PRODUCTION OF BERYLLIUM COATINGS FOR INCONEL CLADDING AND BERYLLIUM TILE MARKERS FOR THE ITER-LIKE WALL PROJECT-1 JW6-TA-EP2-ILB-01 (EFDA/06-1526)

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1. Objectives

Manufacturing of 8 μm Be-coatings of Inconel Cladding and Beryllium tile Markers for installation in JET

2. 8 µm Be-coatings of inconel cladding tile production preparation

In order to prepare the documents for the qualification of the thermal evaporation method for beryllium coating of the inconel cladding tiles, the specialists from the Nuclear Fuel Plant (NFP), Pitesti and National Institute for Laser, Plasma and Radiation Physics, Magurele, Bucharest, Romania prepared the complete set of the documents that were approved by JET, Culham:

- Manufacture Engineering Procedure (MEP-JET-01)
- Manufacture Operating Procedure (MOP-JET-01)
- *QUALITY PROGRAMME FOR BE-JET PROJECT (QPP-JET-01)*
- QUALITY INSPECTION OF BERYLLIUM COATING (QVI-JET 01)
- QUALITY PLAN PC_JET
- SRP-JET-01- Reconstruction of Be coat thickness
- SRP-JET-01- Reconstruction of INCONEL pieces Be coated

To improve the thermal evaporation deposition method, the specialists from NFP Pitesti tested the evaporation system in order to simulate the complicated tile surfaces. There have been designed and manufactured policarbonate moc-ups at 1:1 scale in order to simulate the deposition on real tiles. The design of the rotating cupola support of the tiles during evaporation has been also optimized. The cupola and jigging devices of the inconel tiles are shown in Figures 1 and 2. The design of the cupola and jigging devices was performed using the AUTOCAD software.



Figure 1. The design of the new cupola for supporting inconel tiles



The preliminary tests for the qualification of the Beryllium films deposition method on inconel tiles have been started using the special designed cupola at NFP Pitesti

2.1 Thickness tests for the beryllium layers

There were coated with Be and measured 9 witness samples positioned at lower, middle and upper part of the new cupola. The MICRODERM backscattering based apparatus of the NFP Pitesti was used to perform the measurements. The measured values were within the thickness margins imposed by the project (Be films of $7 - 9 \mu m$ thickness):

- ✓ Average thickness: 8,41 µm
- ✓ Minimum thickness : 7,65 μ m
- ✓ Maximal thickness : $9 \, \mu m$

2.2 Adherence tests:

Using a hard steel device sharpened at a 30° angle, parallel grooves, at 2 mm distance each to other, were scratched on the beryllium coated surface, so that the Beryllium film to be fully penetrated. The adherence was good if the film between the grooves was not completely removed after the passage of the sharpened steel device.

As presented in Figures 3-6, the adherence of the beryllium layers was very good.



Figure 3. Coating of beryllium on inconel in the left-up side

Figure 4. Coating of beryllium on inconel in the right-up side.





Figure 5. Coating of beryllium on inconel in the left-down.

Figure 6. Coating of beryllium on inconel in the right-down side.

3. Beryllium tile markers production preparation

For the deposition process, using thermionic vacuum arc (TVA) method [1] it was designed and manufactured a new evaporation system and a new sample heating rotative system to improve the Ni and Be deposition system and to ensure increased adhesion and uniform depositions. The technical drafts were previously sent for approval to JET.

As a result of JET experts discussions and amendments there have been finalized the following documents for thermionic vacuum arc coating method:

- Marker coating process procedure
- Inspection procedure for marker coated tiles)
- Quality plan for markers production
- Quality program for markers production
- Handling, packing and transport specification for markers

During October-December 2007, have been performed activities for the qualification of the deposition method for the Be-Ni test samples, which involved the deposition on parallelepiped shape stainless steel blocks coated with a Ni layer of $2\pm 0.5\mu$ m and then a Be layer of 7-9 μ m.

For this purpose, it was designed a detailed program for Ni/Be films coatings, with a view of improving their operating parameters and also the substrates temperature during samples sputtering cleaning in Argon plasma glow discharge. After the experimental preliminary depositions, it was decided that this temperature to be of 300°C±20°C; accordingly, adequate modification of the conditions included in the software that controls the system parameters during the deposition was made.

For monitoring the substrate temperature during Ni and Be coatings, it was designed a Germanium windows viewing system allowing the monitoring of infrared radiations in the range $8-14 \mu m$ using a thermovision sensor device.

Figure 2 shows the temperature distribution on the test sample surface before starting the sputtering cleaning treatment in Argon glow discharge.



Figure 7. The calibration curve of the thermovision device.



Figure 8. Temperature distribution on the test sample surface ($94\pm4^{\circ}C$ thermovision device values, conforming with the real values of $300^{\circ}C\pm20^{\circ}C$)

The prepared films, coated on the test surface, were analyzed using a GDOES device (glow discharge optical emission spectroscopy) in support of Horiba-Jobin Yvon Company. Figure 9 shows the depth profile of the coated layers composition, including their interface and the base material. There have not been identified any carbon impurities in the beryllium layer. In the nickel layer, carbon concentration was below the allowed limits: <5%.



Figure 9. Ni-Be film compositional profile, coated on stainless steel substrate.

4. High-heat flux tests.

As a collaborative action a series of marker samples produced at NILPRP were analysed using several material analysis techniques [2-6] before and after high-heat flux (HHF) testing with an electron beam in the JUDITH facility at FZJ Juelich, Germany. HHF screening tests allowed the determination of power and energy density limits deposited onto the surface until the damage to a marker occurred. A cyclic test served to assess the thermal fatigue under repetitive power loads. The major results may be summarised as follows: (i) the markers survived without noticeable damage power loads of 4.5 MW m⁻² for 10 s (energy density 45 MJ m⁻²) and fifty repetitive pulses performed at 3.5 MW m⁻² each lasting 10 s, i.e. corresponding to the total energy deposition of 1750 MJ m⁻²; (ii) in both cases the surface temperature measured with an infrared camera was around 600 °C; (iii) the damage to the Be coating occurred at power loads of 5 MW m⁻² for 10 s.

Plots in the Figure 10 show depth profiles obtained by secondary ion mass spectrometry (SIMS) for two marker coupons: (a) unexposed to heat loads and (b) after HHF test carried out

168.2 °C

for 10 s at power density of 4 MW m⁻², i.e. total energy density of 40 MJ m⁻². Both profiles are quite similar (Be coating thickness approximately 9.5 μ m) thus indicating that the applied power loads neither damage the coating nor cause intermixing of Be and Ni. There are some impurity species (Al, Si, Fe) but their content is below 1 % as determined by ion beam analysis, energy and wavelength dispersive X-ray spectroscopy. Figure 11 shows a metallographic cross-section of the HHF tested coupon. A clear separation of beryllium and nickel proves the durability of the coatings.



Figure 10. SIMS depth profiles for two markers: a) "as produced"; b) HHF tested at 40 MJ m⁻².



Figure 11. Metallographic cross-section of a marker sample after cyclic test (50 pulses) at 3.5 MW m⁻².

To check the adherence and thermo-mechanical properties of the Be layers deposited on inconel by thermal evaporation method, a number of test samples were exposed to high power loads in JUDITH [6]. The screening test was carried out in the range from 0.4 to 2.6 MW m⁻² in pulses lasting up to 11 s. In the cyclic test fifty consecutive 10 s pulses were performed at the power of 1 MW m⁻², i.e.10 MJ m⁻² per pulse. Figure 5 shows the layer structure before (a) and after the test at the power load of 1.8 MW m⁻² for 11 s corresponding to the energy load of 20 MJ m⁻² (b). In both cases the coating topography is nearly identical. It proves that no damage (e.g. melting or exfoliation) is caused by energy loads exceeding at least three times the level characteristic for a regular plasma operation. As assessed, the coating on inconel would melt at energy loads exceeding 30 MJ m⁻² [6].



Figure 12. Beryllium coatings on inconel: (a) "as produced"; (b) after HHF test of 20 MJ m².

5. Conclusions

The required documentations for the production of the beryllium coatings on the inconel tiles and markers to be installed on the first wall of JET, were elaborated. A new evaporation system, a new cupola and inconel jigging devices were designed and manufactured. Witness samples for uniformity and adherence tests were also produced.

Samples were tested at high-heat flux using JUDITH facility at FZJ Juelich, Germany. The results of samples analysis before and after HHF testing indicate that the beryllium coatings on inconel and marker limiters should withstand conditions of the regular JET operation without melting, exfoliation or phase transformation. This is particularly important in the case of the marker tiles for long-term Be erosion studies in the main chamber. However, local melting of Be tiles (with and without markers) cannot be excluded in the case of excessive power loads experiments. In this case the extent of erosion will be assessed by mechanical methods. The scientific and technical program has led to the selection of methods for a large-scale manufacturing of protective coatings on the inner wall cladding and marker tiles. The thickness markers, prior to their installation in JET, will be determined by means of ion beam analysis methods.

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