JET PROGRAMME IN SUPPORT OF ITER

ITER-like Wall Project

Task: JW5-TA-EP-BEW-02
R&D on W coating on CFC and bulk W tiles development in support of the ITER-like Wall Project (BEW)

JW5-AEP-MEC-03: Development of W-coating technologies for CFC tiles using CMSII and TVA methods.

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1. Introduction

Neither the primary materials combination (Be Wall/CFC+W divertor) for ITER nor its backup (Be Wall/W divertor) has ever been tested in a tokamak. The information that could be obtained from such an experiment in JET might be critical for reducing the risk of material-related problems in ITER. Therefore, the ITER-like Wall Project has been launched at JET and, six Associations (IPP, FZJ, CEA, ENEA, MEdC and TEKES) were involved with the objective to develop W coating technologies (4 μm, 10 μm and 200 μm) for CFC tiles and a bulk W tile design for use in areas near the strike points. The R&D activities were managed by IPP for W coating and FZJ for bulk W. In accordance with the EFDA Task Agreement JW5-TA-EP-BEW-02 and with the Individual Task Description (ITD No. JW5-AEP-MEC-03), two technologies had to be developed by MEdC for W coating of CFC tiles. These were:

A. Combined Magnetron Sputtering and Ion Implantation (CMSII) and

B. Thermionic Vacuum Arc (TVA)

On the other hand, because only one set of samples has been attributed to MEdC Association, only one of these technologies was applied for coating of the test samples. This one was chosen depending on the performances of the W coatings and on the capability of the coating technology to be applied at the industrial scale. The primary tests were carried out with in house facilities of NILPRP (National Institute for Laser, Plasma and Radiation Physics) and together with CEA Association.

The research activity in MEdC Association was focused on the following objectives:

- Development of "in house" testing techniques for W layers, in order to be used for the optimization of the coating parameters.
- Identification of the best material for an interlayer between CFC substrate and W coating, able to ensure a very good adhesion between the CFC and W under high thermal loads.
- Optimization of the coating parameters for both CMSII and TVA techniques in order to produce on CFC, tungsten coatings of 10 μm (perpendicular to the fiber) and 4 μm (parallel to the fiber).

The coating characteristics and the testing conditions were specified in a special document entitled "Specifications for the R&D of tungsten coatings on CFC within the JET ITER-like Wall project", produced in due time (29.04.2005) as Milestone M2. The machined samples have been delivered by JET (Milestone 3) with a small delay, but in due time for coating. The test samples were W coated with the optimized CMSII technology (Milestone 4) and delivered to IPP Garching by 15.09.2005. The delay of 2 weeks was negotiated with the Technical Leader. X-Ray Radiography was proposed by MEdC as a Non Destructive Test (NDT) for determination of the coating thickness and uniformity (Milestone 5). Intense activities have been carried out by the National Institute for Laser, Plasma and Radiation Physics (NILPRP) in order to demonstrate its capability to apply the CMSII technology at the industrial scale with relevant Quality Control standards. In this respect ISO 9001 and ISO 14001 standards have been implemented.

2. Results and discussions

According to the Specifications for the R&D of W coatings on CFC, the following characteristics of the layers must be determined: the thickness, the coating impurities, the adhesion to the CFC substrate and the resistance to high heat flux. In order to evaluate these properties the following testing techniques for W coating were developed:

- Metallographic procedure for optical examination of the coatings deposited on CFC. Using this technique, the thickness of the coating was determined.

- Pulling test to check the adhesion of the coating to the CFC substrate

- Cyclic Thermal Fatigue test

A short description of these techniques and the results of the measurements are given below.

2.1. Metallographic procedure for optical examination of the coatings deposited on CFC

SEM method was proposed to be used for thickness measurement at IPP Garching. Alternatively, a special metallographic technique for optical examination of the W coatings on CFC substrate has been developed. By this way the coating thickness was measured. Because the CFC is a soft material, the conventional technique for metallographic preparation cannot be applied. The direct deposition of 10 μm W coating on CFC was not successful

by both CMSII and TVA techniques. An interlayer seemed to be necessary and this appeared to be the key factor for obtaining a good W coating on CFC. Various materials have been checked. For TVA an interlayer of ~0.2 μm of Re was found to be the best solution. As far as concern CMSII technique, Cu, Cr, stainless steel and Mo were used as interlayers. A special technique for sample preparation for optical investigation has been used. In Figure 1, an image of a Cr-Cu-W multilayer is presented. The thickness of the interlayer is also important for the performances of the coating. This thickness was modified from 2 to 10 μm. As far as concern the adhesion at room temperature, no significant difference have been observed, but the tests under high thermal loads indicated the best behavior for the coatings deposited with an interlayer of 2 – 3 μm. The micrographs of the W coatings deposited on CFC with 8 μm and 2 μm Mo interlayer are shown in Figures 2 and 3. A difficult task was to find a reagent to discriminate between W and Mo. The reagents known in the literature can reveal both W and Mo, but cannot make the difference between them. After a number of experiments, a combination between Marble's and Nital reagents used in succession was able to discriminate the two layers.

2.2. The adhesion of the W coating to the CFC substrate

Schematic arrangement for pulling test is shown in Figure 4. A pulling rod made of brass with a diameter of 5 mm is stuck with the flat base to the W coating by a commercial strong adhesive called poxipol (a two-component adhesive used for sticking various materials including metals, ceramics, wood, plastics, etc.).

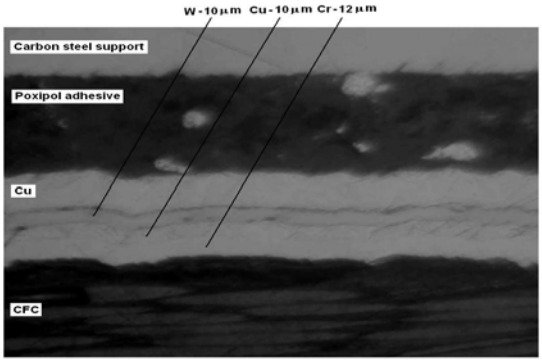

Figure 1. Optical micrograph of a Cr-Cu-W multilayer deposited on CFC substrate

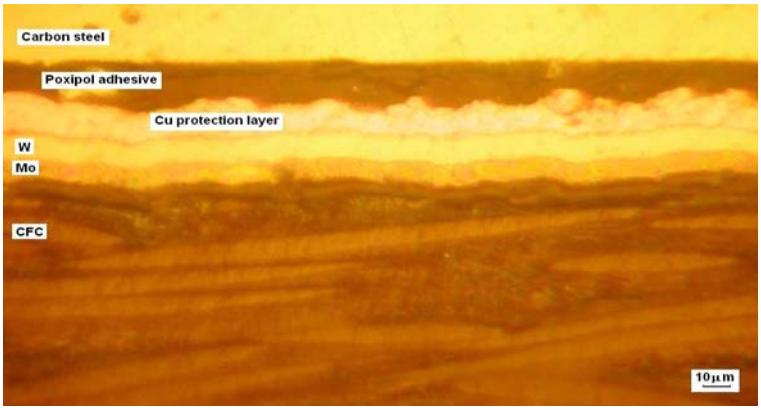

Figure 2. Tungsten coating deposited on CFC substrate with 8 μm Mo interlayer

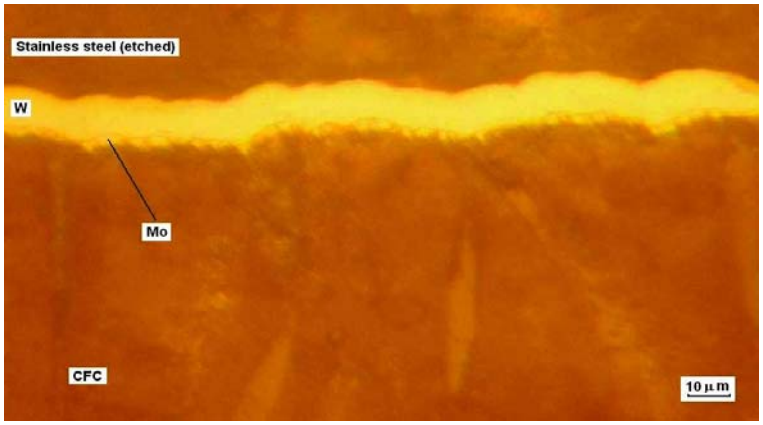

Figure 3. Tungsten coating deposited on CFC substrate with 2 µm Mo interlayer

It is important that the pulling rod to be perpendicular to the W coating. The pulling force is gradually increased until the detachment of the pulling rod from the W coating occurs. The ratio between the detaching force and the area of the removed layer can be defined as Detaching Specific Load (DSL). A picture of the CFC sample coated with Cr + Cu + W, together with the detached rod can be seen in Figure 5. As it can be seen, at the first test the adhesive failure occurred. This happened at 3.9 MPa. The W-coatings remained intact. The surface was cleaned and the second test has been done. In this case, a complex failure occurred. In some regions the adhesive was detached from the W surface, in other regions the adhesive was broken itself and in other regions the CFC was broken. No traces of metal have been detected on the detached rod. This means that the detachment did not occur from the CFC-interlayer or from interlayer-W coating interfaces.

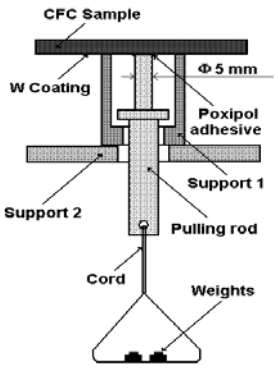

Figure 4. Pulling test device

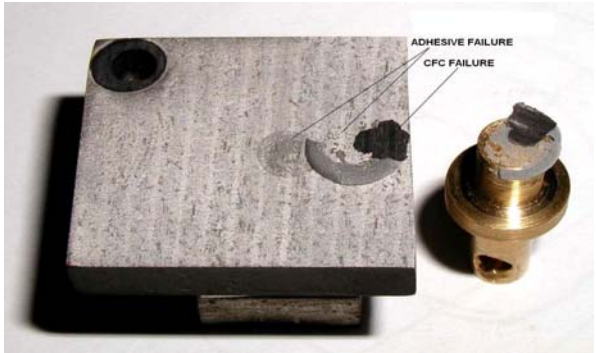

Figure 5. Result of a pulling test

A summary of the pulling test results is given in Table 1. According to the preliminary results, a Cr + Cu interlayer seemed to provide a very good adhesion, but the melting temperature of Cu is quite low (1083 °C) and this might affect the integrity of the whole layer under high heat fluxes. The idea was to replace Cu and this is why austenitic stainless steel has been taken into consideration as an alternative. It ensures a good adhesion as well and it is quite refractory. The next step, as an alternative to stainless steel, was Mo. The melting temperature is ~2600 °C and the expansion coefficient has a value between W and CFC. The final decision for CMSII deposition method was Mo as an interlayer material.

Table 1 Pulling test results for particular W/interlayer coatings

No	Interlayer / layer / characteristics	DSL (MPa)	Observation
1	None + W (CMSII)		Poor adhesion (can be removed with scotch tape)
2	Cu (4 μm) + W (CMSII)		Poor adhesion (can be removed with scotch tape)
3	Cu (10 μm) + W (CMSII)		Can not be removed with scotch tape
4	Cr deposited on CFC without W (CMSII)		Can not be removed with scotch tape
5	Cr with W (10 μm) (CMSII)		Poor adhesion (the layer was delaminated)
6	Cr + Cu (10 μm) + W (10 μm) (CMSII)	> 3.9	Very good adhesion - At the first test the poxipol* was detached from the W surface at 3.9 MPa - At the second test a complex failure occurred (detachment from the W surface, from the adhesive and from inside of CFC)
7	Stainless steel + W (10 μm) (CMSII)	2.2	Good adhesion
8.	Mo (~3 μm) + W (11 μm) (CMSII)	3.1	Good adhesion Can not be removed with scotch tape
9	Re + W (3.7 μm) (TVA)	3.2	Good adhesion
10	Re (0.2 μm) + W (10 μm) (TVA)		Can not be removed with scotch tape
9	No layer, just CFC	5.4	Reference

In addition, abrasive resistance was checked using silicon carbide paper with grade of 800 to 240. The coatings deposited by TVA had a good abrasive resistance but not as well as those deposited by CMSII.

2.3. The resistance of the coatings to the high thermal loads

Before sending the samples for final testing at IPP Garching, preliminary thermal loading tests were carried out at NILPRP. Schematic diagram of the test facility based on a hollow cathode discharge is shown in Figure 6.

Practically, a modified Plasma Nitriding unit of 70 kW was used for these experiments. The main characteristics of the power supply are: U_{max} = 850 V, I_{max}=150A (for 500 V). The unit is equipped with an arc suppression electronic system.

A high intensity discharge has been initiated between a tungsten hollow cathode and a W coated sample as anode. The discharge is stable and can be controlled. Two working regimes have been identified.

A. Hollow Cathode Glow Discharge (HCGD) and
B. Hollow Cathode Arc Discharge (HCAD)

In the HCGD regime, the discharge current is in the range of 6 – 12 A and the discharge is partially localized inside the hollow cathode. The cathode is red on the whole length

(Figure 7).

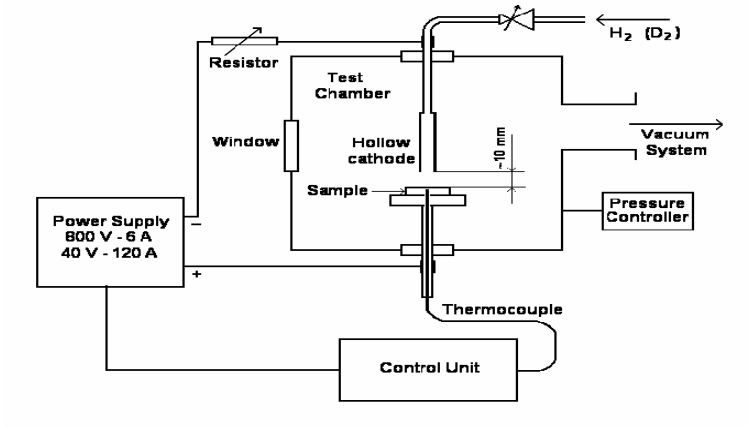

Figure 6. Schematic diagram of the CTF test facility

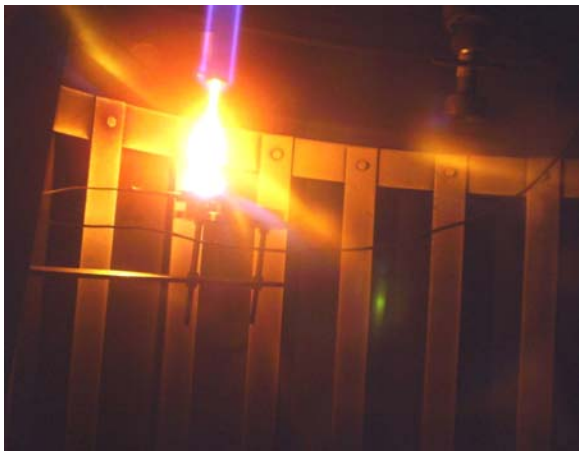

Figure 7. Hollow cathode discharge

The transfer rate of the energy from the discharge to the anode (sample) is relative low (~ 10 %). The discharge power is ~ 1.5 kW. If the input power is increased, the discharge goes into the HCAD where the transfer rate of the energy exceeds 20%. The discharge is localized at the lower part (10-15 mm) of the cathode and the discharge current exceeds 20 A.

The equipment was tested with a CFC sample of 27 x 28 x 8 mm supported by a stainless steel pin of 5 mm in diameter. The temperature was monitored by a thermocouple mounted through the bottom side, but near to the coated surface as it can be seen in the Figure 6.

The time dependence of the sample temperature for the two working regimes is shown in Figure 8.

The CFC sample had 8.5 g.

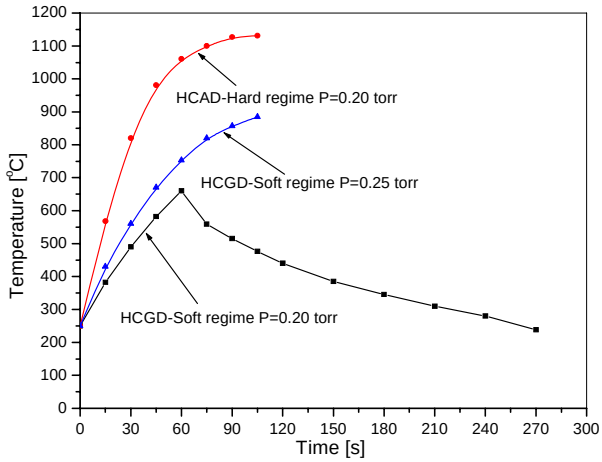

Figure 8. Time dependence of the sample temperature during the thermal loading

The input power density on the sample surface is quite difficult to be calculated, but some estimation can be made.

$$P_{in} = \frac{m \cdot c \cdot \Delta\theta}{\Delta t} + P_{rad} + P_c + P_{cv} \qquad (1)$$

The input power P_{in} is used to increase the sample temperature $\frac{m \cdot c \cdot \Delta\theta}{\Delta t}$ and to compensate the loses produced by radiation (P_{rad}), conduction (P_c) and convection (P_{cv}).

In the case of HCAD the maximum obtainable temperature for a set input power was 1130°C. If we consider that at this temperature the majority of the power is lost by radiation, according to equation (1) $P_{in} \sim P_{rad}$. Using the Stefan – Boltzmann law for P_{rad} , a value of ~ 520 W is found for P_{in}. The surface area covered by the arc discharge cannot be measured precisely, but it can be estimated to 2 – 3 cm^2. Under these conditions the input power density is ~ 2 MW/m^2. This value is less by a factor of 5 than the standard 10 MW/m^2, but the pulse duration can be increase by the same factor in order to keep the same input energy per pulse. In 60s a temperature of 1060°C is reached and the temperature seems to be uniform for the whole sample. The time is long enough to allow the energy propagation within the sample.

Samples of CFC coated by CMSII with 10 μm W, but at different thicknesses of the Mo interlayer have been tested at 600°C, 800 °C and 1000 °C with 5 pulses for each temperature. For an interlayer of 10 μm, the coating did not survive at 1000 °C. The coatings deposited with 2-3 μm Mo interlayer resisted very well to 30 pulses at 1000 °C.

A W coating of 3.7 μm deposited by TVA technique survived at 5 pulses at 1000 °C.

2.4. Determination of the O and C impurities by XPS and AES techniques

Preliminary measurements of C and O concentrations have been carried out using XPS and AES techniques. Values of 15 – 17 at.% have been obtained (Figure 9). Similar values have been obtained for the coatings produced by both CMSII and TVA techniques.

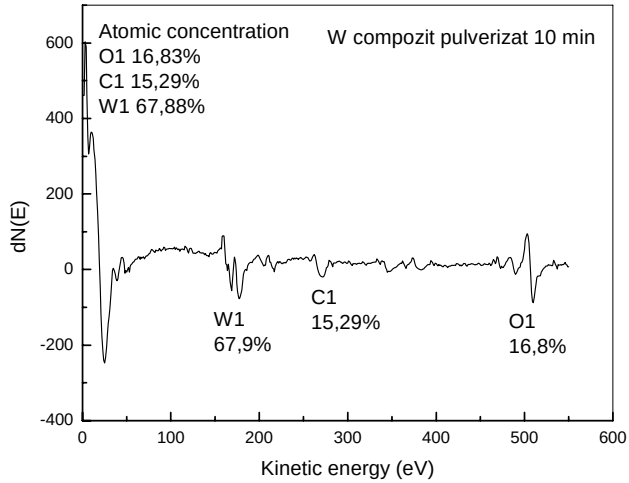

Figure 9. AES diagram for a W coating deposited by CMSII

In order to clarify the real concentrations of the C and O in the "bulk" of coating, alternative analyses have been carried out. Optical Glow Discharge Spectrometry (OGDS) indicated for W coatings deposited by CMSII and TVA on titanium and steel substrates concentrations of less than 1 at.% for both carbon and oxygen. Other analyses have been performed by EDAX on the surface of the W coatings deposited by CMSII and TVA. The carbon concentration was ~ 35 at.% for the sample deposited by CMSII and 0 % for the sample deposited by TVA. No oxygen has been detected for both samples. The machine had no possibility to make a depth profile of these concentrations by sputtering with argon ions.

2.5. ISO 9001 and ISO 14001 certification

The NILPRP should prove, by the end of the project, its capacity to coat at the industrial scale a large number of CFC tiles (~ 1000 Off) with the relevant quality control standards.

In this respect, a special activity was carried out in order to certify the NILPRP in accordance with the ISO 9001 and ISO 14001 standards. A contract has been signed with SC CERTROM SRL, a company that has the habilitation to certify the implementation of the above mentioned standards. During the 2005 year, all the documentation (quality manual, procedures, etc.) has been issued and an internal audit has been performed. The implementation of the ISO 9001 and ISO 14001 standards requirements in the laboratories involved in this project has been mostly done.

2.6 Coating of real samples for IPP Garching

The real samples for IPP Garching have been coated by CMSII technique. A picture of these samples is shown in Figure 10.

2.7. X-Ray Radiography examination of the coating uniformity.

A general task for the consortium was to define the non-destructive tests (NDT) to be applied to a large number (~1,000 Off) of real CFC tiles coated under industrial conditions. The MEdC Association proposed an X-Ray Radiography (XRR) technique to measure the coating thickness and uniformity.

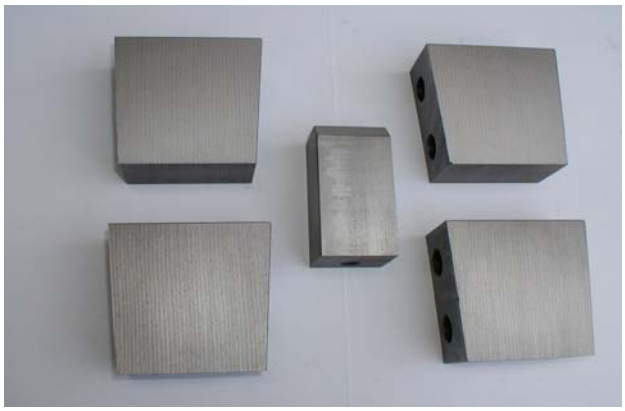

Figure 10. The CFC tiles coated by CMSII technology with 10 μm tungsten, which have been sent to IPP Garching for testing

This technique has been developed using the X-Ray Microtomograph facility existing at the NILPRP. In order to check the capabilities of the technique a non-uniform coating was produced using a special shield. The results are shown in Figures 11 and 12.

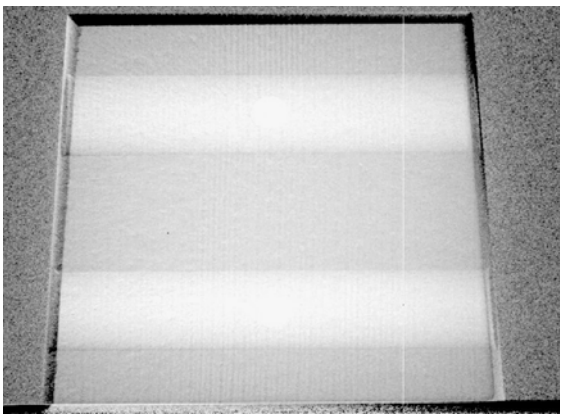

Figure 11. CFC tile coated with a non-uniform W layer

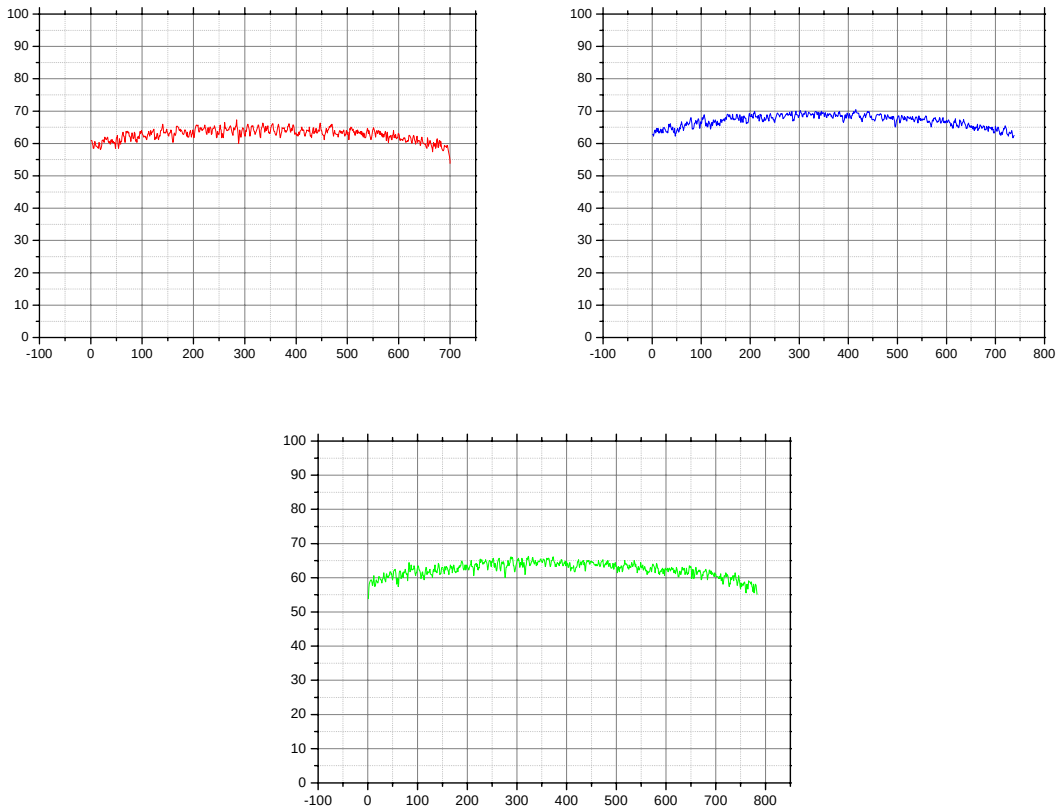

Figure 12. Profiles (along colored lines) showing the non-uniformity of the W layer

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The thickness of W coating deposited on CFC tiles is determined by absorption contrast of transmitted X-rays.

In order to mitigate the beam hardening (due to polychromatic character of the X-ray energy spectra) and scattering effects two X-ray transmission measurements are recommended: one before and one after coating. The useful result is obtained by subtraction of the two radiographies.

The grey values in the image are proportional to the coating thickness. The profiles are along the colored horizontal lines. The coating thickness is in arbitrary units. The non-uniformity is around 6%. As it can be seen, a lower non-uniformity (~ 2%) could be detected.

The ratio between the maximum thickness (central area) and the thickness in the lateral areas was checked with another sample, which was cut and examined by metallographic technique. The values obtained by the two techniques were very similar.

3. Conclusions

- By both CMSII and TVA techniques have been produced coatings of 10 μm. Some difficulties seemed to appear for TVA due to the limited lifetime of the filament. This problem was solved by successive use of three TVA guns during the same cycle.

- The test with SiC paper revealed a better abrasion resistance of the W coatings deposited by CMSII in comparison with those deposited by TVA.

- The CMSII technique has been chosen for coating of the five official test samples for IPP Garching.

- In addition, two samples were coated with W by TVA technique and they were also sent to IPP at the discretion of the Technical Leader of the Project.

- An X-Ray Radiography method was proposed as a non-destructive test in order to check the thickness and the uniformity of the W coatings deposited on real tiles of the JET divertor.

The objectives assumed by Task Agreement for the year 2005 have been fulfilled.

4. Activities to be performed in 2006

The Task Agreement for this project started on 20 May 2005 and ends on 28 February 2006. The activities envisaged for 2006 can summarized as follows:

- Finalization of the tests performed by IPP Garching. MEdC will contribute by comments and discussions in connection with the final results.
- Final audit and certification of the implementation for ISO 9001 and ISO 14001 standards at the National Institute for Laser, Plasma and Radiation Physics.
- Further development of CMSII facilities in order to coat at the industrial scale a large number of tiles (~ 1000 Off).
- Publication of some non-patentable results.

5. Collaborative actions

Collaboration with CEA

During the period 27-28 July 2005 two representatives from CEA, Dr. Raphael Mitteau and Xavier Courtois have visited the laboratories from NILPRP involved in this project. An experiment of thermal loading has been carried out together and selected samples have been taken in France for thermographic examination. This examination revealed a good adhesion of the coatings deposited by both CMSII and TVA techniques to the substrates. Unfortunately, there is no a reference standard so far, in order to quantify the adhesion of the coatings to the substrate by thermographic method.