QUALIFICATION OF THE X-RAY MICRO-BEAM ABSORPTION/FLUORESCENCE METHOD FOR EROSION ANALYSIS

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1. Introduction

Currently, the primary materials choice for ITER (International Thermonuclear Experimental Reactor) is a full beryllium main wall with CFC at the strike points and tungsten at divertor baffles and dome. Since this combination has never been tested in a tokamak, ITER-like Wall project has been launched at JET, consisting of 10 μm W coating of approx. 1,000 CFC tiles.

In connection with this task, the main objectives of our project consist in developing of a non-destructive technique for the evaluation of the W coating uniformity as well as a quantitative mapping of the multilayer compositions and thicknesses.

During the reporting period a combined X-ray absorption/fluorescence method for erosion analysis was evaluated. Preliminary tests proved that the method is able to provide information about the uniformity of the CFC coating and can be used to determine its thickness. The preliminary experiments proved also that the lateral resolution is ~30 μm and the in-depth resolution is ~ 3% of the layer thickness. The work was continued by establishing a well defined and reliable experimental setup and by validating the technique on real-life samples from ASDEX-Upgrade.

2. Methods and results

The method for erosion analysis was implemented using the Tomo-Analytic system (Figure 1), which we developed especially for fusion materials analysis [1,2]. Tomo-Analytic is a combined X-ray fluorescence (XRF) and cone-beam tomography (3DCT) system for the noninvasive 3-D morphology and composition mapping. With its high space resolution, delivered by X-ray focalization with a policapillary lens, the XRF method permits the characterization of complex structures with lateral resolution of around 20 μm. The implementation of a confocal geometry realized with the attachment of a polycapillary conic collimator to the X-ray detector further allows the extension of capabilities of the instrument up to fluorescence tomography (3-D composition mapping). The XRF analysis with Fundamental Parameters (FP) converts elemental peak intensities to elemental concentrations and/or film thicknesses.

Overall performances
Microtomography

Spatial Resolution ≅ 20μm
Density Resolution > 1 %
Sample Dimensions: Diameter < 40 mm,
Height < 200 mm

Reconstruction time ≅ 5 min

Microfluorescence

Spatial Resolution ≅ 30μm

Thickness Resolution ≅ 2 % of total layer
Probe Dimensions: 100x100 mm2
http://tomography.inflpr.ro/

Figure 1 – View of Tomo-Analytic (left) and main technical characteristics (right)

The 3DCT component is configured to take highly resolved (48 μm) radiographic views of the object in order to build a 3-D model of its internal structure. 2-D slices through this volume can be viewed as images, or the 3-D volume may be rendered, sliced, and measured directly. For the NDT inspection of miniaturized samples the microtomography analysis is guaranteed for feature recognition better than 15 μm. 3-D tomographic reconstructions are obtained by a proprietary highly optimized computer code based on a modified Feldkamp algorithm.
The problem of evaluating the thickness uniformity of graphite/CFC with metallic coatings for a large number of samples was addressed also in case of the X-ray fluorescence analysis. In order to reduce the measuring time we replaced the multichannel analyzer (MCA) of the Tomo-Analytic system - Amptek MCA8000A - with a new one FAST MCA-3Series. The new MCA ensures a much faster and more flexible data transfer of spectral data to the PC and allows an improved control of the acquisition process. The MCA-3 Series is a family of PC-based software controlled PCI-bus Multichannel Analyzers. The design is capable to convert incoming signals at up-to 10^6 events/s or collect data at rates of up-to 5 Megaevents/s. The on board ultra fast pulse height analyzing 8k ADC is characterized by a 500 ns conversion time for Pulse-Height Analysis. No dead-time between channels, no end-of-sweep dead-time are also extremely valuable characteristics of the MCA. The dwell time extends from 100 ns to 50s (200ns to 50s using two inputs). The large data memory can be segmented to enable to accumulate successive measurements. Spectra accumulated in sequential PHA mode can be displayed in a two-dimensional array. The operating software allows the integration in the Tomo-Analytic application which was developed on a combined Labview/C++ platform.

2.1 Microbeam fluorescence benchmark

A number of experiments were performed in order to determine the parameters of the system and its usefulness for erosion analysis. The spatial resolution of the micro-beam fluorescence component was determined using a Cu grid (Figure 2.a). The double grid structure (32 μm and 18μm, respectively) was scanned along a line, using a step of 10 μm and the following X-ray tube parameters: HV = 40 KV, I = 400 mA. At each step the X-ray spectrum was recorded and the evolution of the Cu Kα = 8.06 keV peaks areas was recorded along the scanning line. Figure 2.b demonstrates the ability of the system to recognize the thick grid, so a spatial resolution of ~30 μm is guaranteed.

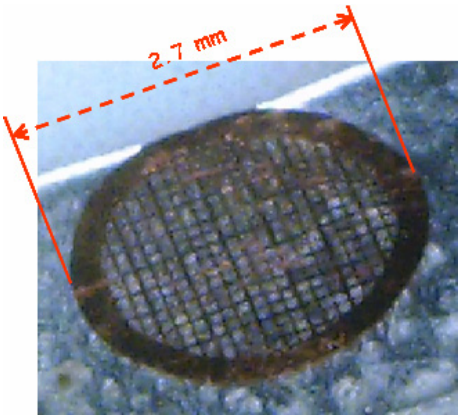


Figure 2.a – Cu grid used for spatial resolution determination. Grid characteristics: border thickness = 260 µm, thick grid = 32 µm, thin grid = 18µm.

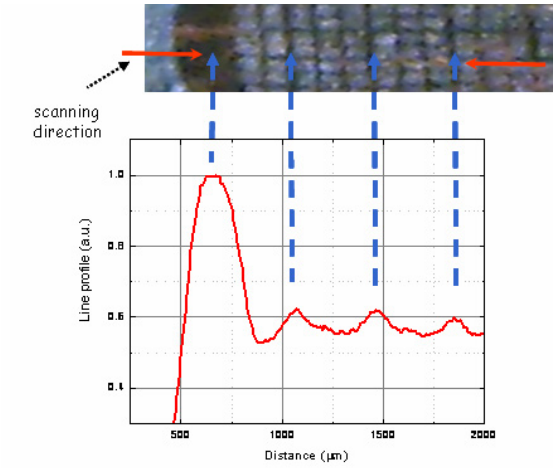


Figure 2.b – and the evolution of the Kα= 8.0 keV peaks areas along the scanning line

The possibility to determine the thickness of a coated layer was tested using a test sample with 10 µm Cu on Fe substrate layer (Figure 3). The coating was produced by combined magnetron sputtering and ion implantation technology, developed at INFLPR [4].

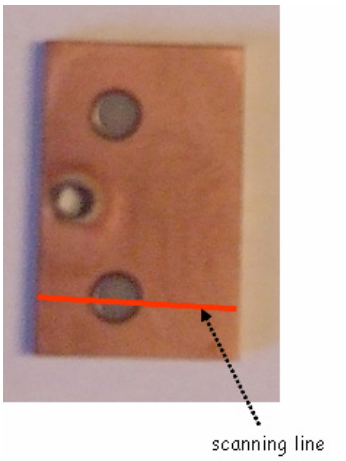


Figure 3 – Test sample for thickness determination: 10 µm Cu on Fe substrate layer.

Erosion circular patterns were produced by a glow discharge optical spectrometry (GDOS) device.

A full width line was scanned using a step of 0.1 mm and the following X-ray tube parameters: HV = 40 KV, I = 400 mA. The stack of spectra is presented in Figure 4. Both 3D and 2D representations demonstrate the ability of the system to map the composition of the sample. It can be observed that Fe peaks are visible even in the regions where Cu coating exists. This is also revealed by the evolution of Cu/Fe peak areas along the scanning line (Figure 4 bottom-left panel)) - the peak areas of Cu (K_{α} = 8.06 keV) and Fe ($K_{\alpha,\beta}$ = 6.4/7.1 keV) were recorded at each scanning step.

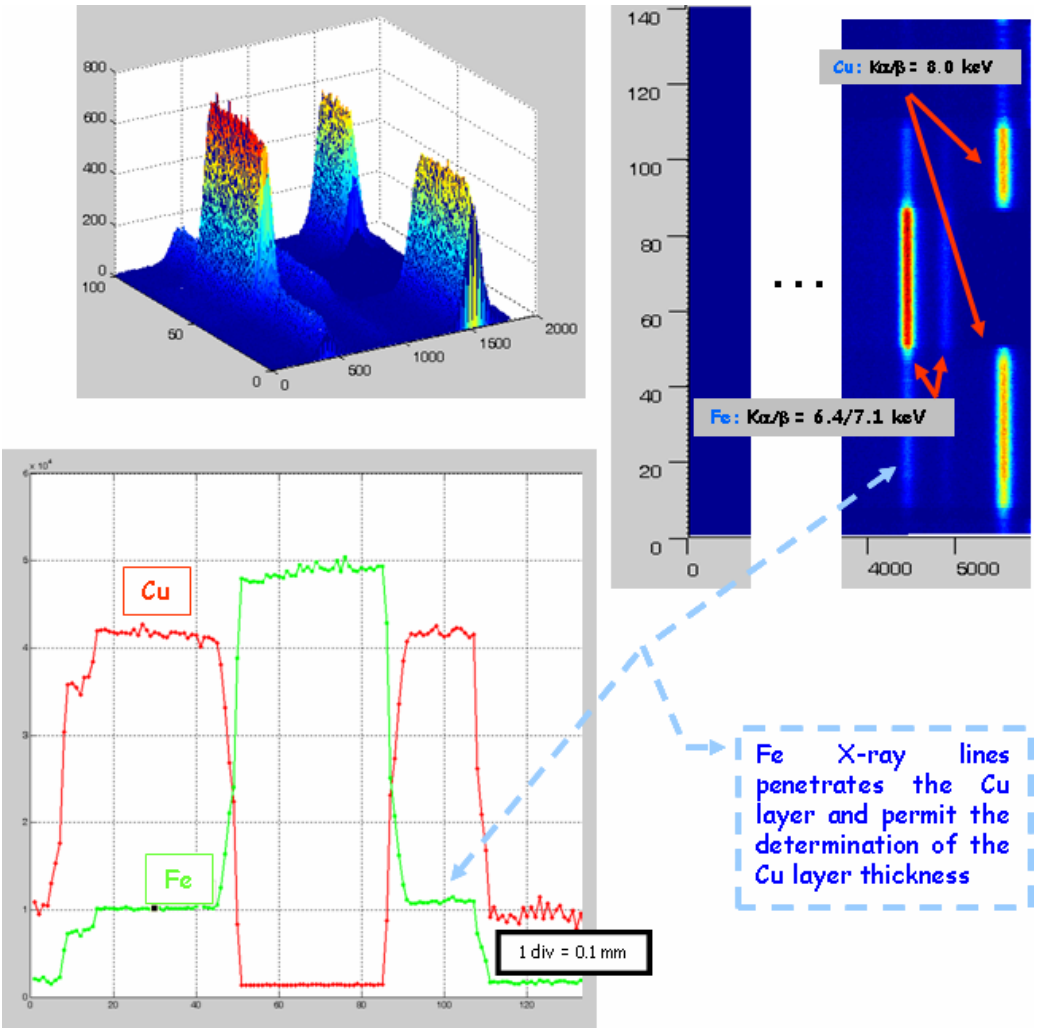


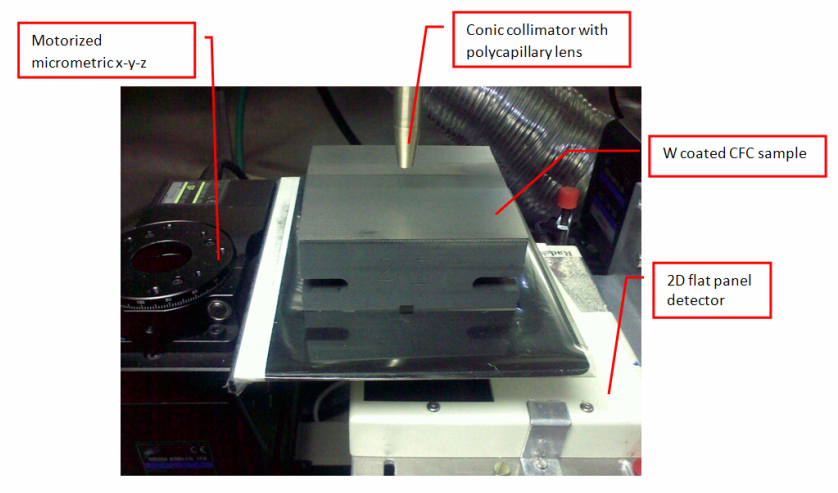
Figure 4 – Results of the fluorescence mapping of a coating layer test sample (10 µm Cu on Fe substrate layer): 3D representation of the stack of fluorescence spectra (top-left), 2D representation of the stack of fluorescence spectra (top-right) and evolution of Cu and Fe $K_{\alpha,\beta}$ peak areas along the scanning line.

As the energy of Fe X-ray fluorescence lines are known, together with the attenuation parameters of the X-ray in Cu, the thickness of the Cu layer can be determined. The accuracy of this determination was ~ 5%.

2.2 Coating thickness of carbon samples

The same method can be also applied for the determination of the coating thickness of CFC samples, but not in a straightforward manner. The micro-beam fluorescence device is not operated in vacuum but in air and the carbon X-ray lines energy is below 1 keV. In consequence the penetration of C X-ray lines through the coating cannot be used for thickness calculation. However, usually, the coating process makes use of an intermediate layer; for example Mo is used as an interface layer between C substrate and the W coating layer. Therefore the Mo X-ray lines penetrating the W layer can be used to determine its thickness. The maximum thickness of W layer for which this technique remains sensitive enough is limited by the energy of the Mo X-ray lines.

For the case of the assessment of the thickness uniformity of graphite/CFC with metallic coatings for a large number of samples, we found that an X-ray transmission technique is a more pragmatic solution in comparison to the X-ray fluorescence analysis. As the Tomo-Analytic system is a configurable and versatile measuring tool, we modified the geometry of the system (Figure 5). The X-ray source has a direction of emission perpendicular to the flat panel detector. The X-rays are detected after passing through the investigated sample where they are attenuated accordingly with the composition and thickness of the materials. The optimal measurement configuration and irradiation parameters were obtained by MCNP-5 Monte Carlo simulations [4].



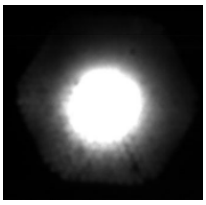
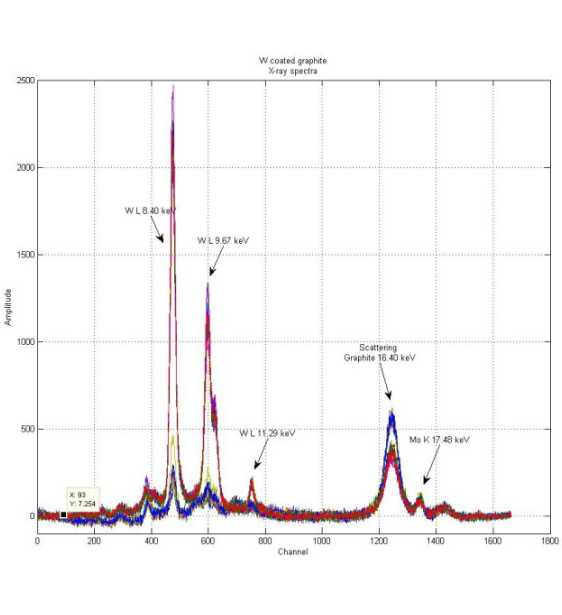


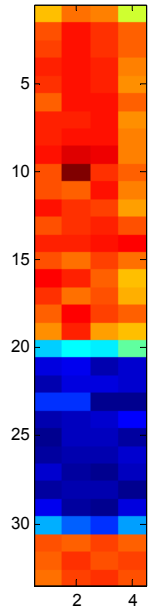
Image of the X-ray source on the flat panel detector

Figure 5 – View of the X-ray transmission geometry

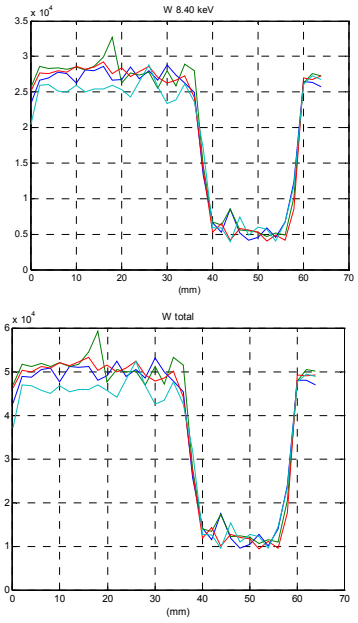
Typical results of the combined X-ray transmission and X-ray fluorescence analysis are presented in Figures 6. a,b.



X-ray fluorescence spectra of a W coated fine graphite tile from ASDEX-Upgrade



Map of the tungsten layer thickness scanning step: 2 mm

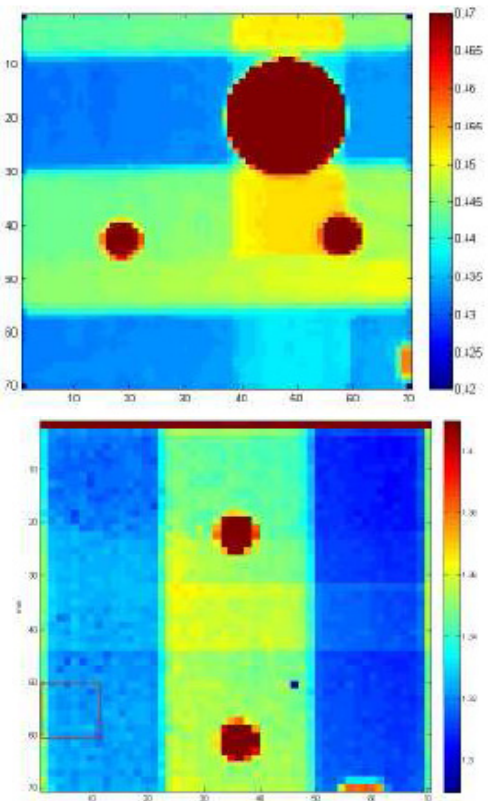


Line profiles along the map

Figure 6.a - Post-mortem analysis of W coated fine graphite tiles from the divertor of ASDEX-Upgrade by X-ray Microbeam fluorescence

The X-ray transmission map can be used for the determination of the absolute value of the thickness of the W coating layer. In order to remove the influence of the fine graphite layer, a calibration sample must be used. The calibration sample is a multi-step one, which has regions with different thickness of fine graphite. As the real tiles contain several cutting up profiles, the CAD model of the tile must be also taken into account. The X-ray transmission ensures fast and high resolution analysis. The images presented in Figure 6.b are obtained for a scanning time of approximately four hours. The 70 x70 pixels images are obtained for a resolution of 1.0 mm/pixel. The main advantages of the X-ray fluorescence method are: i) it doesn't need a calibration sample for the determination of the thickness of the coating layer and ii) it can detect the composition and thickness of possible deposited layers of intrusions. However the time needed for the inspection of the sample in considerably larger: approximately 60-90 s are needed for the acquisition of a spectrum. Therefore a spatial resolution equal with that one obtained in the case of the X-ray transmission method becomes prohibitive regarding the acquisition time needed. Also, due to the attenuation of the low energy of the emitted X-ray lines, the fluorescence method is limited to thin coating layers (up to ~10 μm) depending on the coating material. However the combined use of X-ray transmission and X-ray fluorescence methods represent a unique instrument for the post-mortem analysis of the coatings.

Graphite tiles with W coating



X-ray transmission map. The X-ray transmitted intensity was normalized to an average value of the transmission through the graphite. In this way we ignored the very low amount of redeposited W.

Figure 6.b – High resolution post-mortem analysis of W coated fine graphite tiles from the divertor of ASDEX-Upgrade by X-ray Microbeam transmission technique

3. Conclusion

An evaluation of the combined X-ray absorption/fluorescence method for erosion analysis was performed. Individually, each method has its own advantages/disadvantages, but the combined use of X-ray transmission and X-ray fluorescence methods represents a unique instrument for the post-mortem analysis of the coatings. It can provide fast analysis, high spatial resolution and detection of deposited layers and intrusions. The combined method was validated on W coated fine graphite tiles from the divertor of ASDEX-Upgrade.

The work will continue with X-ray fluorescence and tomography coating evaluation for a set of ASDEX-Upgrade tiles and of ITER-like CFC tiles. Marker probes of Al C Ni W will be also measured. A comparison to previous quantitative analysis with EPMA, RBS and NRA techniques will be carried out. Finally, a technical concept for a compact/low cost instrument based on X-ray micro-fluorescence to be used in high productivity coating analysis will be elaborated.

4. Acknowledgement

The research activities to be conducted in this project are relevant for the EFDA Task Agreement TA-4 “Erosion, transport and deposition of wall materials” organized under the Special Expert Working Group (SEWG) “Material Migration".

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