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** See annex of F. Romanelli et al, "Overview of JET Results", (24th IAEA Fusion Energy Conference, San Diego, USA (2012)).*

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ABSTRACT

Combined Magnetron Sputtering and Ion Implantation (CMSII) technology was used for W coating of about 1800 CFC tiles for ITER-like Wall (ILW) at JET and more than 1300 fine grain graphite tiles for ASDEX Upgrade tokamak. Thermal fatigue and carbidization due to the diffusion of the carbon from substrate were found as main phenomena limiting the service lifetime of the W coatings. Detailed investigation of these coatings using SEM and STEM together with the FIB (Focused Ion Beam) cutting technique revealed two fine networks of nano-pores with the size of 10–20nm for zones of 250–350nm at CFC-Mo and Mo-W interfaces. Formation of these nano-pores is associated with the energy of the ions striking the coating during the deposition process. By optimizing this energy the nano-pores disappeared. The structure and the performances of the W coatings produced by the new modified technology (CMSII-M) were compared with those deposited by standard benchmark technology. The HHF (High Heat Flux) tests carried out with an electron beam at a temperature of 1450°C indicated an improvement of the thermo-mechanical properties by a factor of about 1.7.

1. INTRODUCTION

The actual configuration of the ILW consists of bulk Be and W coated CFC tiles for the main chamber whereas the divertor sections are made of W coated CFC and one row of bulk W. W coatings have been deposited by standard, benchmark CMSII technology [1]. The coating thickness is in the range of 10–15µm and 20–25µm depending on the location of tiles in the wall. A Mo interlayer of 2–3µm was introduced for all coatings between the W layer and substrate in order to improve the adhesion. The CMSII coating technology was qualified by HHF tests in the hydrogen beam facility GLADIS at the Max-Planck Institute for Plasma Physics, Garching, Germany [2]. The impact of thermal fatigue and carbidization on the thermo-mechanical properties of these coatings was investigated as well [3]. During that research, nano-pores structures were revealed at CFC-Mo and Mo-W interfaces for regions of 350nm and 250nm respectively. The HHF tests indicated that the dimensions of the pores increase significantly depending on the peak temperature and on the number of heating pulses. Finally this leads to destroying of the coating.

A significant research effort has been carried out with the aim to establish a correlation between the coating parameters and the formation of the nano-pores structures. A new coating technology (CMSII-M) able to produce pores free interfaces was developed.

In this paper new results concerning the structure and performances of the W coatings deposited by CMSII and CMSII-M technologies are presented.

2. EXPERIMENTAL SETUP

Four samples (30 × 30 × 6mm^3), made of Dunlop DMS 780 CFC material, were W-coated perpendicular to fiber planes using CMSII and CMSII-M technologies. A special technology (CMSII-Gr) was used to produce a gradual interface from pure Mo to pure W in 3 steps. A detailed description of the deposition method (CMSII) and equipment can be found elsewhere [1].

The HHF tests have been performed using the electron beam High Temperature Test Facility (HTTF) at National Institute for Laser, Plasma and Radiation Physics – Romanian Euratom Association [3]. The main operating parameters of the electron beam were: high voltage 17–19kV, beam current 70–80mA, pulse duration 25 ± 2 s and interpulse duration 35–45s. The thermal loaded area of the sample was estimated to 4.5cm^2. After a number of testing cycles the samples surface was inspected by means of an optical stereomicroscope in order to estimate the delaminated area. After this operation the HHT was resumed. In order to get relevant information about the HHF resistance of the coatings, a large number of heating-cooling cycles (about 3000) were applied to the coatings.

The surface temperature was measured by an IMPAC IGA-5 pyrometer operating in the wavelength 1.45 ÷ 1.8μm. A value of 0.5 for the W coating emissivity was used to measure the temperature.

Coating thickness and the main testing conditions are shown in Table 1.

The following designations have been used: d represents the coating thickness (μm); T_{peak} represents the maximum test temperature measured by the pyrometer (°C) and P is the power density on the W coating estimated by calorimetric method (MW/m^2).

The changes of coatings structure produced by thermal fatigue and carbidization phenomena were investigated using a dual beam FIB/SEM device (Hitachi NB5000) equipped with 40kV ion beam source and 30kV Schottky field emission electron source. FIB/SEM equipment was used for two purposes - preparation of thin lamellas for STEM observations and production of cross-sections for SEM observations.

High resolution microstructure observations were performed on dedicated STEM equipped with 200kV Schottky field emission electron source with the beam current of ~100nA and Cs spherical aberration corrector.

Together with imaging, phase analysis was performed by nano-diffraction analysis.

Sampling for SEM/FIB and STEM analysis was always selected close (about 200μm) to the coating delamination area (if visible delaminaton was observed), or near the middle of the sample (when there was no visible coatings delamination after HHF test).

Our previous work [3] showed that there is a direct relation between the coating thickness and delaminated area, namely 10 microns W coatings are less damaged than 20 microns W coatings tested in similar conditions. This behavior has been explained at that time by a better thermal transfer of the 10μm W coatings compared with 20μm W coatings. It was supposed also that the sharp transition at the interface might cause a poor performance to HHF tests. That is why in this set of experiments for one sample a gradient layer, at the level of the interface between Mo and W layers, has been applied.

The sample no. 1 was coated in similar conditions with 20μm W coatings that are applied on JET divertor CFC tiles. For this sample a cathodic sputtering stage precedes the start of the deposition process. Usually this process is performed at a bias voltage of –800V for 20min. and then the magnetron current is increased gradually up to the nominal value. The samples designed as 2, 3 (CMSII-M) and 4 (CMSII-Gr) were deposited at different conditions. Compared to standard

CMSII, for samples 2 and 3 (CMSII-M) the energy of the ions, during the sputtering stage, was substantially reduced. The sample no. 4 (CMSII-Gr) has a gradient structure of the interface from 100%Mo to 100%W.

3. RESULTS AND DISCUSSION

As a result of an intense technological research it was found that the nano-pores structure depends on the energy of the ions/atoms striking the coating during the deposition process. By optimizing this energy pores free interfaces were produced. This can be seen in Figure 1 where SEM/FIB images of CFC/Mo and Mo/W interfaces for W coating deposited by standard benchmark CMSII and W coating deposited by CMSII-M before HHF test are shown.

A fine network structure of pores (10–20nm in diameter) is visible for the coating deposited by standard CMSII (Figure 1a). It can be observed that for the CMSII-M coating, deposited with a fast transition from sputtering regime to deposition, the interfaces are sharp without pores, cracks or other defects (Figure 1b).

The fine structure of the gradient coating (sample 4) as revealed by STEM investigations can be observed in Figure 2. It can be seen that the gradient coating consist of three sublayers with a band structure. The top layer of the gradient region consist just of W. On top of the gradient coating there is a thin (~200nm) layer filled with nano pores.

After HHF tests, carried out in accordance with the data from Table 1, the structures of the coatings change significantly.

The images of the interfaces after HHF test are shown in Figure 3.

For the sample no. 4 (gradient coating) almost all the coating structure has been carburized. The resulted WC grains have width up to 1μm whereas their length spans on the entire layer. The W_2C grains have width up to 1μm and length up to 7μm. A large pore network was developed at the interface CFC-Mo and Mo-W. As it can be observed in Figure 3a the pores concentration is higher in the vicinity of the interfaces (CFC-Mo_2C and Mo_2C-WC). These pores have dimensions in the range 50–250nm. Large pores (up to 1 μm) can be observed at the bottom of W_2C layer at the border with WC phase. As the formation of this pores are in the vicinity of two new formed phases a possible explanation of their origin might be the recrystallization processes or phase transformation induced by heating.

For the sample no. 2 (CMSII-M) the dimensions of the pores increased significantly (up to 100nm) and so that they became more visible than in the initial state. Compared with benckmark CMSII coating [3] where micro-pores chains are formed, for CMSII-M coating these pores are isolated. The formation of pores inside tungsten carbide phase resulted after HHF test was considerably reduced compared with sample 4.

It can be noticed that the interface damage of the W coating deposited by CMSII-M is less than that produced on the interface of the coating deposited by CMSII-Gr. As far as the interface carbidization is concerned there is no significant difference between the two coatings. In both cases the Mo interlayer was carbidized.

The phenomena of pores formation has been reported by other authors during helium ion irradiation of W [4,5]. The process called the formation of fuzzy structures has been observed at high temperature (in general above 800°C) and at different He fluences. The formation of this structure has been observed at high temperature in the absence of He bombardment too [5] and consequently can be the result of recrystallization processes.

The thermo-mechanical properties of the W coatings are evaluated by the degradation curves obtained by the HHF tests. The ratio between the damaged area due to buckling, blistering or local melting and the thermal loaded area by the electron beam was represented relative to the number of heating pulses. The results are shown in Figure 4 for the peak testing temperature of 1450°C. As it was expected the sample 2 has a better behavior in comparison with the sample1.

As far as concern the coating with a gradient structure of the interface (CMSII-Gr), after just 1000 pulses a percent of ~ 0.6% of the coatings was delaminated.

The delaminated percentage for the W coated samples produced with modified technology (CMSII-M) was reduced by a factor of ~ 1.7 compared with standard 20μm W coating.

The peak temperature is an important factor for the HHF test results. For the same coating the delaminated percentage increases from 0.016% to 0.47% when the testing temperature rises from 1300°C to 1450°C. This can be seen in Figure 5 where the degradation curves for samples 2 and 3 are shown.

For the same coating the delaminated area reached a percent of 0.47% at 1450°C compared to a fraction of 0.016% at 1300°C.

The optical inspection of the sample surface after HHF tests showed that the dimensions of the delaminations/defects are quite small. Typical dimensions are in the range 0.005 ÷ 0.15mm^2. A typical dimensional distribution of defects (delaminations) is presented in Figure 6. The small defects prevail comparatively with the larger ones, and from the cumulative frequency distribution it can be seen that almost 97% of the total number of defects have dimension less than 0.04mm^2. No major differences have been observed between the dimensional defects distribution of the coatings tested at 1300°C and coatings tested at 1450°C.

CONCLUSION

It can be concluded that the state of the interface affects significantly the high heat flux resistance of W coatings. A smooth transition between the Mo layer and W can lead to the formation of a fine pore network. During the HHF tests these pores coalesce and as a result large pores or even cracks are formed. This phenomenon has a negative impact on the resistance to HHF of the W coatings.

On the other hand a pore free interface improves the resistance to HHF of W coatings. This pore free interface can be obtained by adjusting transition regimes of the deposition process. A reduction with a factor of 1.7 of the delamination percent has been evidenced for the coatings deposited in modified conditions compared with benchmark W coatings deposited by CMSII. This improvement is due to the extent of pores formation after HHF test which in this case in much reduced.

ACKNOWLEDGMENTS

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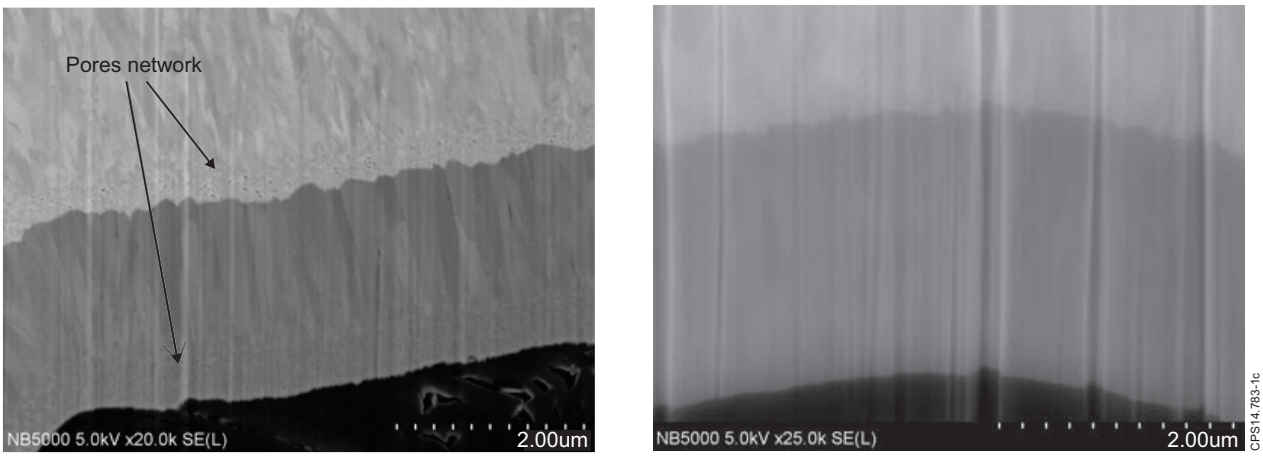
No	Tech.	d (μm)	T_{peak} (°C)	P (MW/m^2)
1	CMSII	20	1450	4.0
2	CMSII-M	20	1450	4.0
3	CMSII-M	20	1300	3.5
4	CMSII-Gr	10	1450	4.0

Table 1. Testing conditions.



(a) (b)

Figure 1: SEM/FIB images of CFC/Mo and Mo/W interfaces for W coating deposited by benchmark CMSII (a) and W coating deposited by CMSII-M (b) before HHF test.

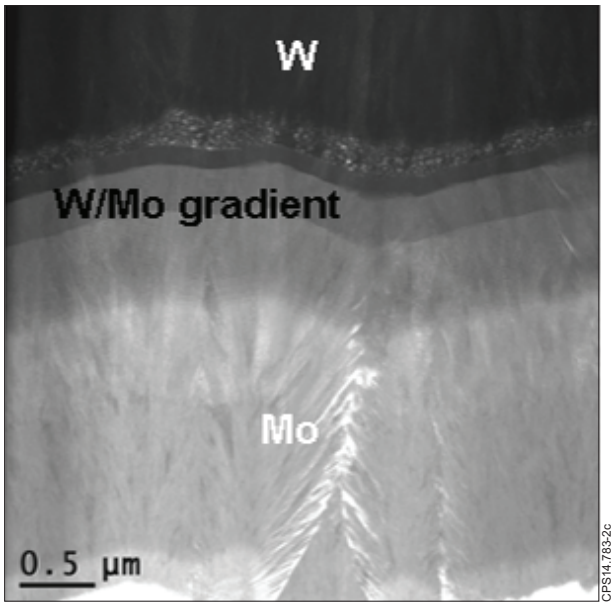


Figure 2: Bright field STEM image of the interface of sample no. 4, before the HHF test.

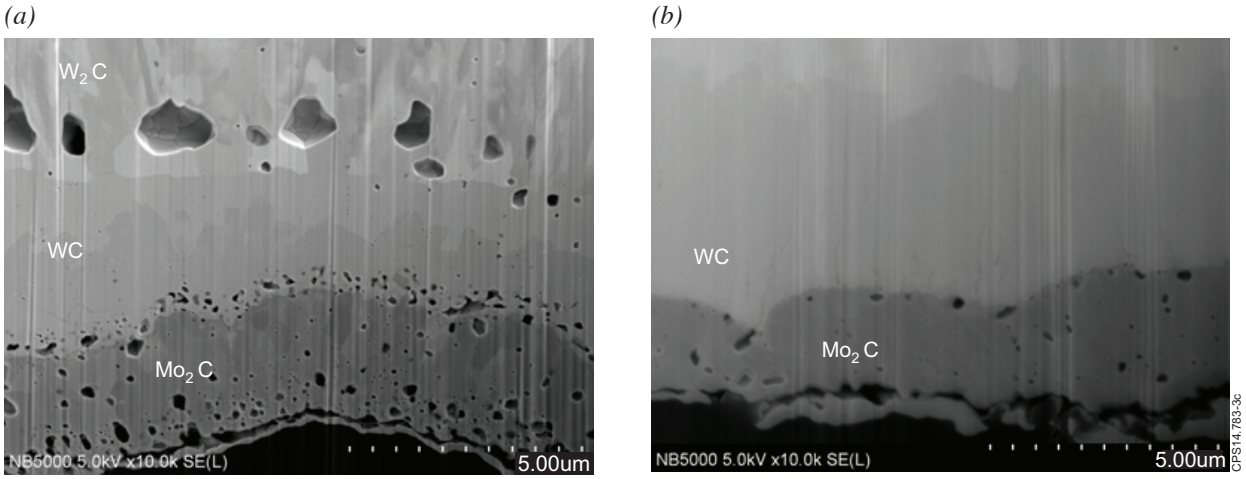


Figure 3: SEM interface after HHF tests of sample 4 (a) and sample 2 (b).

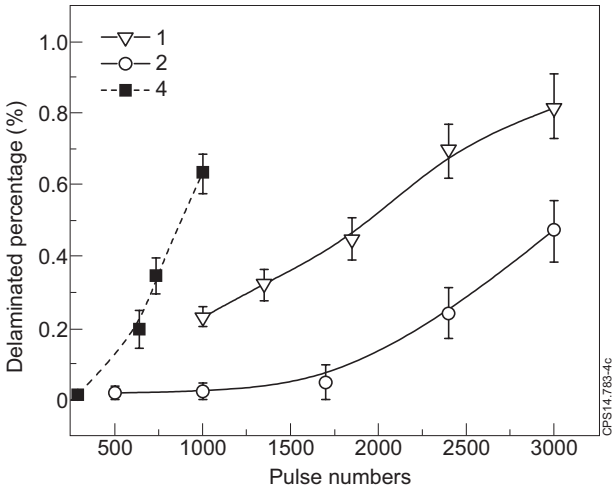


Figure 4: The evolution of delamination percent as a function of pulse numbers (Testing temperature 1450°C).

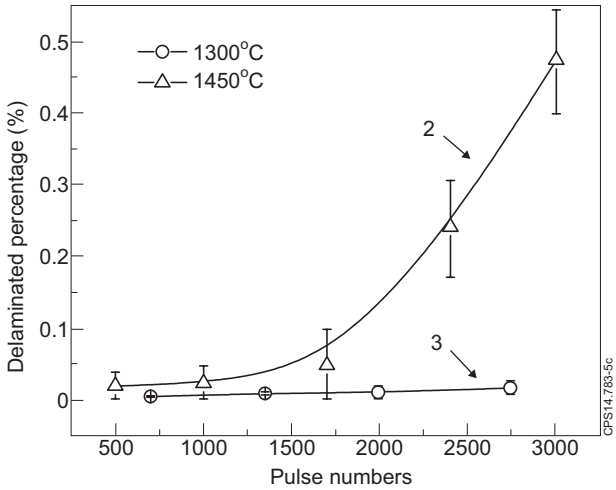


Figure 5: The evolution of delamination percent as a function of the testing temperature.

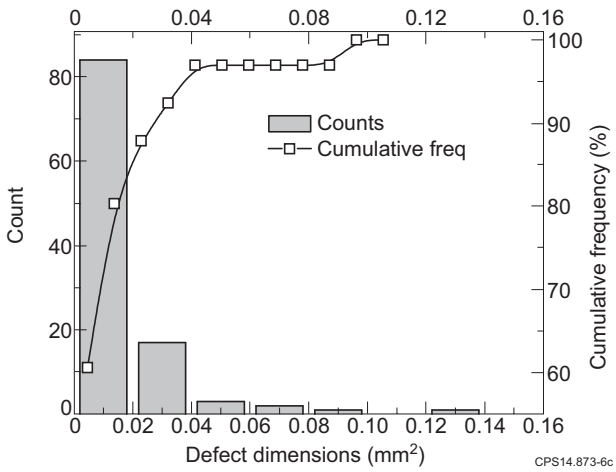


Figure 6: The dimensional distribution of defects for the sample No. 2.