EUROfusion

EUROFUSION CP(15)04/12

E. Grigore et al.

Thermo-Mechanical Properties of W/Mo Markers Coatings Deposited on Bulk W

(18th May 2015 – 22nd May 2015)
Aix-en-Provence, France

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Thermo-mechanical properties of W/Mo markers coatings deposited on bulk W

E Grigore[1], C Ruset[1], M Gherendi[1],D Chioibasu[1], A Hakola[2], and JET contributors[3]

[1] *National Institute for Laser, Plasma and Radiation Physics, Bucharest, Romania*
[2]*VTT Technical Research Centre of Finland Ltd, P.O.Box 1000, FI-02044 VTT, Finland*
[3]*EUROfusion Consortium, JET, Culham Science Centre, Abingdon, OX14 3DB, UK (See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference 2014, Saint Petersburg, Russia)*

In the present paper marker structures consisting of W/Mo layers were deposited on bulk W samples by using a modified CMSII method (CMSII-M). This technology, compared to standard CMSII, prevents the formation of nano-pore structures at interfaces. The thicknesses of the markers were in the range 20-35 μm to balance the requirements associated with the wall erosion in ITER and thermo-mechanical performances. The coatings structure and composition were evaluated by GDOES (Glow Discharge Optical Emission Spectrometry), and EDX (Energy Dispersive X-ray spectroscopy) measurements. The adhesion of the coatings to the substrate has been assessed by scratch test method. In order to evaluate their effectiveness as potential markers for fusion applications, the marker coatings have been tested in an electron beam facility at a temperature of 1000°C and a power density of about 3 MW/m^2. A number of 300 pulses with duration of 420 s (35 testing hours) were applied on the marker coated samples.

Keywords: W coatings, High heat flux test, W markers, ITER divertor

INTRODUCTION

During the JET campaign August 2011-July 2012 it has been demonstrated that the ITER-divertor (ILW) configuration meet and exceeded the expectations concerning power and energy handling capabilities [1,2]. As far as the erosion phenomena is concern, it has been observed that new ILW lead to a reduction of the carbon content impurity by more than a factor of ten [3]. On the other hand the dust collected during JET-ILW campaign was about 0.7 g and 0.3 g compared with 137 g and 51g from inner and outer divertor, for the same surface area compared with JET –C campaign 2008-2009 [4]. These achievements confirmed the capabilities of PFC coated with W coatings. However the assessment of the erosion phenomena still represents an issue that needs a special attention.

Multilayer marker coatings are considered to be a very useful tool in evaluating erosion/deposition for the first wall in a plasma fusion device. The markers have a well defined structure (composition and thickness) and are deposited on specific wall tiles installed in different locations in a fusion device. By analyzing their configuration before and after plasma exposure, a map of erosion/deposition processes can be obtained. Combined Magnetron Sputtering and Ion Implantation technique (CMSII) [5] was successfully used to coat CFC (Carbon Fibre Composite) tiles for ILW including markers [6]. Tungsten/molybdenum marker coatings are currently used on specific CFC tiles and bulk W lamellas in the ITER-like Wall divertor at JET. The total thickness of ILW W/Mo markers was in the range of 12-24 μm depending on their location and substrate. They behaved very well during the experimental campaigns (2011-2012). The characterization of the markers after a campaign and the comparative analysis with initial marker characteristics will help in defining critical areas of the wall and will lead to an understanding concerning the erosion and deposition patterns.

EXPERIMENTAL

The coatings have been produced by a CMSII-M technology with pores free interfaces. In the first step of the experiments W/Mo coatings with thickness of 20 μm have been manufactured and characterized. These coatings represented the starting point in the attempt to develop thick marker coatings. After the characterization of these samples, the next step was the increasing the thickness of the coating up to 35 μm. Samples made of bulk W (30x30x3 mm^3), Ti (15x25x3 mm^3) and FGG (Fine Grain Graphite) (30x30x6

mm^3) have been used as substrates. Prior the deposition the substrates were polished on SiC paper (grade 360) and then, the roughness of the surface was measured by using a Tribotechnique profilometer. The surface roughness parameters of the samples were Ra= 0.57 ± 0.04 μm and Rz= 5.32 ± 0.037μm. The Ti and FGG samples have been coated in the same run with the bulk W samples having the role of witness samples. These samples were used to assess some characteristics of the coatings. The coatings layout consisted of a Mo layer of ~7μm and a top W layer of ~ 13 μm and ~ 28 μm respectively.

The chemical composition of the coatings has been determined by GDOES analysis using a Spectruma GDA 750 equipment. SEM investigations have been performed in order to investigate the structure and the morphology of the coatings.

One of the main requirements of the coatings used in fusion applications is related to capability to withstand high cyclic thermal loads. In order to check the performance of markers, a high heat flux testing (HHFT) program has been applied. The samples were heated using an electron beam test facility at National Institute for Laser, Plasma and Radiation Physics (IAP - Romanian Eurofusion Research Unit). Details concerning the experimental setup can be found elsewhere [7]. The cycle consisted in heating up the samples to the testing temperature, a steady state representing the pulse duration and a cooling down to a base temperature, below ductile to brittle transition temperature.

The testing temperature was 1000 ° C, the pulse duration was 420 s, the inter-pulse duration was 60 s and the base temperature was 230 °C. The temperature of the surface was measured by using an IMPAC IR Pyrometer operating in the wavelength range 1.45 – 1.80 μm.

The adhesion of the markers has been determined by using a ST 30 scratch tester supplied by Teer Coatings Ltd. The test consists in scratching the surface of the coatings with a diamond tip with a radius of 0.2 mm. The applied load increases progressively from 0 to 100 N over a distance of 10 mm. Subsequent optical inspection of the scratch track indicates regions where failure of the coatings occurs. This corresponds to the critical load, the parameter used to asses the adhesion. The failure can be represented by delamination or spalling of the coating at the edges of the scratch trace. In our work the maximum applied load was 90 N.

RESULTS AND DISCUSSION

The surface roughness of the as deposited coatings indicated the following values: Ra=0.20 ±0.015 μm and Rz=1.72 ± 0.07 μm. It can be noticed an improvement concerning the surface roughness, the surface of the coated samples are smoother compared with the initial roughness of the substrates.

GDOES depth profile measurements have been performed on Ti witness samples. In Fig. 1 are presented the profile concentrations of the elements of interest. At the base of the W coating a Mo layer of ~7 μm can be observed. The thickness of W layer is 13.7 μm for 20 μm marker coating whereas for the 35 μm marker coating the W layer has 28.1 μm.

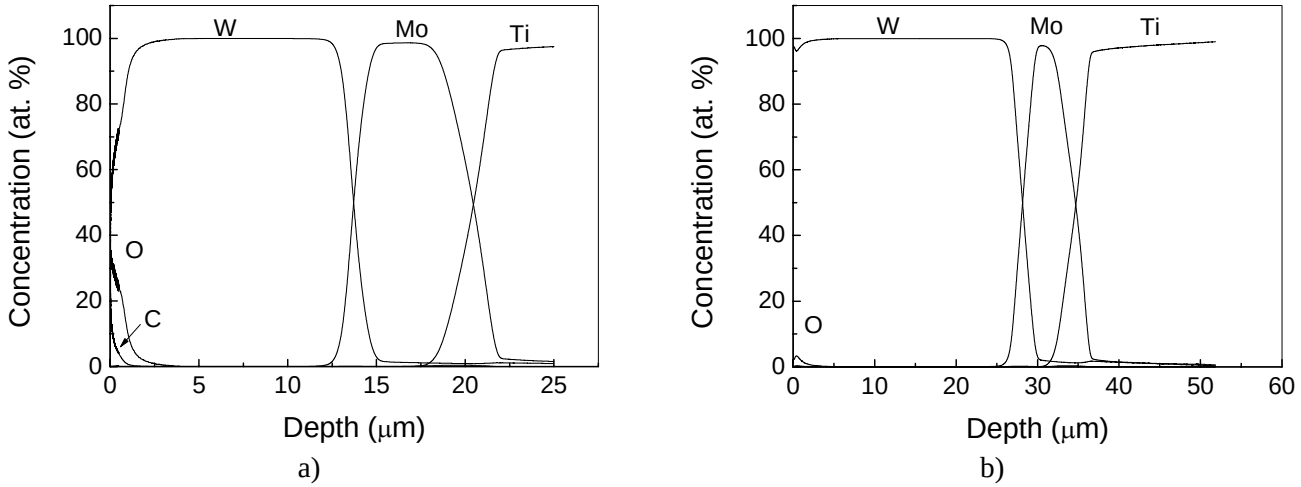

Fig. 1 *GDOES* depth profile for W/Mo markers deposited on Ti witness sample (*a*- 20 μm marker ; *b*- 35 μm marker)

SEM investigations have been performed on markers deposited on FGG substrate. The measurements confirmed the thickness of the coatings determined by GDOES. The SEM images indicated a columnar structure of the layers and a good coherence between the Mo and W layers. An image of the W/Mo marker of 35 μm is presented in Fig. 2.

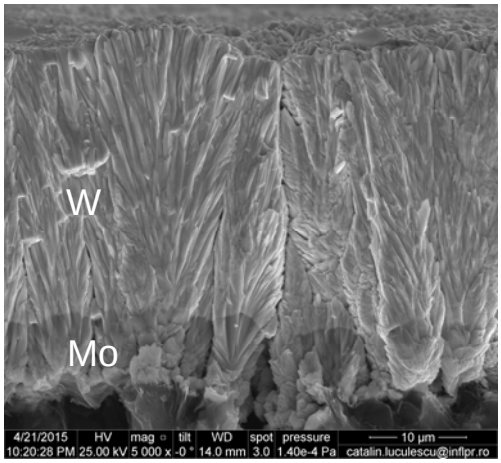

Fig. 2 *SEM* image of a W/Mo marker deposited on FGG substrate

The HHFT program involved a periodical inspection of the surface of the coatings after a certain number of pulses. The defects (buckling, delaminations, melting spots or other defects) appeared on the surface were counted and then the testing program was resumed. In this way it was possible to draw a degradation curve of the surface as a function of the applied pulse numbers.

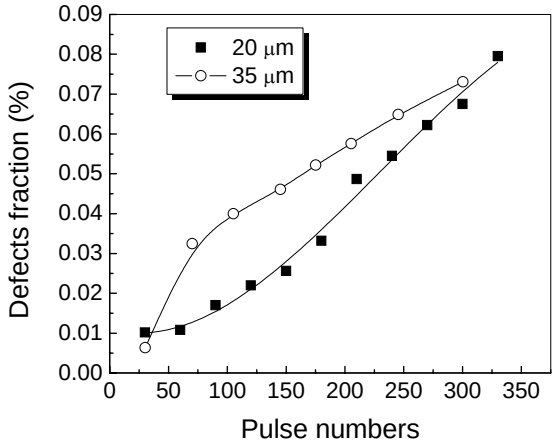

Fig. 3 The evolution of defects fraction as a function of pulse numbers (testing temperature 1000^0 C)

It can be observed that after 35 hours at the testing temperature on the surface of the 20 μm marker a percent of 0.067 % is affected by defects. At the same pulse numbers the 35 μm marker indicated a percent of 0.073%. The difference between the defects fractions of the two types of markers is not significant at the end of the tests. However it can be observed that the defects fraction for 35 μm W/Mo marker are higher than the 20 μm W/Mo marker at the first series of pulses. From the Fig. 3 it can be concluded that the thickness doesn't have a major contribution on the defects fraction resulted on the surface when the testing temperature is 1000^0 C. What is worth to mention is the type of the defects resulted after HHFT. Compared with other substrate material (CFC or FGG) tested before [5], when the failure of the coating was mainly due to buckling and delaminations, in the case of W/Mo markers spallation was the main mechanism. Small fragments (chips) are removed from the surface of the coating caused most presumably by thermal stress and

thermal fatigue phenomena. An image with such defect is shown in Fig. 4. This type of defect has a shallow penetration depth and consequently remains within the W layer.

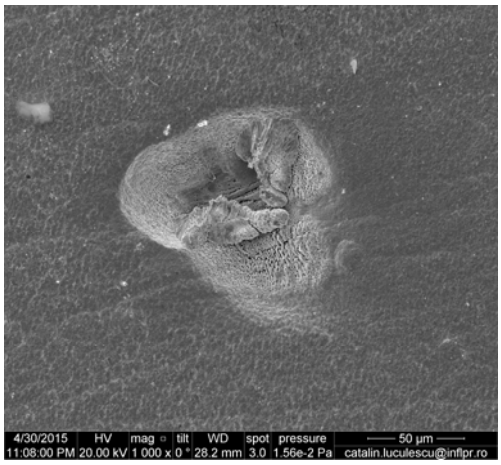

Fig. 4 *SEM* image of a typical defect after HHFT

As far it concerns the dimensions of the defects they are quite similar, for both types of markers the average defects size is less than $9x10^{-3}$ mm^2. For 35µm W/Mo marker typical dimensions of the defects are in the range $0.25x10^{-3} \div 4.37x10^{-3}$ mm^2. The small defects prevail comparatively with the larger ones, and from the cumulative frequency distribution it has been observed that almost 93% of the total number of defects have dimension less than 0.025 mm^2.

The adhesion of the markers has been determined after the HHFT program. An image with the end of the scratch trace can be observed in the Fig. 5. It can be seen that the edges of the scratch track are very smooth with no delaminations or spallations. However at the bottom of the scratch trace it can be observed some characteristics specific to a plastic deformation of the W layer. Under these circumstances the critical load was greater than 90 N and thus a very good adhesion of the marker to the substrate still exists after the HHFT. The EDX analysis performed at the end of scratching trace indicated that the scratch didn't penetrate the W layer (no Mo has been identified). The profile concentrations for C, O, Mo and W across the scratch trace are superposed over the SEM image. These profiles have been measured across the white line and are indicated in Fig. 5.

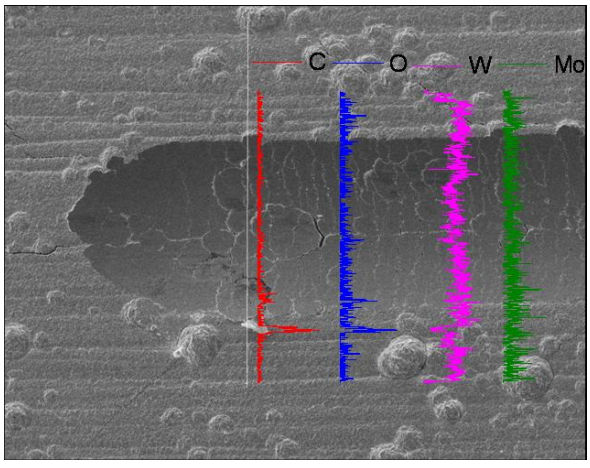

Fig.5 SEM image with EDX analysis performed at the end of the scratch trace (90N load)

CONCLUSION

- W/Mo markers with thickness of 20 μm and 35 μm have been deposited on bulk W substrate. SEM investigations indicated a columnar structure of the layers.
- Both 20 μm and 35 μm marker coatings survived the HHF tests consisted of 300 pulses with duration of 420 s and a surface temperature of 1000 °C. After a cycling time of 35 hours the damaged fraction of the thermal loaded is about 0.07% that is insignificant. These types of markers can be used for quantitative determination of the erosion/deposition in fusion devices.
- The adhesion tests performed after HHFT indicated that the adhesion remains very good even after cyclic thermal loading of the markers.

Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium, and has received funding from the Euratom research and training program 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

The work has been also supported by the CEA-RO Program Code: C4-07.

References:

[1]. L. Horton, Fusion Eng. and Design 88,(2013), 434-439
[2]. G.F. Matthews et al, Journal of Nuclear Materials, 438, (2013), S2–S10
[3]. S. Brezinsek et al, Journal of Nuclear Materials, 438, (2013), S303–S308
[4]. A. Widdowson et al, Phys. Scr. T159, (2014), 014010
[5]. C. Ruset et al, Phys. Scripta, T128 (2007), 171-174
[6]. C. Ruset et al, Fusion Eng. and Design, 86, (2011), 1677-1680
[7]. C. Ruset, et al, Fusion Eng. and Design, 88, (2013), 9-10, 1690- 1693