Comparison of the beryllium films prepared by thermionic vacuum arc and thermal evaporation methods

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1. Introduction
The ITER-like Wall Project, part of the "JET programme in support of ITER" [1-10], to be implemented on JET includes R&D activities to develop a method of depositing Be layers on inconel and an interlayer plus a Be overlay onto Be tiles and characterization of the Be coating purity by surface and structure analysis techniques as well.
Thermionic vacuum arc (TVA) deposition process has been chosen in order to obtain high density of the films [9-11] for the production of the "marker" tiles. The "marker" is a Be tile with a stripe of 2-3 \textmu{}m in thickness of an easily detected heavy metal (e.g. W, Re, Ni) deposited on it as an interlayer, and with a few microns (~ 8-9 \textmu{}m) layer of 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 thickness of the layer from the final surface, for erosion of less than the film thickness. To enable measurement of erosion greater than 9, \textmu{}m there will be notches (10 and 20 \textmu{}m deep) on the tiles.
The major effort in R&D is to produce high-purity layers with optimised composition, and to select the best interlayer material to ensure good adhesion and thermo-mechanical compatibility. The optimisation of the manufacturing process (layer thickness, structure and purity) has been carried out on various substrates: glass, silicon, metals. The optimised marker layers for further testing (including performance under high-heat loads) were deposited on 3 cm thick Be blocks of JET and ITER relevant quality (S65 grade from Brush Wellman).

2. Preparation methods
Two methods of Be deposition have been used to produce test samples: standard thermal evaporation in vacuum and Thermionic Vacuum Arc (TVA) technique [1-2]. The latter one has been selected to prepare 8 \textmu{}m thick films on the “smart”/marker tiles and the former one to produce Be films on inconel tiles.

Fig.1 Set-up for beryllium deposition using thermionic vacuum arc method.
TVA is based on the electron beam-induced evaporation, as shown in the schematic Fig.1. A tungsten hot athode filament surrounded by a Wehnelt cylinder is heated by an external low voltage-high current ac supply. Thermo-electrons emitted from the tungsten filament are accelerated towards the anode. High evaporation rate of the anode material leads to high vapor density in front of the anode. The space density of these particles is high enough to lower the ionization mean free path making it smaller than the cathode – anode distance. As a result, plasma is ignited in that region while the surrounding space is evacuated to below $10^{-3}$ Pa. Under these conditions high purity films of the anode material can be reproduced without reduction or modification.

The coatings of inconel tiles were performed using the vacuum thermal evaporation equipment of the Nuclear Fuel Plant (NFP) in Pitesti. The surface of the samples was prepared before deposition by the standard sand blasting procedure used by the NFP. The schematic arrangement used for beryllium deposition by vacuum thermal evaporation method is schematically shown in Fig. 2.

The evaporation crucible was beryllium oxide and the heating component was a molybdenum wire of 3 mm diameter wounded around the evaporating crucible.

3. Film structure

Images in Fig. 3 and Fig. 4 show the structure of beryllium layers deposited by two techniques: TVA and thermal evaporation, respectovelly. Evaporated films were prepared using the facilities at the Nuclear Fuel Factory in Pitesti, Romania. It is noticed that films obtained by TVA are much smoother than evaporated layers. X-ray diffraction studies have also proven highly ordered crystalline structure of TVA-produced films.

![Fig.2 Set-up for beryllium deposition using thermal evaporation method in vacuum.](image)

![Fig.3 SEM image of Be film prepared by TVA method](image)

![Fig. 4 SEM image of Be film prepared by thermal evaporation method](image)
During depositions, a quartz crystal monitor was used to measure “in situ” the thickness of the deposited layer by TVA method. In order to measure thicknesses larger than 1 μm (the upper limit of the quartz sensor) a cooled holder was designed and built having in the front of the sensor window a disc with a 10 mm diameter hole. An electrical motor working in vacuum drove the disc with 100 rpm, ensuring a factor of 30 between the real thickness deposited on samples and the thickness measured by the thickness monitor. After deposition, the thickness of the layers was measured on the witness samples using a stylus profiler. Steps were produced by masks on the witness surface or by chemically etching a rib in the centre of the sample. Be films thicknesses were in the range of 7.5 ± 0.5 μm.

Atomic Force Microscopy (AFM) measurements, in tapping mode, have proved the smoothness of the deposited films with peak to valley roughness in the range of 300 ± 50 nm for Be coatings (Fig. 5).

The crystalline structure of the deposited films was confirmed by X-ray Diffraction (XRD) analysis. Fig. 6 shows the XRD pattern of the Be coatings on silicon wafer with high intensity and sharpness of the Be peaks. The surface film composition analyzed using an PHI-Perkin Elmer model 3017 Auger spectrometer (Kinetic energy range; 0-3200 eV, resolution; 0.6%) showed less than 12 % oxygen and carbon inclusion into the thin outermost layer. A typical Auger spectrogram is shown in Fig. 7 giving the O, C and Be concentration. The feature around 215 eV corresponds to the Ar implanted by the ion gun used for surface etching prior to the Auger measurement.

The Be coating samples were exposed to stepwise-increased thermal loads from 4.4 MJ/m² to 20 MJ/m² (0.4 MW/m² to 2.6 MW/m²). The tests were done at the starting temperature typically below 100 °C. The maximum surface temperature rose up to around 700 ~ 800 °C. The
temperature distribution was fairly homogeneous at the loaded area and no local overheating was observed by the IR-camera. The oxidation of the coating surface was observed as nano-scale surface roughening of the platelets, showing the potential function of Be layer as an oxygen getter.

4. Conclusions

It has been proved that the TVA and thermal evaporation methods are applicable for Be deposition because they are clean methods of deposition in high vacuum conditions. The mean deposition rates were 5 ± 0.5 nm/s for Be deposition using TVA method and about 1 ± 0.5 nm/s for Be deposition by thermal evaporation in vacuum. The results obtained from the measurements made on the Be films proved that the structures present:

- Smooth surfaces
- High density
- High purity of the films
- Good adhesion of the films to the substrate.
- Good resistance to thermal tests.

References