Production of Beryllium Coatings for Inconel Cladding and Beryllium tile Markers for the ITER-like Wall project
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1. Objectives

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

2. Beryllium tiles Markers coatings

The deposition method used is based on vacuum evaporation. This has the advantage of not having any buffer gas inside the deposition chamber, which makes the gas inclusions in the structure of the thin film to be practically absent. The method used in the preparation of the Ni and Be films is called thermionic vacuum arc (TVA) [1-2]. The principle of this method is the ignition of the plasma in pure vapors of the material. The energy introduced in the system for the ignition of the plasma is given simultaneously by an electronic gun and a high voltage source. The vapors are obtained by heating the anode material using an accelerated electron beam between the electrodes. Due to high adherence degree, high density, and purity of the films, the thermionic vacuum arc method is highly efficient. Another advantage of this method consists in the high degree maneuverability of the ions energy. This is because the TVA plasma is localized and the ions are moving in a straight line in the vacuum up to the walls/substrates that are connected to the ground. The energy is given by the plasma potential, easy to control by the processing parameters: arcs current, filament current and the anodic voltage.

The experimental setup of the thermionic vacuum arc consists in a ground cathode with a hot tungsten filament, surrounding by a Wehnelt cylinder which focuses the electrons toward the anode crucible that has inside the material for deposition.

By applying a positive voltage on the anode, the electrons are accelerated making the heating of the anodes' material. The first vapors appear after a short period of time. A slightly increase of the voltage accelerates the electrons creating more vapors. Plasma appears in pure metal vapors when a specific value of the applied voltage on the anode is reached. The thermionic vacuum arc plasma is localized. The ions created inside the plasma are accelerated also toward the chambers walls due to the potential difference between the plasma and the ground connected walls. The neutral atoms move outside the plasma due to the pressure gradient. The film is formed by both ions and neutral atoms. Rejection of the electrons, which are going toward the substrate, is made by negatively polarizing the samples. The interest parameters monitored and controlled during the deposition are the following:

- I_f – the current value of the filament
- I_a – arc current during the deposition
- I_b – bias current value during the deposition

- U_a – arc voltage during the deposition
- U_b – bias voltage value applied on the substrate
- Deposition rate
- The thickness of the thin film

The beryllium has been evaporated without a special crucible, being heated by an electronic bombardment at the surface. A cooled stainless steel anode had a cylinder of the beryllium in it. The evaporation of the material took place only at the superior face, while the inferior remained in a solid state. This method of the anode evaporation reduces to zero the contamination of the growing film. The deposition rate had values of 6-7 nm/s. For the deposition of nickel the evaporation has been made using a TiB_2 crucible. The ions generated inside the plasma have been accelerated using a bias voltage source having a value of -700V and 10-15 mA the current. The experimental setup outlined in Fig.1 was used for both deposition of nickel and beryllium.

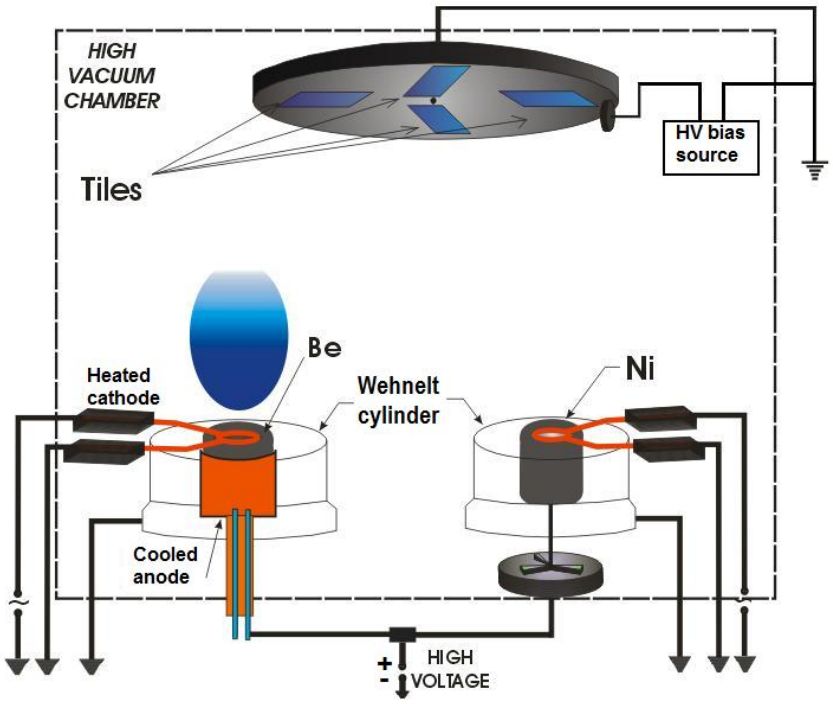


Fig.1 *Nickel and Beryllium deposition experimental setup*

2.1 Evaporation process

The process consisted in five steps:

1. The First step was to obtain a low pressure inside the discharge chamber of about 4-6*10^{-6} torr. An inside oven was heated up to 300^0-450^0C in order to eliminate the gases adsorbed on the walls.
2. The second step was to ignite an argon gas glow discharge for cleaning the tiles. The pressure of argon gas inside the vacuum chamber was set to 2-3*10^{-2} torr. By applying a negative bias voltage on the tiles, the glow discharge was localized on the surface area of the tiles. This process took 15 - 20 minutes, depending on the values of the voltage and the current. The bias voltage applied on the tiles was -700 V, with an electrical current of 50 mA.
3. The third step was the Ni coating of the tiles. The filament heating current was 45-50A and the anode voltage 1.5kV. The process was stopped when the "in situ" quartz balance devices reached a thickness of 2μm.

4. The fourth step was beryllium coating of the tiles. A filament heating current of 50A-55A and 1.7-1.9kV anode voltage were used. The process was stopped when the "in situ" quartz balance devices reached a thickness of 7 μm.
5. Final step consisted in the cooling down of the installation. The coated tiles have been removed when the temperature was under 50^0C.

During the entire coating process, a negative bias voltage was applied on the tiles, with a value of -700V (at a current of 5-15 mA), which ensured a compact coating by rejecting the electrons and accelerating the positive ions from plasma [3].

2.2 Pre-production run

Before coating of the real tiles, an experimental pre-production run was performed using three Be blocks, to test the adherence, thickness and process procedures. The positioning of the tiles is shown in Fig.2

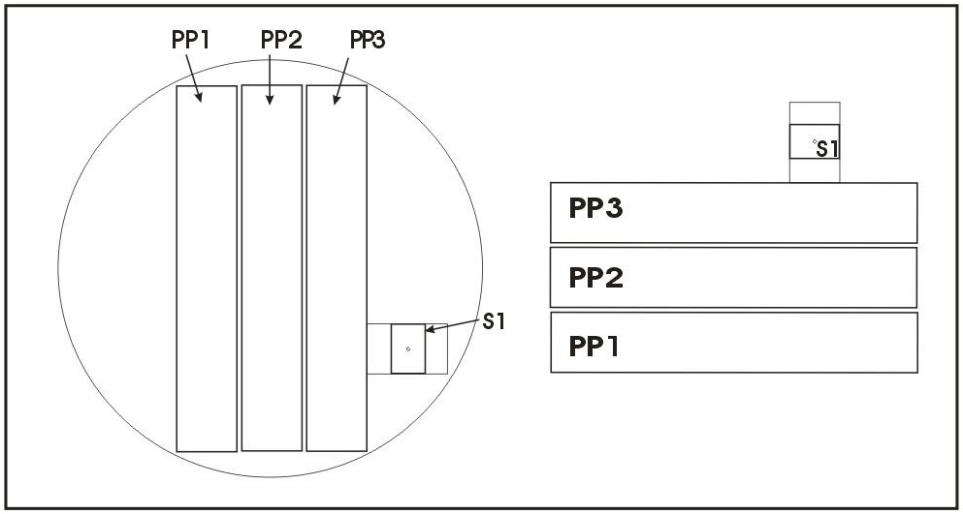


Fig.2 *Be tile positioning in the pre-production run process*

Figs. 3 and 4 show the tiles before and after the Ni+Be coating process, in the pre-run experiment.

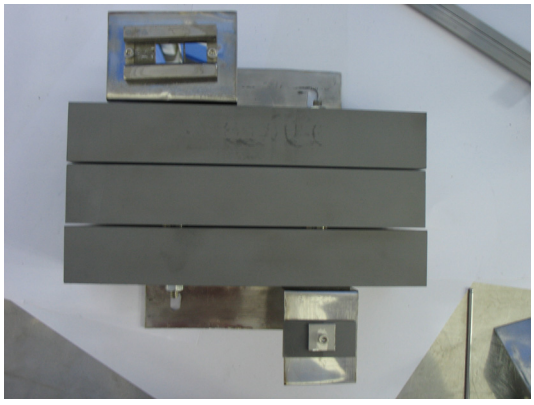


Fig.3 *Be tiles* ***before*** *Ni+Be deposition*

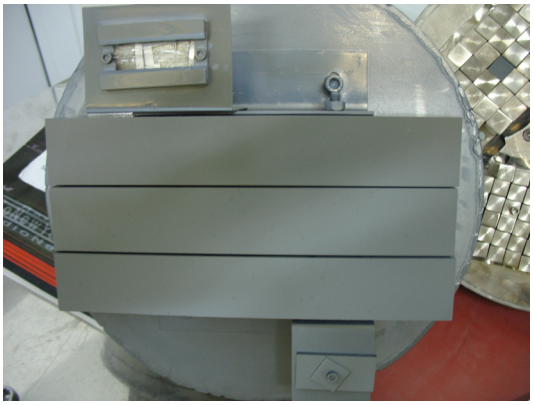


Fig.4 *Be tiles* ***after*** *Ni+Be deposition*

An adherence test of the Be blocks have been made. The test showed that the Ni+Be coating was very adherent to the Be tiles used as substrates. Figure 5 presents the

scratching tracks of the tiles produced by the special device engineered and manufactured at UKAEA. No delamination or pell-of can be observed on the prepared films.

Fig.5 Scratching traces of the Be test tiles after the adherence test.

2.2 Production run

The parameters used in an actual deposition of Ni+Be process for the Be tiles is being presented in the Technological record, an official document, accompanying every deposition batch. Together with the tiles **F70100061, P F70100107,** were coated the witness samples **WP-1, WP-2, WP-3.** The positioning of the tiles inside the vacuum chambers is presented in Fig.6

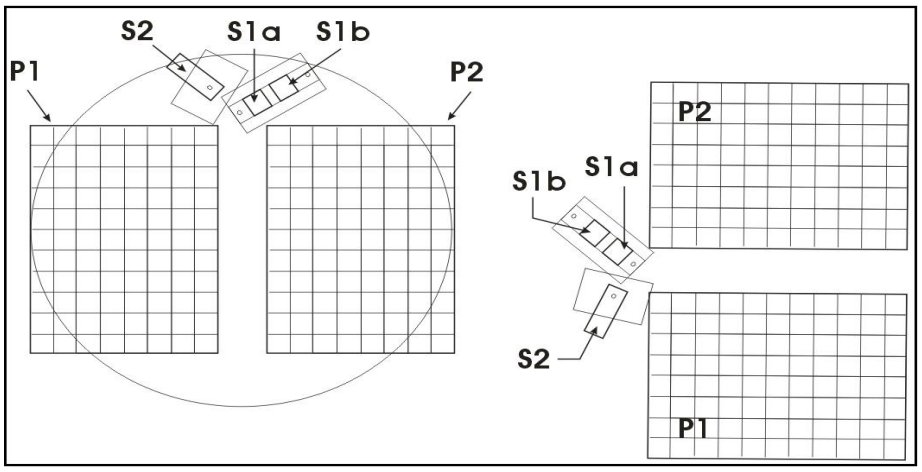


Fig.6 Tile positioning inside the vacuum chamber

TECHNOLOGICAL PARAMETERS used in the process of Ni+Be coating:

Technological Record				
Loading of the Be tiles				
Tile reference number~	**P F70100061**	**P F70100107**		
Tile Position reference~	**P1**	**P2**		
Witness sample reference numbers~	**W P-1**	**W P-2**	**W P-3**	
Sample Position reference ~	**S1a**	**S1b**	**S2**	
Loaded holder, Photograph~	Jpeg No. IMG_2288/3623			

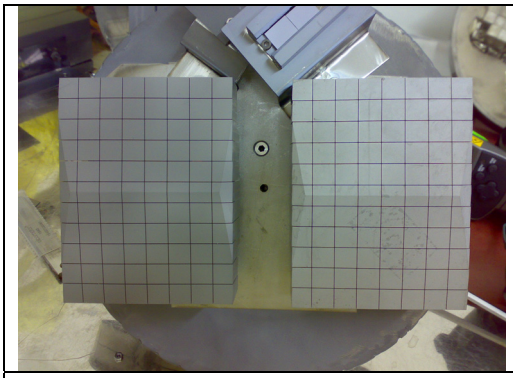
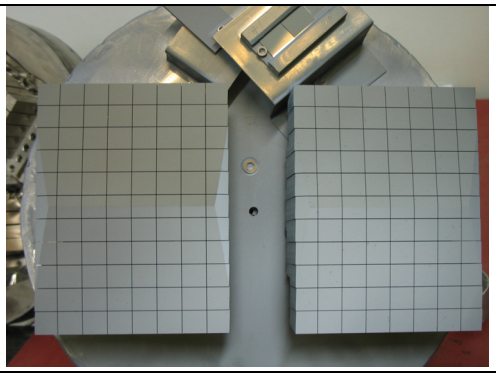
Outgassing
Oven temperature set to 450deg C
Target chamber pressure less than $5*10^{-5}$torr

Time (mins)	Oven temperature deg C	Tile temperature		Pressure (x 10^{-6} torr)
		reading	deg C	
0	**20**		**20**	**4**
30	**427**		**102**	**4.6**
49	**444**		**151**	**4**
88	**447**		**200**	**3.4**
118	**447**		**223**	**3.2**
152	**446**		**245**	**3**
179	**445**		**262**	**2.9**
197	**445**		**266**	**2.8**

Glow Discharge Cleaning
DC Supply ~ Vdc=900V, Idc=50 mA

Time (mins)	Oven temperature	Tile temperature		Pressure (x 10^{-2} torr)
		reading	deg C	
0	**440**		**270**	**4**
5	**317**		**243**	**2.1**
10	**236**		**228**	**1.9**
15	**225**		**222**	**2**

Substrate heating turned off.	
6	**Nickel Coating**
	Cathode filament heating. Vac ____**50**____A
6(a)	Oven pressure (target <5 x 10^{-5}) ~ start **5.5**x **10^{-6}** Torr, end **4.8** x **10^{-6}** Torr
6(b)	Tile surface temperature ~ start **253** deg C, end **398** deg C
6(c)	DC discharge Supply ~ Vdc= **1500V**, Idc= **1.33A**
6(d)	DC bias Supply ~ Vdc= **700V** , Idc= **15mA**
6(e)	Length of time for Ni Coating~ (Target 120mins) **40** mins 14:20→15:00
6(f)	Thickness reading on QMB (Target 2000=2μ) ~ **1.719~2 μm**
7	**Beryllium Coating**
	Cathode filament heating. _____Vac **53.4** A
7(a)	Oven pressure (target <5 x 10-5) ~ start **3.8x 10^{-6}** Torr, end **3.6x 10^{-6}** Torr
7(b)	Tile surface temperature ~ start **370** deg C, end **380** deg C
7(c)	DC discharge Supply ~ Vdc= **1350 V**, **Idc= 870 mA**
7(d)	DC bias Supply ~ Vdc= **700 V**, Idc= **10 mA**
7(e)	Length of time for Be Coating~ **40** mins 15:10→15:50
7(f)	Thickness reading on QMB (Target 7000=7μm) ~ **6.110~7.5 μm**
8	**Stopping the deposition process**
8(a)	Tile temperature when exposed to air Target 50 deg C) **53** deg C

	
IMG_2288 (**Before** Ni+Be deposition)	IMG_3623 (**After** Ni+Be deposition)
PRODUCTION-Run 1/22.10.2009	

This way, the 22 beryllium tiles were coated with 2-3 μm Ni and 7-9 μm Be. After coatings, the tiles were double heat sealed, packed in carton crates and put together in a wooden box. The plastic bag surfaces, carton crates and wooden box were smeared and the smear products (25 mm in diameter paper filters) were sent to NFC-Pitesti in order to analyze the Be contamination.

3. Conclusions

The method used in the preparation of the Ni and Be films is the thermionic vacuum arc. The technique used the coating from two different sources, Nickel and Beryllium, without opening the vacuum chamber between these two depositions. The compactness, adherence and purity of the films were assured by the presence of both Nickel and Beryllium evaporation systems. Another advantage of using this method is controlling the ion energy, electron flux, and deposition rate by external plasma parameters.

Thermionic vacuum arc method was used to obtain layers of 2-3 μm Ni and 7-9 μm Be for coating on a number of 22 marker tiles, part of the ITER-like wall to be installed at JET.

References

[1] C. P. Lungu, I. Mustata, V. Zaroschi, A. M. Lungu, A. Anghel, P. Chiru, M. Rubel, P. Coad G. F. Matthews and JET-EFDA contributors, Beryllium Coatings on Metals: Development of Process and Characterizations of Layers, Phys. Scr. T128 (March 2007) 157–161

[2] C. P. Lungu, I. Mustata, V. Zaroschi, A. M. Lungu, P. Chiru, A. Anghel, G. Burcea, V. Bailescu, G. Dinuta, F. Din, Spectroscopic study of beryllium plasma produced by thermionic vaccum arc Vol. 9, Iss. 4, 2007, J OPTOELECTRON ADV M, Vol. 9, No. 4, 2007, 884-886.

[3] Anghel, I. Mustata, C. Porosnicu, C. P. Lungu, Influence of the bias voltage on the formation of beryllium films by a thermionic vacuum arc method, Journal of Nuclear Materials, Volume 385, Issue 2, 31 March 2009, p. 242.