ITER-like Wall Project

Task: JW5-TA-EP-BEW-03
Development of beryllium coatings for inconel targets and Be marker tiles for the ITER-like Wall experiment project

JW5-BEP-MEC-04: Manufacturing of optimized of Be/ heavy-metal/ Bulk-Be marker samples

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1. Introduction

The ITER-like Wall Project, part of the "JET programme in support of ITER", to be implemented on JET during in 2008, includes R&D activities to develop a method of depositing Be layers on an interlayer plus a Be overlayer onto Be tiles and characterization of the Be coating purity by surface and structure analysis techniques as well.

The JET main wall will be made of solid Be tiles. In order that the erosion rate of the Be wall to be assessed, it is necessary to be measured erosion of a few microns of beryllium. For this “markers” tiles will be distributed in the areas of interest such as Outer Poloidal Limiters (OPL) and Inner Wall Guard Limiters (IWGL).

The “marker” is a Be tile with a stripe of an easily detected heavy metal deposited on it as a thin interlayer, and with a few microns layer of the bulk-like Be on top of that. If the outer layer is eroded at the same rate as the bulk, then the erosion rate can be determined by detecting the distance of the interlayer from the final surface, for erosion of less than the film thickness.

The aim of this task is to obtain heavy-metals/Be coatings on Be samples using the original technique of Thermionic Vacuum Arc (TVA) developed at the National Institute for Laser, Plasma and Radiation Physics (NILPRP), Magurele-Bucharest. The manufactured coatings will be characterized by thickness of heavy metal and beryllium layers, uniformity and structure, purity, density and adherence.

2. Thermionic vacuum arc (TVA) technique

TVA technique is characterized by producing plasma in the pure vapours of the metal to be deposited (Re, Ni, Cr, W, Be, etc.) without using any buffer gas [1-2]. The evaporation of the metal takes place in high vacuum conditions (about 10^{-2} Pa and less). An external heated cathode (W filament) produces thermally emitted electrons of about 100 mA. These electrons are accelerated and focused by a Whenelt cylinder to the anode that is biased to high voltage

(1 – 6 kV). The electron bombardment creates space metal atoms above the anode at a local pressure of about 133 Pa.

The TVA principle scheme is presented in Figure 1.

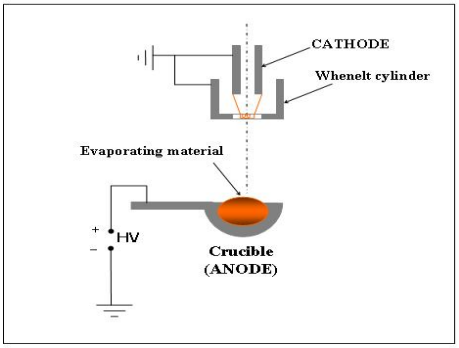

Figure 1. TVA principle scheme.

The thermoelectrons produced by the heated cathode are able to build up plasma by electron-metal atoms collisions. The new electrons generated in the plasma together with the original ones emitted by cathode enhance once more the anode evaporation and produce high quantity of ions. Usually the cathode potential fall is in the range of 200-300 V and therefore the plasma potential in comparison with ground ensure generation of the high-energy ions, which collide the substrate.

By using this technique deposition rates in the range of 0.1-2 nm/s could be obtained with the advantage that the high energy metal ions bombardment during deposition ensures high density layers, compared with those of the bulk material, good structure and high adhesion to the substrate.

The heating currents for filament furnished by the special one to three low voltage sources of the machine are of 200 A each. The high voltage sources can ensure a maximum voltage of 6 kV on the anode and a discharge current of 2 A that are more than sufficient for the purpose of this technique.

3. Results and discussion

The TVA facilities of NILPRP-Magurele were used to obtain W and Ni coatings. The facilities of the Nuclear Fuel Factory (NFF), Pitesti were used for beryllium coatings. The NFF equipment for thermal evaporation coatings was adapted to TVA technique and a preliminary optimization of beryllium coating was made.

3.1 TVA main working conditions

W, Ni and combinations of Re30Cr10Ni were prepared at NILPRP and Be coatings on small size (3 cm x 3 cm x 0.5 cm) samples of stainless steel, graphite and glass were prepared in the best experimental conditions at the Nuclear Fuel Plant facilities in Pitesti by thermionic vacuum arc (TVA) technique as follows:

The anode material:

- a crucible material based on TiB was used for Ni evaporation;
- for the W coatings due to the high melting point of this metal a rod of W was used instead of any crucible. In this case only the upper part of the rod was melted due to the low thermal expansion coefficient of W. The other part of this rod was sustained and kept relatively cold by a Mo cylinder holder;
- for Be coatings a W plate holder was used instead of Mo holder.

Intensity of the heating current of the cathode filament:

- during the evaporation the intensity of the cathode filament has been adjusted in the ranges of 110-120 A for W, 100-110 A for Ni and 80-100 A for Be, respectively. This adjustment was imposed to keep a stable discharge.

3.2 Characterization of the coated W and Ni samples

Scanning Electron Microscopy (SEM)

W, Ni and combinations of Re30Ni10Cr prepared at NILPRP were analyzed using Scanning Electron Microscopy (SEM). The cross sectional SEM image of the composite material Re30Cr10Ni, presented in Figure 2, shows a compact film, without columnar structure.

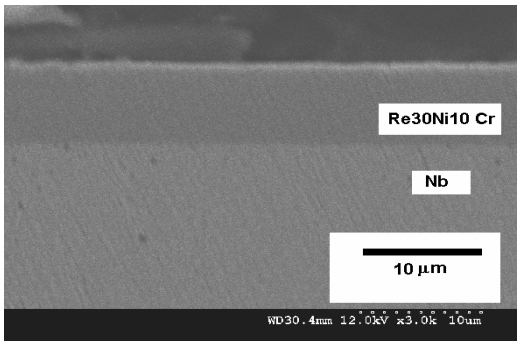

Figure 2. Cross sectional view of the Re30Ni1oCri film.

Fe substrates of 30 mm x 30 mm x 3 mm coated with W, were analyzed. The scanning area was of about 4 μm^2 and depth analysis of about 3 µm. The results presented in Figure 3 show a pure W film without impurities.

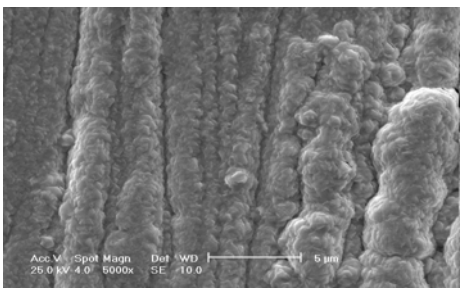

Figure 3. SEM image of W film on metal substrate

Electron Dispersive X-ray (EDX)

The most used method of microanalyzis - Electron Dispersive X-ray, with a typical 70-130 eV resolution, was used to investigate the W coatings. Typical spectrum presented in Figure 4, proves the high purity of the coatings prepared by TVA technique.

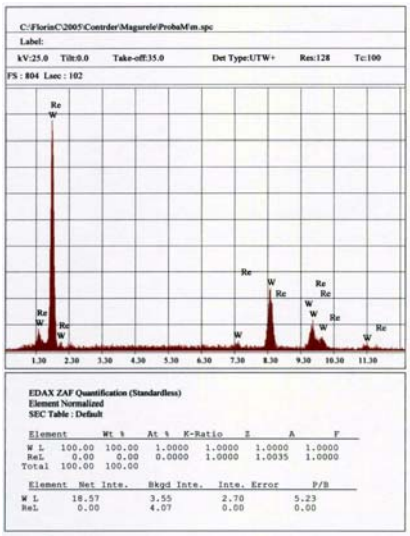

Figure 4. Typical spectrum of EDX analysis of W coating.

Nanoindentation

The W coated samples (graphite substrates 30 mm x 30 mm x 8 mm) were tested using depth sensing indentation tester Fischerscope H100 type. We can notice from the Figure 5 that the tungsten film had significantly higher resistance against indentation than the graphite substrate.

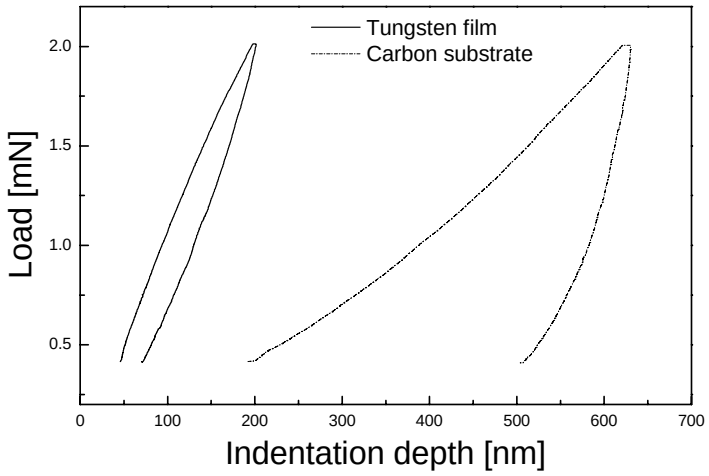

Figure 5. Comparison of loading / unloading curves for W and substrate at maximum indentation load of 2 mN.

The material parameters obtained on the graphite substrate and the tungsten films are listed in Table 1, where: - HU- universal hardness (resistance against elastic and plastic deformation); W_e/W_{tot} – ratio of the elastic indentation work to the total indentation work;

HU_{pl} – plastic hardness (resistance against plastic deformation – equivalent of the so called Vickers hardness; h_{max} – maximum depth at given maximum load- $Y = E/(1-\nu^2)$, where E is the Young's modulus and ν is the Poisson's ratio.

Table 1. Results of the nanoindentation test

Sample	HU[N/mm^2]	We/Wtot[%]	HUpl[N/mm^2]	h_{max}[μm]	Y[GPa]
substrate	158	24,81	205	0,692	6,0
tungsten film	30	0.23	5700	0.250	80,0

Atomic Force Microscopy (AFM)

The AFM measurements have proved the smoothness of the deposited films (however with some droplets as can be seen in Figure 6) with peak to valley roughness in the range of 20-30 nm.

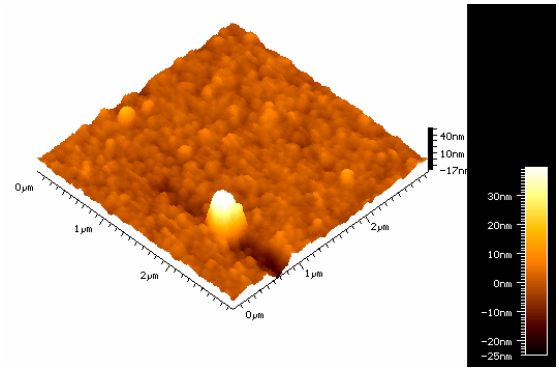

Figure 6. AFM image of the W film obtained by an atomic force microscope in contact mode atomic force microscope.

Auger Electron Spectroscopy (AES)

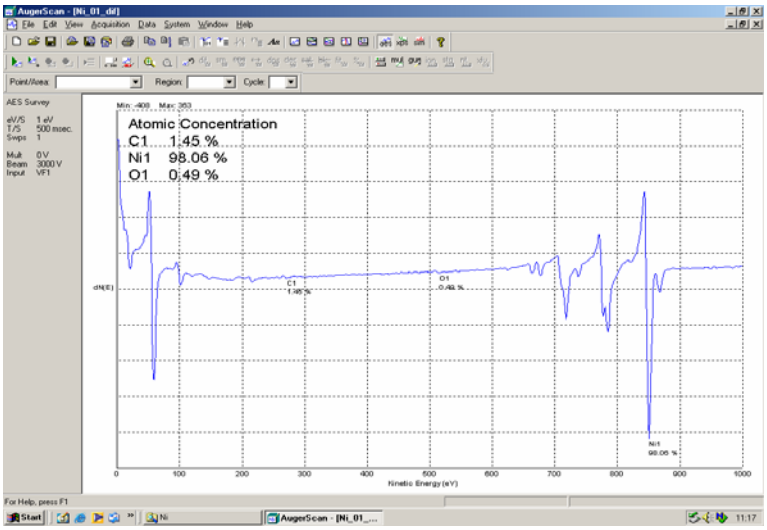

Figure 7. The results of measurements of C and O concentrations in Ni coatings of 2.5 μm using Auger electron spectroscopy (AES) techniques. Values of 1.45%C, 0.49%O have been obtained.

3.3 Characterization of the coated Be samples

Figure 8 shows the photograph of TVA plasma running in Be vapours. The plasma was stable and working parameters were in the range of 500-700V for the discharge potential and 350-400 μA for the discharge current.

Figure 8. TVA plasma running in beryllium vapors.(At Nuclear Fuel Factory in Pitesti facilities)

Coating of graphite samples of 30 mm x 30 mm x 8 mm was performed using the facilities of the Nuclear Fuel factory in Pitesti.

Optical Microscopy (OM)

Figure 9 shows coated and uncoated graphite samples. The Be thickness was found to be in the range of 7.8 ± 0.2 μm.

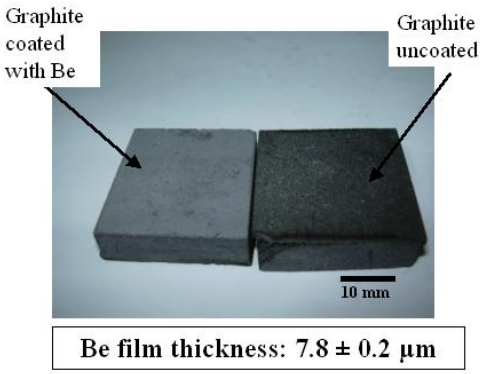

Figure 9. Photograph of a Be coated and an uncoated graphite sample.

Scanning Electron Microscopy

In Figure 10 is shown the morphology of the Be films prepared by TVA obtained by scanning electron microscopy.

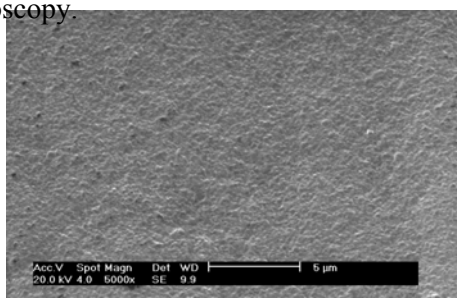

Figure 10. SEM image of the Be film deposited on stainless steel by thermionic vacuum arc (TVA) method

4. Works to be performed in 2006:

Developing TVA technique for the deposition of an heavy-metal interlayer on Be samples and an outer layer of 7-8 microns of Be. The new set of working TVA parameters to be optimized are presented below:

- Distance between cathode and anode;
- Angle between the electron beam and the horizontal line;
- Distance between anode and substrate holder;
- Deposition rate

Investigation of the concentration of C and O and other impurity species in the Be films on the metal interlayer deposited on Be tiles.

Assessment of the surface structure after production and after several months of storage in air.

Measurement of the thickness of the coatings and the adhesion to metal.

To prove that the coatings will be of adequate quality and fully compatible with a metal interlayer on "smart" tiles.

5. Conclusions

The TVA technique was adapted to obtain the first time the coatings W, Ni and beryllium on stainless steel, glass and graphite substrates.

Analyses of the first set of coatings proved that TVA technique can be successfully used to obtain coatings to fulfill the requested conditions.

References

[1] **Lungu C.P., Mustata I., Musa G., Zaroschi V., Lungu Ana Mihaela and Iwasaki K.**: *"Low friction silver-DLC coatings prepared by thermionic vacuum arc method"*, Vacuum 76 (2004) 127.

[2] **Lungu C. P., Mustata I., Musa G., Lungu A. M., Zaroschi V., Iwasaki K., Tanaka R., Matsumura Y., Iwanaga Y., Tanaka H, Oi T., Fujita K.:** *"Formation of nanostructureed Re-Cr-Ni diffusion barrier coatings on Nb super alloys by TVA method"*, Surf and Coat. Techn, 200 (2005) 399.