$ (1022 eV) peaks performed on the sample surface (outside), crater slope (crater) and after 3000 s of ion etching (Depth profile). Left: thick deposition specimen (tile 20), Right: thin deposition specimen (tile 21).

the chemical composition, chemical shift and the compositional depth profile without any sputtering process of toxic and/or activated materials.

Acknowledgments

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References

[1] J.P. Coad et al., Nucl. Fus. 46 (2006) 350.
[2] J.P. Coad, B. Farmery, Vacuum 45 (1994) 435.
[3] D. Whyte, J.P. Coad, P. Franzen, H. Maier, Nucl. Fus. 39 (1999) 1025.
[4] J.P. Coad, M. Rubel, C.H. Wu, J. Nucl. Mater. 241–243 (1997) 408.
[5] M. Rubel et al., Phys. Scr. T111 (2004) 112.
[6] J.P. Coad, P. Andrew, A.T. Peacock, Phys. Scr. T81 (1999) 7.
[7] J.P. Coad et al., J. Nucl. Mater. 290–293 (2001) 224.
[8] J.P. Coad et al., J. Nucl. Mater 313–316 (2003) 419.
[9] A. Kreter, A. Kirschner, P. Wienhold, J. Likonen, M. Mayer, V. Philipps, U. Samm, G. Sergienko, TEXTOR team, Phys. Scr. T128 (2007) 35.
[10] U. Samm, Fus. Sci. Technol. 47 (2005) 73.
[11] K.H. Finken et al., Fus. Sci. Technol. 47 (2005) 126.
[12] Private communication.