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Investigation on the erosion/deposition processes in the ITER-like Wall divertor at JET using Glow Discharge Optical Emission Spectrometry technique

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Abstract

As a complementary method to RBS, GDOES (Glow Discharge Optical Emission Spectrometry) was used to investigate the depth profiles of W, Mo, Be, O and C concentrations into marker coatings (CFC/Mo/W/Mo/W) and the substrate of divertor tiles up to a depth of about 100 μm. A number of 10 samples cored from particular areas of the divertor tiles were analyzed. The results presented in this paper are valid only for those areas and they can not be extrapolated to the entire tile.

Significant deposition of Be was measured on Tile 3 (near to the top), Tile 6 (at about 40 mm from the innermost edge) and especially on Tile 0 (HFGC). Preliminary experiments seem to indicate a penetration of Be through the pores and imperfections of CFC material up to a depth of 100 μm in some cases. No erosion and a thin layer of Be (< 1 μm) was detected on Tiles 4, 7 and 8. On Tile 1 no erosion was found at about 1/3 from bottom.

Keywords: Tungsten coatings, Glow Discharge Optical Emision Spectrometry, ITER-like Wall

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Introduction

Safe and reliable plasma operation in future fusion devices such as ITER requires good plasma performance with compatibility of the first wall and divertor materials [1]. ITER will start operation with a beryllium main chamber wall and a bulk tungsten divertor [2]. The ITER-like wall project at JET utilizes as the first fusion experiment the same material mix of plasma-facing components as ITER. It aims to investigate the plasma performance and the plasma-surface interaction processes (especially material erosion, transport, re-deposition, and fuel inventory) of this material configuration.

The current JET divertor with the ITER like wall (ILW) contains one row of bulk W lamellas (Tile 5) and W coated CFC tiles. The coating thicknesses are 10-15 μm for inner Tiles 3 and 4 and 20-25 μm for the outer Tiles 6, 7, 8 and Tile 1 (inner). A picture showing the JET divertor area and tile numbers is shown in Fig. 1. Erosion and deposition processes affecting these tiles are key issues for operation of a fusion reactor. In order to investigate the erosion/deposition processes on the divertor wall a number of CFC tiles were coated with a particular marker CFC/Mo/W/Mo/W multilayer structure and installed in different areas of the divertor. For all W coatings from ILW including the markers, the Combined Magnetron Sputtering and Ion Implantation (CMSII) deposition technology was used [3]. Rutherford Back Scattering (RBS) analysis and Secondary Ion Mass Spectrometry (SIMS) are currently being used to determine the erosion/deposition on marker coatings exposed to JET plasma [4]. RBS provides information on the total amount of W, Mo, Be and other elements into the marker coatings up to a depth of more than about 40 μm in light materials and more than about 10 μm in high-Z materials. SIMS has been used to measure depth profiles of elements. Main disadvantage of SIMS are the large matrix effects especially with beryllium, which render a quantification of SIMS results difficult. Due to the absence of matrix effects the GDOES method can be quantified easier and more reliably. GDOES shows the depth profiles of the elements up to about 100 μm. As a recent comparative study shows the GDOES performances are similar with those obtained by SIMS and NRA (Nuclear Reaction Analysis) [5]. In the present paper GDOES is being used to investigate the W/Mo coatings after their exposure to JET plasma.

The results of RBS and GDOES have to be correlated with SEM analyses of the investigated coatings in order to get the full picture of the coating/substrate structure and to understand its modification as a result of plasma exposure in JET.

Experimental

The GDOES system involves a high intensity glow discharge (GD) that is initiated between the sample surface as a cathode and an inner hollow anode. The light from the glow discharge source is transmitted to a concave holographic grating. A slit assembly is installed on the Rowland circle and behind the slits the light is recorded by a set of multipliers on specific wavelengths that are associated with the elements that can be analyzed. The material from the sample is sputtered layer by layer and it is removed by an Ar flow. A crater with a diameter of 4 mm is produced on the sample surface.

Depending on the application a specific method is defined. For the W coated CFC the following parameters were used: U_{GD}=1200 V, I_{GD}=15 mA, $p_{GD} \approx 3\cdot10^2$ Pa. These parameters were optimized with the aim to get the crater cross section as close as possible to a rectangular shape. The rim effect exists, but its influence on the results is not significant due to the relative large area of the crater (12.5 mm^2). The contribution of the rim effect can be estimated to about 2-3%. The sputtering rate is about 70 nm/s. With this method the machine was calibrated for W. Mo, Be, C, O and Ti using reference samples.

Normally, the GDOES technique cannot be applied for coatings deposited on carbon substrates (CFC or fine grain graphite) because the vacuum sealing cannot be achieved on their surfaces. In order to extend GDOES technique to porous surface (particularly W-coated CFC) a special device was used. The samples cored from the CFC tiles exposed in JET have a cylindrical shape with a diameter of 7 mm and a length of about 10 mm. On one base of this cylinder there is the W coating. The sample is mounted in the device and the vacuum sealing is achieved around the sample on the metallic surface of the device. The quality of the GD cut is good so the flank of the crater can be analyzed by SEM. In Fig.2 the flank and the bottom of the crater produced on a sample cored from Tile 3 can be partially seen. The image was taken at 45° in respect to the sample axis. One can see the carbon fibers parallel to the W coated surface (A), the carbon fibers perpendicular to this surface (B) and the felt between the fibers (C). The W/Mo coatings penetrate mainly along the perpendicular fibers, but through the felt as well up to 100 μm and even more. Traces of W can be seen on the crater bottom at 40 μm. The pores of the CFC can also be seen on the surface around the crater. At higher magnification the W/Mo coating can be seen on the flank of the GDOES crater. In this way a direct correlation between the GDOES depth profiles of Be, W, Mo and C and the real configuration of the coating/substrate interface can be done. The GDOES machine was supplied by Spectruma Analytik GmbH.

Results and discussion

A number of ten samples cored from Tiles 1, 3, 4, 6, 7, 8 and HFGC have been analyzed by GDOES after their exposure to JET plasmas in 2011-2012 campaigns. The location where the sample was cored from the tile is important since in some cases significant non-homogeneities have been detected by RBS on the same tile [4]. The tiles ID, the location of the cored samples and the main GDOES results are shown in Table 1. The position of the analyzed samples is also shown by arrows in Fig.1. Typical marker configuration was: CFC /3 μm Mo / 12 μm W / 4 μm Mo / 4 μm W. In the case of Tile 3 the outermost layer of W was not present. It should be pointed out that the Mo/W/Mo/W marker coatings were applied on divertor tiles perpendicular to fiber planes. As far as the HFGC tile is concerned it was coated with typical 10-15 μm of W with a Mo interlayer of 2-3 μm parallel to the fiber plane.

No erosion was detected on the investigated tiles in specific areas where the samples were cored from except Tile 3. A very thin layer of Be was measured on Tiles 1, 4, 7 and 8, while thicker deposits of Be were detected on Tiles 3 and 6. Typical GDOES depth profiles before and after plasma exposure are shown in Fig.3 for Tile 7. Unfortunately CFC samples coated in the same run with the real tiles installed in JET are not available so the comparison was performed between GDOES profiles obtained with Ti witness samples coated in the same run with the real JET tiles and CFC samples cored from the same tiles after their exposure in JET. As it can be seen, the profiles are quite similar except at the CFC/Mo interface where the irregularities of the CFC influence the Mo and W profiles. A sharp peak of Be appears at the surface followed by a fast drop in a few microns. Small Be "tails" like that shown in Fig. 3b were observed on Tiles 1, 4, 7 and 8. They can be explained by the roughness and porosity of the surface. Significant higher concentrations of Be (60 at.% and 75 at.%) were measured on Tiles 3 and 6. Be was detected up to a depth of 40-70 μm (Fig.4). As can be seen from Fig. 2 the CFC material shows a substantial open porosity at the surface, with dimensions of pores in the range of a few ten up to a few hundred μm and depths up to 250 μm. The interior of these open pores is coated with Mo and W during the coating process and with beryllium during plasma operation (see areas B in Fig. 2). It appears that to some extent even interior areas of open pores without direct line of sight to the exterior are being coated. However, more experiments are necessary to confirm this hypothesis. On the other hand the percentage of ~ 5 at.% of W at the surface indicate a low deposition of W together with Be. At the same time the decrease of the Mo concentration at the surface from 100 at.% to about 10 at.% could be associated with the local erosion of Mo. The SEM analyses confirmed the missing of Mo top layer in some areas.

A special attention was paid to the HFGC tile where a thick layer of Be (20-25 μm) was measured on the W coating in three locations. A picture of the HFGC tile in the ILW and the positions of the samples are shown in Fig.5. A dark zone covering about 70% of the total area of the HFGC tile can be clearly seen. The samples used for GDOES analyses were cut from the dark area. Apparently the dark area on HFGC in Fig. 5 could be interpreted as a strong erosion area where the W was removed from the CFC substrate. However, in accordance with the GDOES results shown in Fig. 6 this is an area with a strong Be deposition on W coating. Be but also W and Mo was detected up to a depth of 100 μm. This large depth does not represent the coating thickness, but it is due to the porosity, roughness and imperfections of the CFC material as it is shown in Fig.7. The pores dimensions are in the range of a few tens-hundreds of microns with the depth reaching sometimes 0.25 mm. This was demonstrated by X-ray micro-tomography [6]. The GDOES results are in agreement with those obtained by RBS on the same tiles [4].

Conclusions

GDOES is a reliable technique to investigate quantitatively the depth profiles of the constituents for surface layers deposited on metallic and carbon substrates before and after plasma exposure in a nuclear fusion device. By comparison the surface modification produced by plasma wall interaction can be identified and quantified. GDOES results have to be correlated with SEM analyses.

Beryllium appears to be present on all divertor tiles, but the quantity is different. For the particular areas of the tiles where the GDOES analyses were performed very thin layers (< 1 μm) of Be were detected on Tiles 1, 4, 7 and 8, thicker layers 3-8 μm have been measured on tiles 3 and 6 and very thick layer (20-25 μm) was measured on HFGC tile.

Erosion was not measured on any tile except tile 3 where the top Mo layer seems to be removed in certain areas.

The GDOES results are valid only for the area of the tiles where the samples were cored from and they cannot be extrapolated to the entire surface of the tiles due to the limited number of samples and poloidal asymmetries in the deposition pattern.

Acknowledgments

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Figure captions

Fig.1 JET divertor area and tiles identification; arrows show the position of the samples analyzed by GDOES

Fig. 2 GDOES crater with the depth of 40 μm on Tile 3; A-carbon fibers parallel to surface; B-carbon fibers perpendicular to surface; C-felt

Fig.3 GDOES depth profiles for the W/Mo marker deposited on Ti witness sample coated in the same run with Tile 7 (before plasma exposure) (a) and on sample cored from Tile 7 after plasma exposure (b)

Fig.4 GDOES depth profiles for the W/Mo marker deposited on Tile 3 after plasma exposure

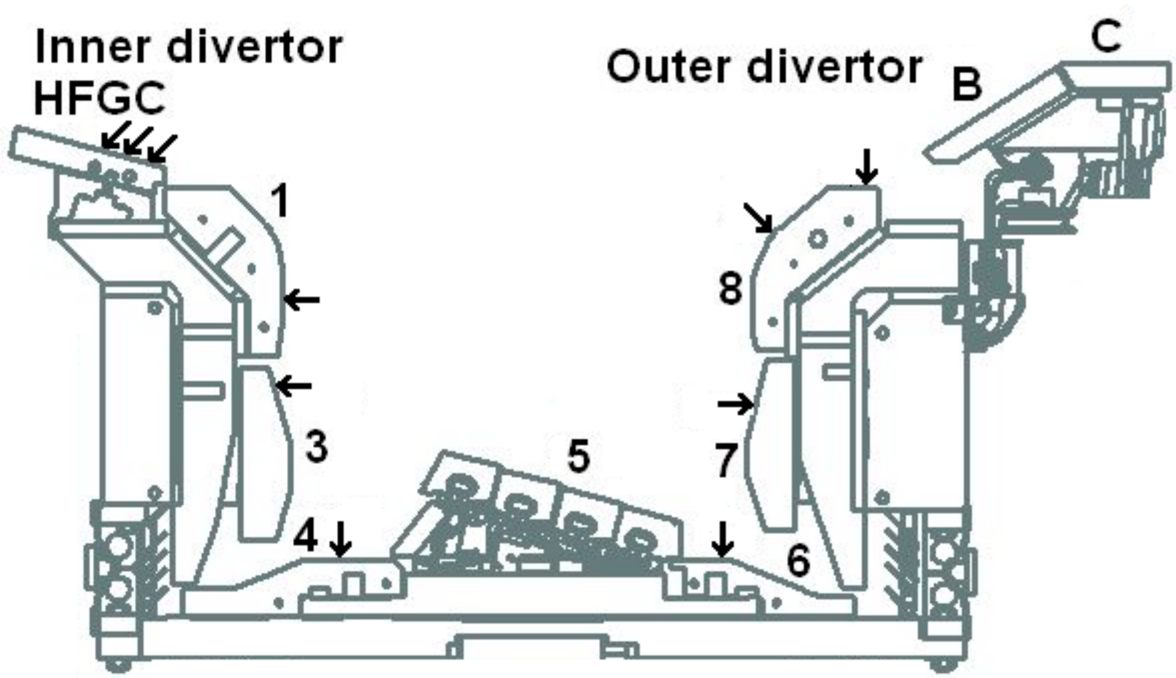
Fig.5 Tiles HFGC in JET wall

Fig.6 GDOES depth profiles for the W-coated CFC sample (3c) cut from HFGC tile exposed in JET

Fig.7 SEM image of the GDOES crater (flank and bottom) for a sample cored from HFGC tile

Table caption

Table 1 - GDOES results on the W coatings exposed to JET plasma



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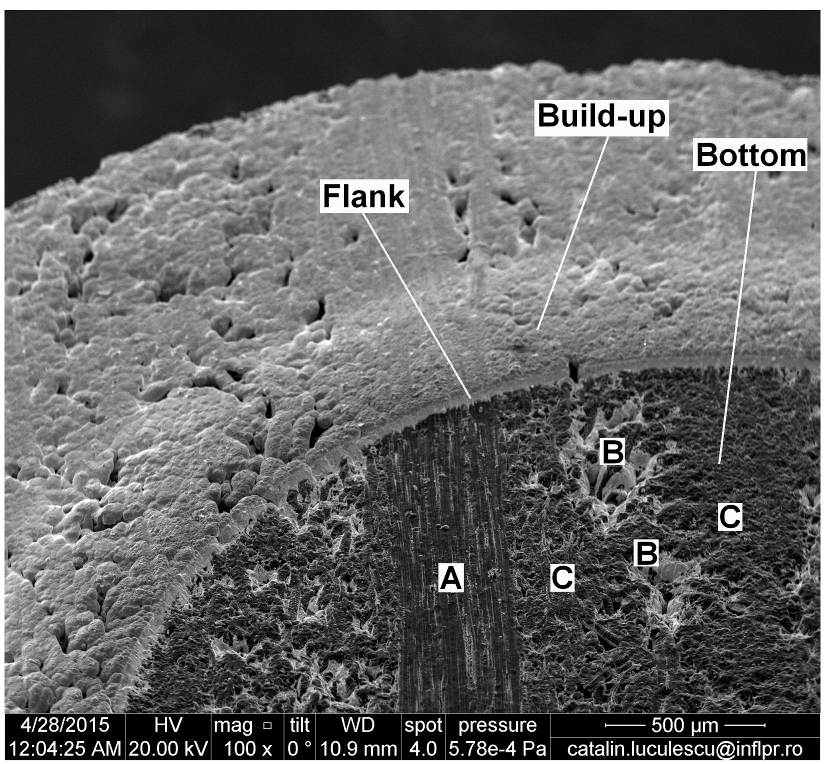

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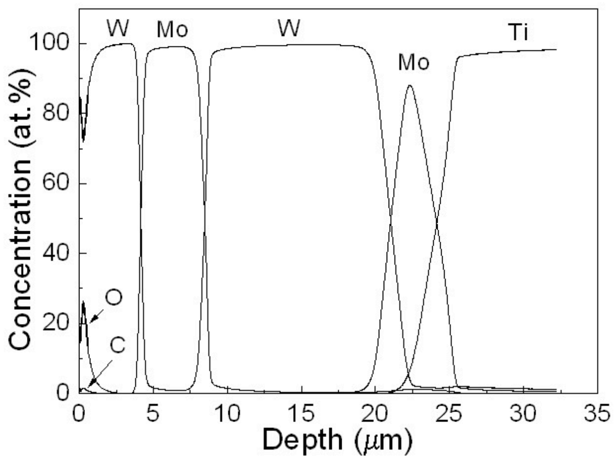
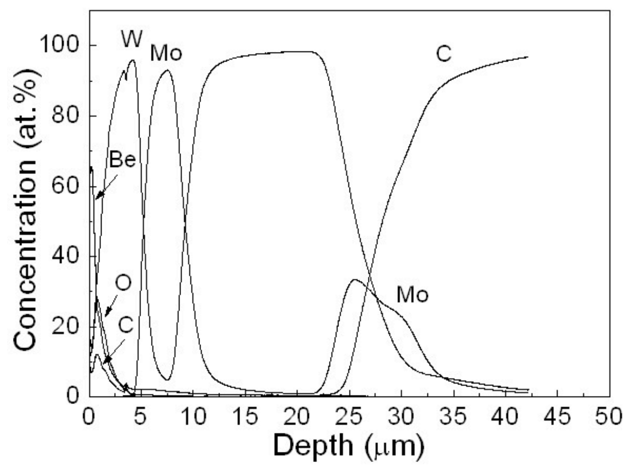


(a)



(b)

Fig.3 GDOES depth profiles for the W/Mo marker deposited on Ti witness sample coated in the same run with Tile 7 (before plasma exposure) (a) and on sample cored from Tile 7 after plasma exposure (b)

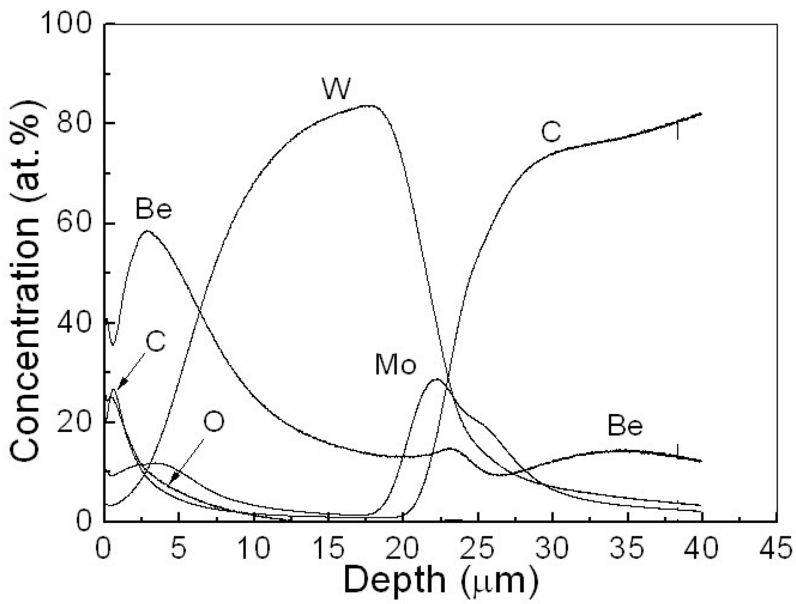


Fig.4 GDOES depth profiles for the W/Mo marker deposited on Tile 3 after plasma exposure

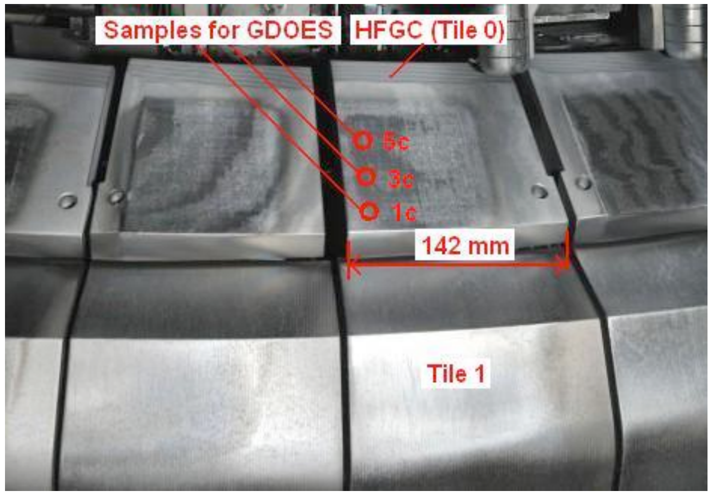


Fig.5 Tiles HFGC in JET wall

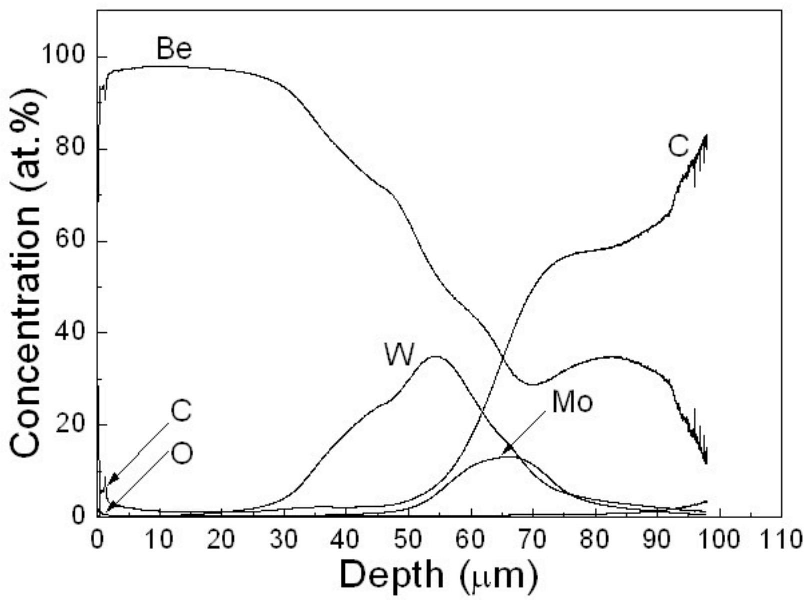


Fig. 6 GDOES depth profiles for the W-coated CFC sample (3c) cut from HFGC tile exposed in JET

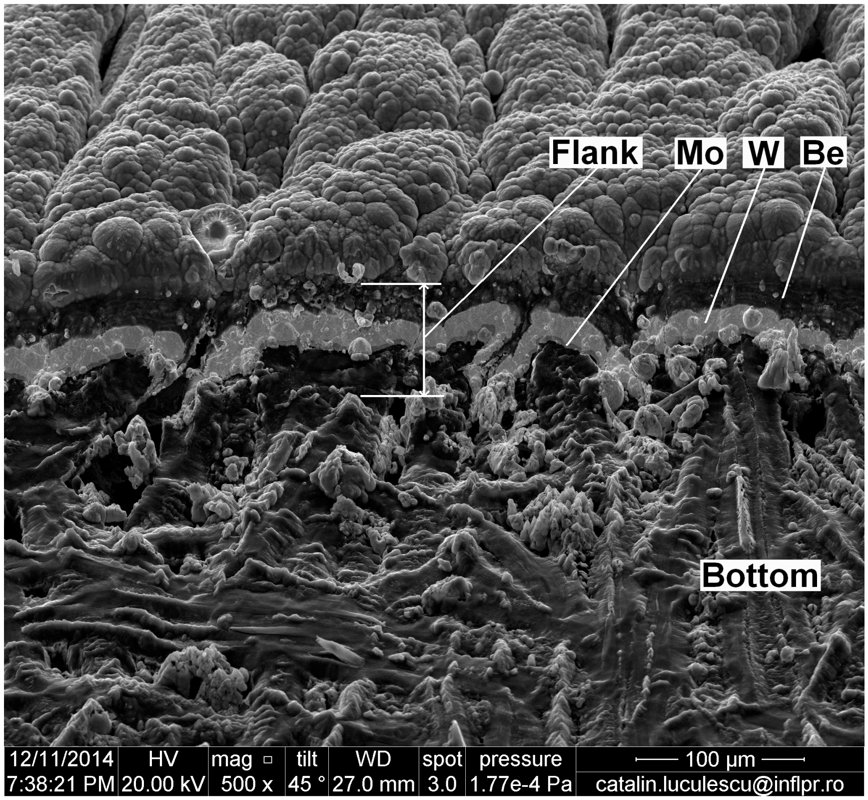


Fig.7 SEM image of the GDOES crater (flank and bottom) for a sample cored from HFGC tile

Table 1 - GDOES results on the W coatings exposed to JET plasma

Tile ID/ sample location	GDOES main results
Tile 0 (HFGC) 14IN-1c; 3c; 5c (see Fig.4)	- no erosion - massive deposition of Be (20-25 μm); Be penetrates the W/Mo layers and the substrate up to ~ 100 μm
Tile 1-14IN G1C at ~1/3 from bottom	- no erosion - thin layer of Be (<1 μm) at the surface; Be - up to ~ 15 μm
Tile 3-14IN G3B at ~15% from top	- Mo concentration at the surface is ~ 10 at.%; this might be associated with an erosion - significant deposition of Be at the surface (~ 3 μm); Be - up to more 40 μm - deposition of W (~ 5 at.%) on the Mo surface layer
Tile 4-14BN G4D at ~1/3 of the innermost edge	- no erosion - thin layer of Be (< 1 μm) at the surface; Be - up to ~ 15 μm
Tile 6-2BN G6C at ~1/3 of the innermost edge	- no erosion - significant deposition of Be (about 8-10 μm); deep penetration of Be into the marker and substrate up to more than 65 μm.
Tile 7-2ONG7A at ~1/3 from top	- no erosion - thin layer of Be (< 1 μm) at the surface; Be - up to ~ 12 μm
Tile 8-2ONG8B about in the middle	- no erosion - very thin deposition of Be (~0.2 μm); Be - up to ~ 10 μm
Tile 8-2ONG8B on the horizontal face	- no erosion - thin layer of Be (< 1 μm) at the surface; Be - up to ~ 26 μm