

CEFDA05-1261

**Task Title: JW5-BEP-CEA-28: ITER WALL MATERIALS IN JET:
SUPPORT OF THE OPERATOR IN DESIGNING JET PFC
DEVELOPMENT OF W COATINGS ON CFC SUBSTRATE**

INTRODUCTION

In the framework of the JET-EP2 project, ITER-like wall project is planned to be implemented on JET during a shutdown in 2008. The project foresees the use of beryllium on the first wall and tungsten in the divertor. For the latter R&D activities on W coating on CFC tiles has been launched in the Associations. Based on the knowledge obtained previously on tungsten coating by Plasma Spray [1], [2] and by CVD assisted by plasma [3], the Association Euratom-CEA proposed to develop thin tungsten coating by means of CVD techniques and thick tungsten coating by means of Plasma Spray techniques.

2005 ACTIVITIES

The scope of the work in 2005 was to develop and qualify processes for tungsten coating on CFC tiles aiming at a possible application at an industrial scale for the full divertor tiles of JET. It was also required to characterize the coating, in particular with respect to the thickness and impurity content.

CFC substrates have been received from JET at the end of June and tungsten coated CFC tiles have been sent back to Garching in October 2005. The CFC material is bidirectional (fibres are in planes in x-y direction) and two types of tiles have been manufactured: Type-1 was large (80x80x40 mm³) with fibres perpendicular to the surface (good thermal conduction toward the back side of the tile) and type-2 was smaller (80x40x40 mm³) with fibres parallel to the surface (low thermal conduction toward the back side of the tile). The required thickness for the type-1 tiles was 4, 10 and 200 µm and 4 µm for the type-2 tiles. CVD deposition has been performed at WTCM in Belgium. The company is equipped with a large CVD reactor, which can handle large quantity of toxic gas such as tungsten hexafluoride used as tungsten vector. The reactor is pumped down to 90 mbar in order to reduce the oxygen contamination. The cracking temperature is set at 500°C. Additionally to WF₆; argon and hydrogen gases are introduced in the reactor. The figure 1 shows one CFC tile coated with 10 µm tungsten layer on all except bottom surfaces. A set of 4 type-1 tiles coated with 10 µm and a set of 4 type-1 and one type-2 tile coated with 4 µm have been sent to IPP Garching for thermal analysis.

Plasma Spray deposition has been done at St Gobain in Avignon (France). The company is equipped with several

plasma torch mounted on robot, one of them being installed in a large vacuum tank. Different parameters have

been tested (gas fluxes, pressure, powder, power) in order to minimize the lamellar effect and to increase the bonding of the layer to the substrate.

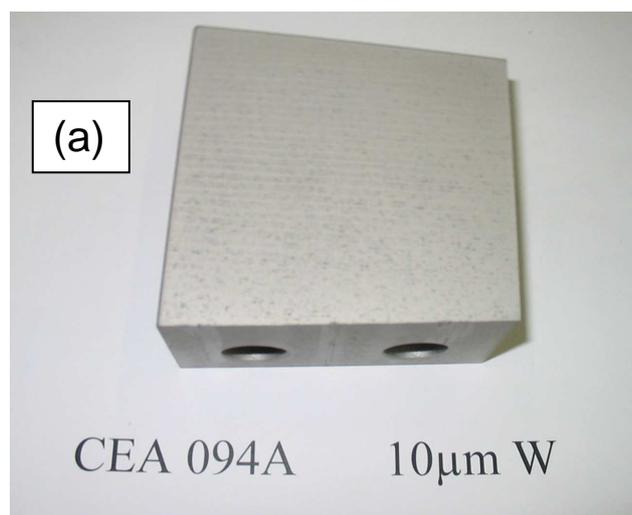


Figure 1: Type-1 CFC tile coated with 10 µm tungsten by CVD

To accommodate the large differential expansion coefficient between tungsten and carbon and to reduce the tungsten carbide formation, the JET specifications implied to use a sub-layer with a material, which had to be defined. Rhenium has been chosen due to its compatibility with tungsten (similar mass, high thermal conductivity, high melting point, alloy formation with tungsten). The thickness of the rhenium layer has been set at 30 µm, leading with a 170 µm for the tungsten layer. During the R&D phase it has been observed that the best rhenium coating is achieved under a low pressure (~100 mbar) in argon while tungsten layer showed better metallographic structure at atmospheric pressure in argon. In both cases, temperature of the substrate is kept below 100°C in order to avoid stress formation in the layers. The figure 2 shows a type-1 tile coated with a 30 µm Re/170 µm W layer. A set of 4 type-1 tiles has been sent to Garching for thermal analysis.

Preliminary analysis performed by means of Scanning Electron Microscopy (SEM) and X-Ray analysis (EDX) at Marseilles University shows that the thickness of the layers fitted within the requirements and that impurity content is very low in both CVD and PS tungsten coating.

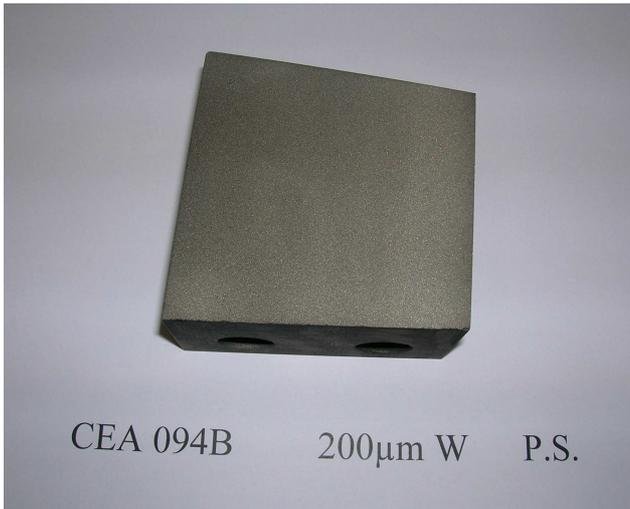


Figure 2: Type-1 CFC tile coated with 200 µm layer (30 µm rhenium sub-layer and 170 µm tungsten) by means of Plasma Spray

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CONCLUSIONS

The R&D phase on tungsten coating on CFC tiles has been successfully achieved and two types of coating have been performed. One by means of CVD aiming at producing thin layers of 4 and 10 µm on type-1 and 10 µm on type-2 tiles. One by means of plasma spray coating aiming at producing a 200 µm thick layer of tungsten including a 30 µm sub-layer of rhenium.

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- [2] Re-emission and thermal desorption of deuterium from plasma sprayed tungsten coatings for application in ASDEX-upgrade
Journal of Nuclear Materials, Volumes 233-237, Part 1, 1 october 1996, Pages 803-808
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- [3] Development of tungsten coating for fusion, S. Bentivegna applications
Fusion Engineering and Design, vol.56-57 (2001) p.331-336 (2001)
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JW5-AEP-CEA-26

**Task Title: R&D ON W COATING ON CFC AND BULK W TILES
DEVELOPMENT IN SUPPORT OF THE ITER-LIKE FIRST WALL
EXPERIMENT PROJECT**

INTRODUCTION

The JET project "ITER-like wall" is ongoing with the aim of replacing all due plasma facing components. The complete JET wall replacement is to take place in 2008. Element manufacture in industry is planned in 2006/2007. A R&D program for the fabrication of W-and Be-covered elements has been launched in 2005 to define and assess the technologies required by the project.

The first option for the divertor tiles consists in W-coated CFC blocks. Different Associations and companies are carrying out technical developments. Among these associations, MEC (Romania) is a recent producer of tungsten coating has been judged useful to associate their task with a collaborative to fusion lab.

The goal of this task is to support MEC in the R&D to identify and qualify technologies for the production of W coated CFC tiles to be installed in the JET divertor during the 2008 shutdown. MEC uses CMSII (Combined Magnetron Sputtering and Ion Implantation) and TVA (Thermoionic Vacuum Arc) techniques to produce tungsten coatings having thickness of 4 μm (3-6 μm) or 10 μm (9-12μm) on CFC substrate with samples and tiles provided by JET.

MEC has the following testing capability:

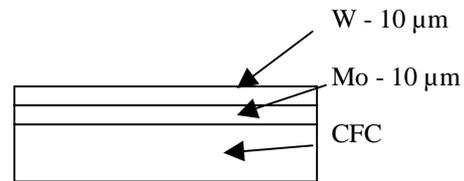
- metallographic examination
- heat flux tests (hollow cathode)
- adhesion tests

The task was initiated in may 2005, and a close collaboration started effectively in July with the visit of R. Mitteau and X. Courtois to MEC. During this visit, the advantages and drawbacks of both coating techniques were discussed, along with various other points (samples testing including high heat flux tests, quality control, industrialisation). The contours of this task were also specified. It was decided that CEA should focus on the following points:

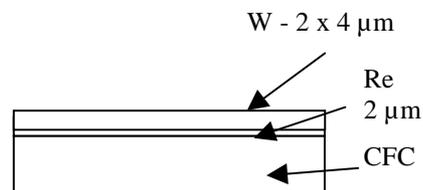
- SEM examination, chemical analysis
- Non-destructive examination through thermal lock-in techniques
- cross check of adhesion tests

The TL (H. Maier) was kept informed of the talks. His visit to CEA lab in Cadarache was the opportunity to confirm the decision over the actions to be done by CEA lab.

The design morphology of the MEC samples is as follows

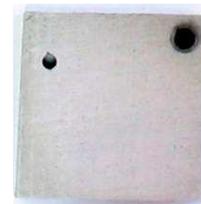


CMSII

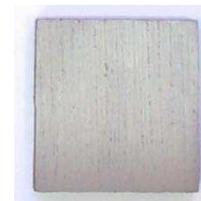


TVA

Figure 1: Sketch of the samples



CSMII-4, CF-stainless steel / W



*TVA-3, CFC / Re / W / Re / W
total coating thickness = 8 μm*

Figure 2: Photos of 2 samples

In July 2005, 6 samples were delivered to CEA for the investigations

2005 ACTIVITIES

MORPHOLOGY

Both processes produce uniform coatings (figures 3 and 4).

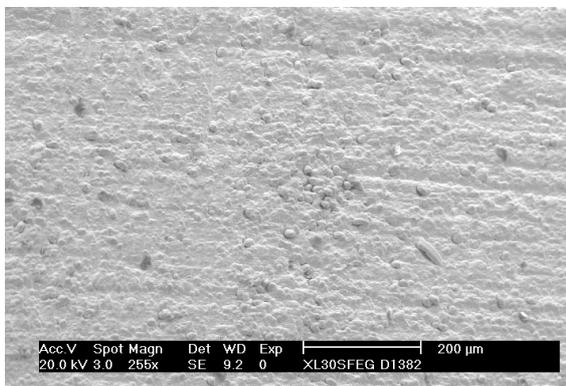


Figure 31: Surface in an area with few grains (CMSII-4)

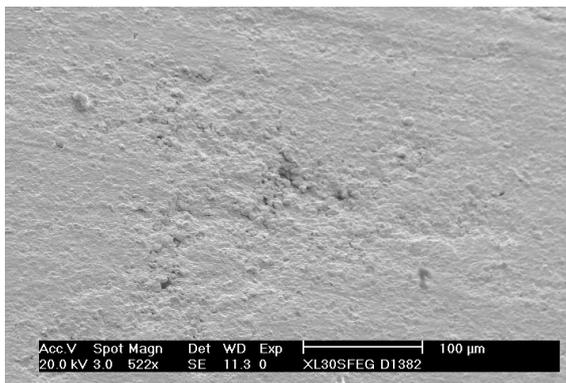


Figure 4: Typical TVA-3 surface

Holes in the coating are apparent in some locations. The hole size is up to 400 µm wide. Carbon composite is not directly apparent at the bottom of the holes, so that the defect may be acceptable. A reason for the holes may be the inherent roughness of the carbon composite. Large holes (> 10 µm) are present at the surface of the carbon composite, and the thin coating has not the possibility to fill it. In order to reduce the occurrence of these holes, a possibility could be to control the roughness of the tile before coating.

The coating surfaces are grainy, a common feature for tungsten coatings. Grains are typically 5-10 µm for CSM-II, somewhat smaller for TCA (2-5 µm).

Wedges have been done in the coating, using a Focused Ion beam, in order to investigate the morphology of the coating. For CSM-II, the total thickness of the W layer is of the order of 10 µm. This value is within the specification (9-12 µm). Adhesion appears good (no crack between layers).

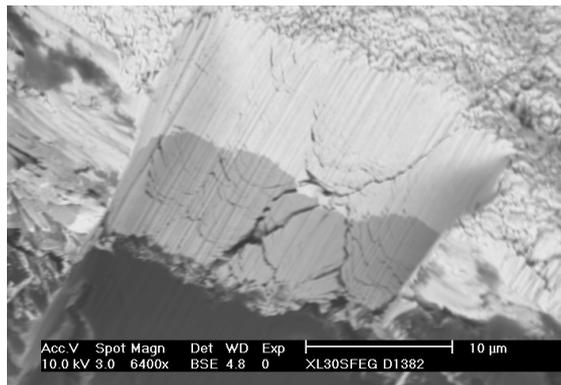


Figure 5: Ion beam wedge

For the TVA sample, the thickness is not sufficient (2 µm). the very thin rhenium interlayer could be observed. Its thickness is evaluated to be 300nm at the location of the observation.

COMPOSITION

The composition is evaluated using energy dispersive X-ray microanalysis. Surface analysis shows a majority of tungsten, usually with carbon as impurity. The oxygen is at a level which is sometimes not noticeable with this technique (figure 6), and reaches 2% (weight %) at the highest.

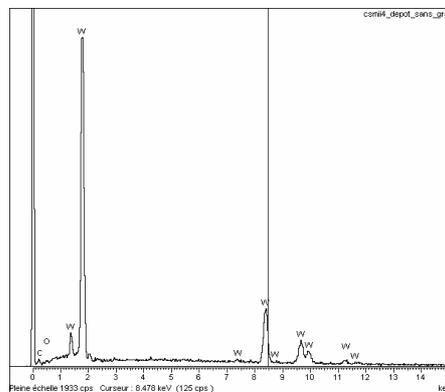


Figure 6: Energy dispersive X-ray microanalysis for CMSII-4, on the coating

NON DESTRUCTIVE EXAMINATION

A non destructive technique (NDT) is essential to control the coating quality after manufacture. Conventional NDT techniques such as Xrays are not usable, because the quantity of material to investigate is small compared to the substrate, so that the useful signal is usually very small and lost in the noise.

CEA has developed an expertise in thermal NDT, which was essential during the reception of the high heat flux elements of TS limiter. Recently, a new NDT branch was investigated, based on modulated signal (lock-in techniques). A test bed has been set up. The applicability of such techniques to tungsten coating was studied.

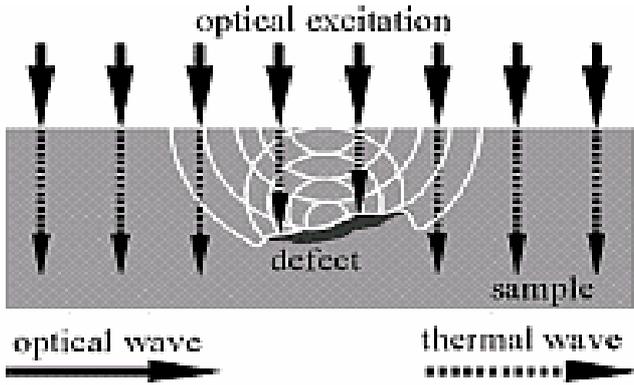


Figure 7a: Principle of thermal lock-in techniques

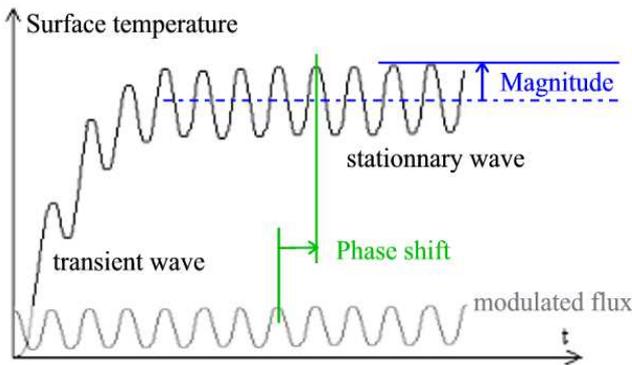


Figure 7b: Illustration of the measured phase shift

The following cartographies show the phase shift (in degree) of the Lock-in examination. The absolute amplitude is not significant, only phase contrast give the relevant information.

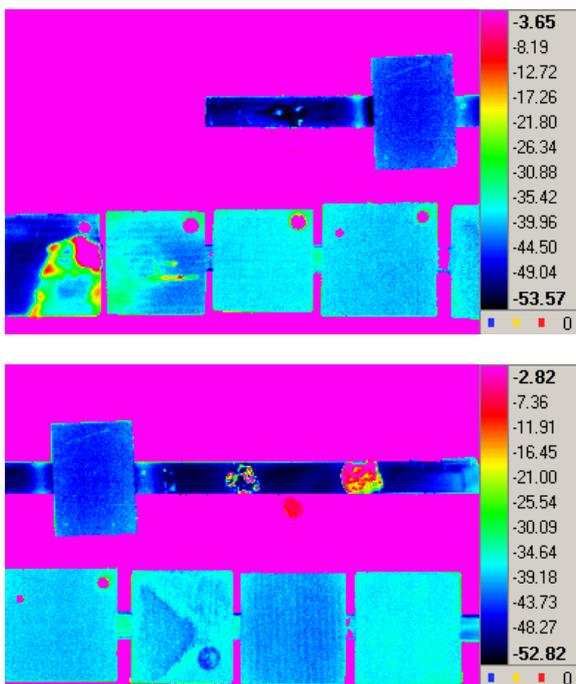


Figure 6: Phase-shift cartographies

CMSII 1 & 2 and TVA 1 where known in advance to be strongly damaged. They were given by MEC to assess the possibility to realise NDT using lock-in techniques.

The other samples (thought to be the good one) have a homogeneous phase map: the coatings appear rather uniform from the point of view of the lock-in technique.

The phase-shift is not far from that of the CFC slab used as reference, which means that the coating do not change significantly the diffusion path, and therefore that the adherence of the coating is not bad.

For CSMII 1 and TVA 1, this contrast is linked to the non-uniformity of the coating material, which is strongly damaged or delaminated. The case of CSMII 2 is peculiar, because the coating is visibly delaminated over a band of 1 mm x 10 mm

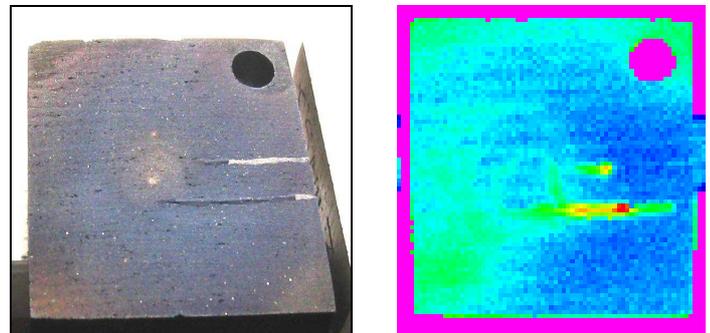


Figure 9: CSMII 2, photo and phase shift

The zooms off the CSMII 2 picture and phase shift show that the delaminated area where the coating has been removed is not contrasted. On the contrary, the area where the coating is delaminated but still fixed is very contrasted. In that case, the thermal behaviour of the surface is significantly modified compared to the previous one, due to a thin air layer, which produces a thermal contact resistance between the surface and the substrate. This shows that the thermal lock-in allow to evidence the delaminated area.

CONCLUSIONS

The characterization done at CEA and the fruitful collaboration with the Romanian colleges have allowed to optimise the deposition process. The deliverable were sent in due date to the task leader. The task is pursued in 2006 toward the realisation of adhesion tests.

REPORTS AND PUBLICATIONS

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