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Influence of the bias voltage on the formation of beryllium films by a thermionic vacuum arc method

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ABSTRACT

Beryllium is intended to be used as suggested material for the first wall in the thermonuclear power plants. Some tiles of the first wall will be of inconel coated by a beryllium layer that must be adherent to the substrate and have a compact structure in order to resist as much as possible the dramatic interaction with the high energetic plasma particles, ions, electrons and neutrons. Applying bias voltages (−200 to + 700 V) on the substrates, the morphology of the prepared Be layers using the original thermionic vacuum arc method developed at NILPRP was controlled in order to obtain smooth surfaces, free of holes and lamellar structures. The prepared films were studied and characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS) and Auger spectroscopy (AES). The films prepared using negative bias voltages were found to be more compact and smooth with an average roughness (R_{ms}) of 7 nm.

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1. Introduction

Since beryllium is intended to be used as a component of the first wall for the International Thermonuclear Experimental Reactor (ITER) [1–4] it is important to study the properties of this material. As a part of these studies, some tiles of the first wall will be of inconel coated by a beryllium layer that must be adherent to the substrate and have a compact structure so as to resist as much as possible the dramatic interaction with the high energetic plasma particles, ions, electrons and neutrons. The erosion rate of the Be layers, as well as of the bulk tiles will be determined for Be 'smart tiles' made of bulk Be, Ni interlayer and Be overlay [5].

The thermoionic vacuum arc (TVA) method was adopted for the deposition of the beryllium marker tiles. This technique, developed at the National Institute for Laser, Plasma and Radiation Physics, Magurele-Bucharest, Romania represents an extension of the electron beam evaporation method [6,7]. Thermo-electrons emitted by an externally heated cathode and focused by a Wehnelt focusing cylinder are strongly accelerated towards the anode whose material (Be) is evaporated and a bright plasma is ignited by a high voltage (1–5 kV) DC supply. Applying bias voltages (−200 to +700 V) on the substrates, the morphology of the prepared layers was controlled in order to obtain smooth surfaces free of holes and lamellar structures.

The prepared films were studied and characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS) and Auger spectroscopy (AES).

2. Experimental set-up

The TVA method principle scheme presented in Fig. 1 shows an A.C. power supply providing the current for heating the cathode filament (I_{fil} = 0–120 A, at 0–24 V) and an HV power supply of high voltage (U_{dis} = 0–5 kV) at 0–2 A current (I_{dis}). A TiB_2 crucible containing Be metal is surrounded by a focusing Wehnelt cylinder. The circular tungsten filament acting as externally heated cathode was placed at 5–10 mm above the anode. The evaporation of the Be metal was performed in high vacuum conditions (about 10^{-3} Pa and less). The necessary local pressure for plasma ignition in pure Be vapors was determined to be of the order of 10–100 Pa.

The thermo-electrons produced by the heated cathode are able to build up plasma by electron-metal atoms collisions in the space above the anode. The new electrons generated in the plasma together with the original ones emitted by the cathode enhance once more the anode evaporation and produce high quantity of Be ions. Usually the fall in the cathode potential was in the range of 300–600 V and therefore the plasma potential in comparison with ground ensured generation of energetic ions, which collide with the substrate. Using the TVA method the deposition rates were in the range of 4–20 nm s^{-1}. The heating currents for the tungsten filament heating given by the A.C. low voltage/high current (I_{fil}) supply were in the range of 30–50 A. The necessary voltage for plasma

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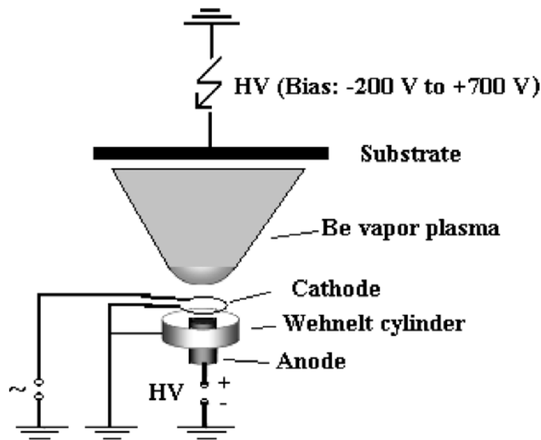

Fig. 1. Thermionic vacuum arc principle scheme.

ignition was in the range of 2–3 kV and the stable vacuum arc discharge was running at U_{dis} = 800–1200 V at I_{dis} = 1.5–2.0 A.

In order to study the influence on the morphology of the films, four different biases were applied on the substrates relative to the ground during the film growth: −200, −100, +400 and +700 V. The applied substrate bias was the only difference in the deposition process; the other parameters, like chamber base pressure, filament current, the discharge voltage and current were the same.

The surface morphology of the prepared film was characterized using an environmental scanning electron microscope PHILIPS ESEM XL 30 TMP and an atomic force microscope AFM Q-Scope 350 from Quesant. The surface film composition was analyzed using an PHI-Perkin Elmer model 3017 Auger spectrometer (kinetic energy range; 0–3200 eV, resolution; 0.6%) and the chemical state of the beryllium films was analysed by X-ray photoelectron spectroscopy (XPS), using a VG ESCA III Mk 2 instrument. Photoelectrons were excited by an Al Kα (1486.6 eV) X-ray source operating at 250 W/12 kV, 0.65–0.90 eV resolution.

3. Results and discussion

The films used in this study were coated using the standard TVA deposition procedure by means of beryllium plasma parameters:

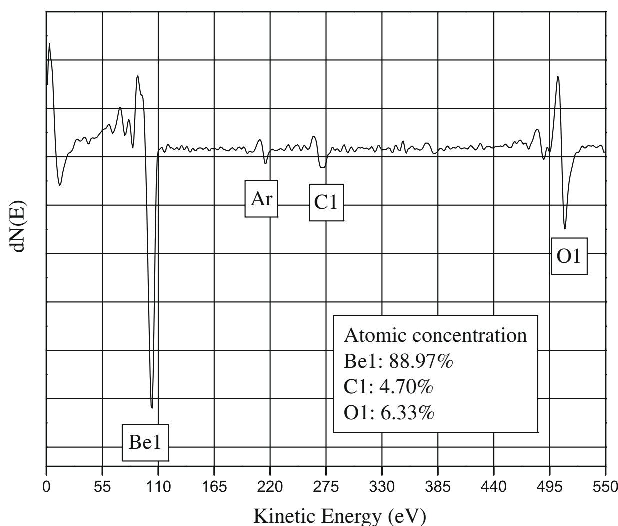

Fig. 2. Typical Auger spectrogram of the analysed Be film.

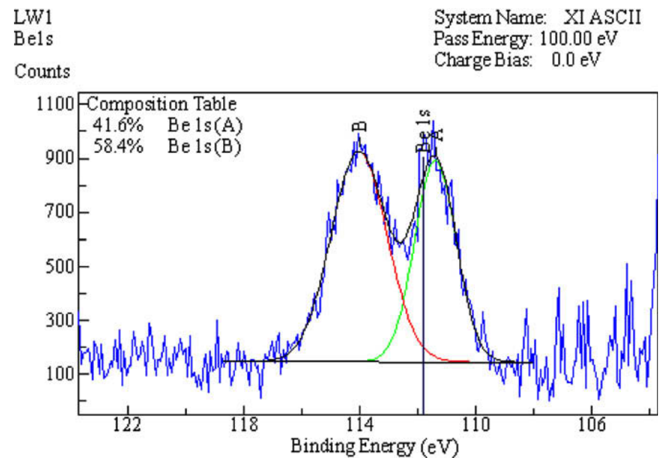

Fig. 3. XPS spectrum of the Be film coated on silicon. XPS Be1s spectrum is represented by two peaks: peak A situated at 111.37 eV (peak area: 335.45 counts) showing a binding energy corresponding to BeO and peak B situated at 114.00 eV (peak area 471, 199 counts) assigned to metallic Be.

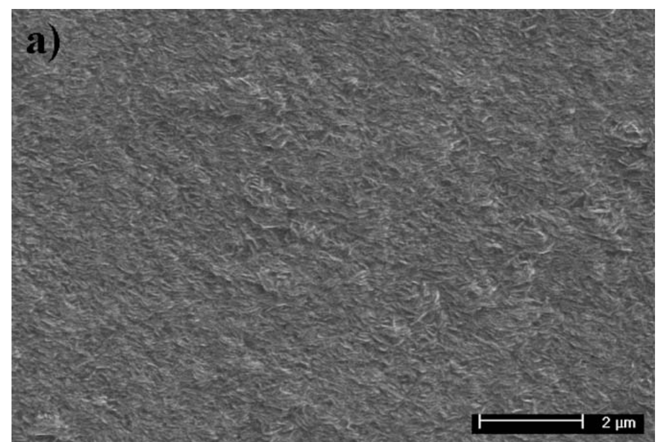

Fig. 4a. SEM image of the Be film deposited on graphite using negative bias (−200 V).

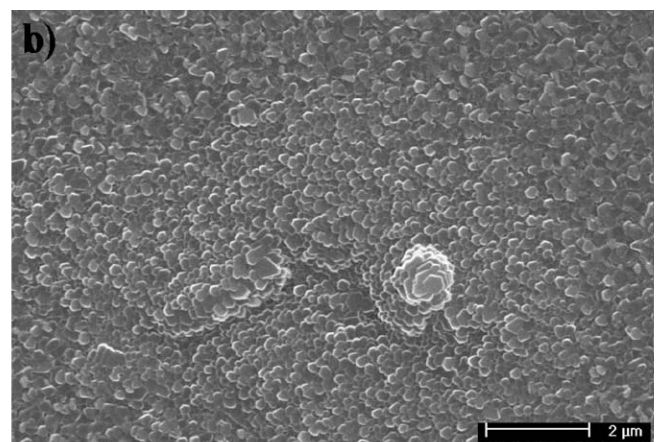

Fig. 4b. SEM image of the Be film deposited on graphite using positive bias (+700 V).

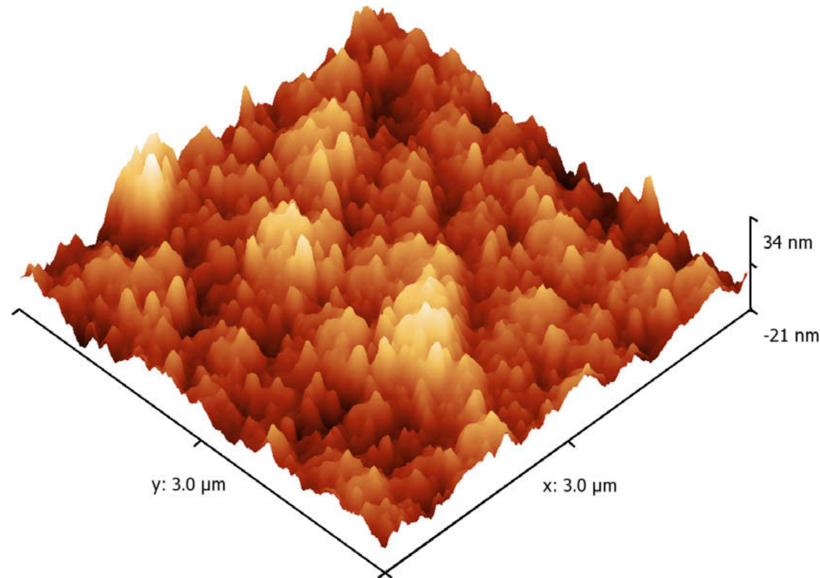

Fig. 5a. AFM image of the negatively biased (−200 V) beryllium films deposited on Si substrate.

base chamber pressure of 1×10^{-3} Pa, I_{fit} = 45 A, U_{dis} = 1000 V and I_{dis} = 1.5 A. The bias applied on the substrate during the deposition was varied between −200 and +700 V.

The surface film composition of the samples prepared under different bias voltages were analyzed using a PHI-Perkin Elmer Auger spectrometer (kinetic energy range: 0–3200 eV, resolution; 0.6%). Fig. 2 shows a typical Auger spectrogram giving the O, C and Be concentration. The atomic concentration of C and O were found to be less than 10% and 5%, respectively. The feature around 215 eV corresponds to the Ar implanted by the ion gun used for surface etching prior to the Auger measurement.

The XPS spectrum of the Be films (Fig. 3) revealed two peaks of Be 1s; the Be 1s peak centred at 111.37 eV (metallic Be: 111.8 eV) corresponding to the metallic Be and the peak centred at 114 eV assigned to BeO, demonstrating the sensitivity of the Be film surface to the oxidation of the Be films prepared at different bias voltages.

The films deposited using negative bias were more adherent to the substrates than the ones grown using positive bias, most of the samples grown using a positive bias applied on the substrate pealed off a short time after opening the deposition chamber. This behavior could be explained by the fact that applying a negative bias on the substrate the beryllium ions generated in the TVA plasma were accelerated towards the substrate while applying a positive bias, the Be ions were decelerated and even rejected depending on the bias value. The samples deposited using negative substrate bias did not fail even after a scratching test.

The scanning electron microscopy (SEM) images show that the morphology of the films grown using positive bias were similar to those films grown by thermal evaporation [8]. Figs. 4a and 4b show the SEM images of beryllium films grown using negative bias (−200 V) and positive bias (+700 V), respectively, applied on the substrate during the deposition process. The films prepared under the negative bias were smooth, compact and adherent.

AFM measurements revealed also that all the films deposited applying a negative bias were smoother than the ones grown using positive bias applied on the substrate.

Figs. 5a and 5b show the AFM images of Be films grown on Si using negative and positive biases, respectively. The average roughness (R_{ms}) of the films grown under negative bias was 7 nm in contrast to the large R_{ms} values (19 nm) of the films prepared under positive bias.

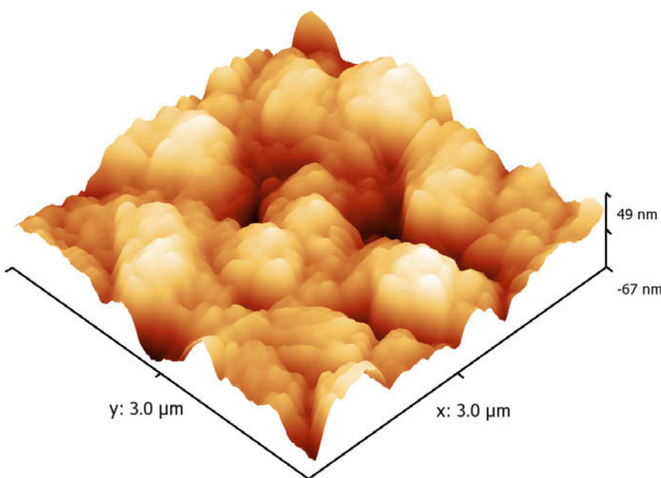

Fig. 5b. AFM image of the positively biased (+700 V) beryllium films deposited on Si substrate.

4. Conclusions

It was observed that the Be films grown using a positive substrate bias presents poor adherence and large R_{ms} roughness while the samples deposited using negative substrate bias did not fail even after a scratching test. In addition, the SEM and AFM measurements revealed that Be films obtained were adherent, compact and smoother when applying a negative bias on the substrate during the deposition.

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