

CHARACTERIZATION OF FUEL RETENTION IN ITER RELEVANT MIXED MATERIALS

TW6-TPP-RFTMIX

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Objective: Provision of Be-coated W, Graphite and CFC specimens for implantation/retention studies

One of the major issues related to the operation of ITER concerns the issue of tritium retention, which is linked directly to the material choice for ITER plasma facing components. Most studies of fuel retention (and removal) have been focused, so far, on the pure materials to be used in ITER, namely C, W and Be.

The experiments carried out within this task characterized the fuel retention properties of the foreseen mixes of materials to be formed in ITER and as far as possible, in conditions close to those expected in ITER: (i) the mixed-materials have included possible combinations of C, W and Be; (ii) deuterium (to simulate the behavior of tritium) was introduced in the mixed material layers by ion implantation at low energy (~200 eV) similar to the energy range expected at the ITER divertor.

Following implantation, the deuterium content and depth profile of the deuterium layer was determined and was compared to the depth profile and chemical composition of the mixed-material layer by surface analysis techniques. Other techniques, such as Thermal Desorption Spectroscopy (TDS), were applied to determine quantitatively the absolute deuterium retention in the samples and, from this technique, the baking temperature which would be required in ITER in order to remove the retained fuel in these mixed-material layers by heat treatment.[1]

The production of Be coated C and W samples for studies of hydrogen retention was performed using the original technology of Thermionic Vacuum Arc (TVA) developed at NILPRP, Magurele-Bucharest, Romania.[2]

1. Preparation of Be-coated W, Graphite and CFC specimens for implantation/ retention studies

The Be-coated W, Graphite and CFC specimens for implantation/retention studies were made in four series (one series for each substrate temperature of 200, 300, 400 and 500°C). For each series 8 specimens of each substrate (polished CFC and mirror polished W and graphite of 15mm x 15 mm x 1 mm) were used. Also, for each deposition series, witness samples made of the same materials were used for comparing the morphological and compositional studies like AFM, SEM and EDS.

The substrates delivered to EURATOM MEdC Association by IPP Garching, were coated with Be films of 20 -100 nm and sent to IPP for fuel retention tests.

The impurities concentrations of the witness samples Be/CFC and Be/graphite deposited at 200 °C were extracted from Electron Dispersive X-ray Spectroscopy (EDXS)

measurements. More impurities were found on Be/CFC specimens (2.17 at%O, 0.13at%Mg, 0.2 at% Al and 0.11 at%Si) compared with those of the Be/graphite specimens (2.47% O) prepared at 200°C substrate temperature.

At the request of the IPP Garching, W films deposited on Be substrates (15mm x 15 mm x 1 mm) at RT and 450°C temperature were prepared for D retention studies. Tungsten films were prepared using the following parameters: the filament current ($I_f = 60$ A), the discharge voltage: 1200V \pm 100V, the current of the discharge: 1800mA \pm 200 mA.

1.1. Influence of the applied bias on the substrate during the coating process

In order to study if the morphology of beryllium films is influenced by applying a bias on the substrate during the coating process, films having hundred nanometers thickness were prepared using the Thermionic Vacuum Arc method. The substrates coated with beryllium consisted in tiles made of materials relevant to ITER technology like graphite, CFC and tungsten. The coated samples were exposed to deuterium ion beam in order to study the deuterium retention.

The films used in this study were coated by the standard TVA deposition procedure using a circular tungsten cathode using beryllium plasma parameters: base chamber pressure of 1.2×10^{-5} Torr, $I_{\text{filament}} = 45$ A, $U_{\text{discharge}} = 300$ V and $I_{\text{discharge}} = 1.5$ A. We applied on the substrates during deposition potentials between -200 V and +700 V. These potentials were applied using a special voltage source with respect to the ground potential. Taking into account that the TVA plasma potential is of some hundreds volts above the ground namely of +200 V, it results that when applying -200 V from the source, the potential difference between the plasma and the substrate connected to a negative potential is in fact of 400 V and this makes that the energy of the ions arriving at the substrate to be of 400 eV. On the contrary, when applying +700 V from the source, the potential difference between the plasma and substrate is in fact only +500V.

The samples were analyzed by means of AFM and SEM for morphological characterization, EDXS (Electron Dispersive X-ray Spectroscopy) and RBS (Rutherford Backscattering Spectroscopy) for composition, NRA (Nuclear Reaction Analysis) and TDS (Thermal Desorption Spectroscopy) for deuterium retention.

Be coatings on polished silicon, substrates were also prepared as witness samples in order to study the bias voltage influence on surface morphology. After the film deposition, the thickness and composition of each film were measured by RBS. The surface morphology was observed by SEM.

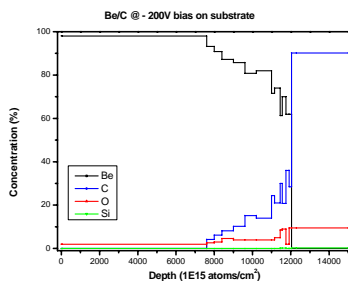


Figure 1. Depth profile composition of the Be film coated on graphite (bias: -200V)

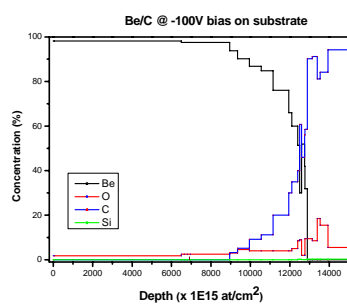


Figure 2. Depth profile composition of the Be film coated on graphite (bias: -100V)

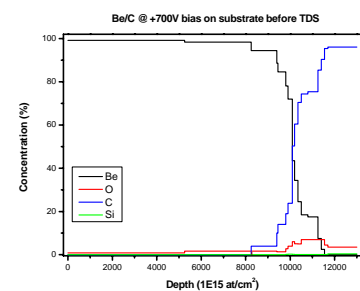


Figure 3. Depth profile composition of the Be film coated on graphite (bias: +700V)

Figures 1-3 show examples of each film composition obtained by RBS for the case of Be coated on graphite using -200 V, -100V and +700 V bias applied from an external voltage source on substrates. The thickness of coated films was basically situated around 700 nm, values in good agreement with the ones obtained from the “in situ” measurements by a QBM. The RBS measurements were performed using 2.6 MeV He⁴ ion beam.

In order to obtain the film composition from RBS experimental data, the SIMNRA code developed at IPP Garching was used. The only impurities found were oxygen from the BeO layer formed on the film’s surface due to air exposure. Figure 4 shows an example of RBS spectrum and fitting procedure.

As a first result, it was observed that the films deposited using negative bias were more adherent to the substrates than the ones grown using positive bias. This behavior was expected since by applying a negative bias on the substrate the positive beryllium ions generated in the TVA plasma were accelerated to the substrate, while applying a positive bias, the ions were decelerated and even rejected depending on the bias value.

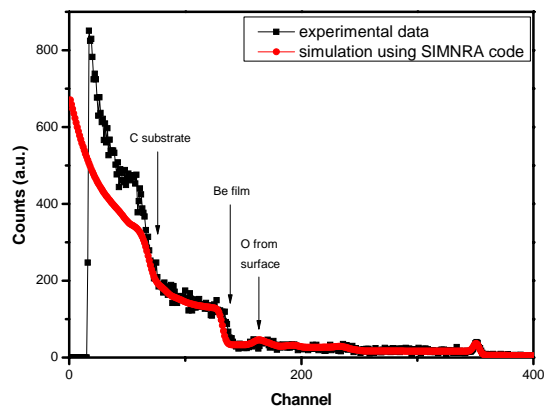


Figure 4. RBS spectrum of the Be film coated on graphite substrate.

The smoothness and the compactness of the beryllium films grown using negative bias was higher than that of the films grown using positive bias, as shown in Fig. 5 from the SEM and in Fig. 6 from the AFM images. This is the result of the bombardment of the substrate surface by energetic ions.

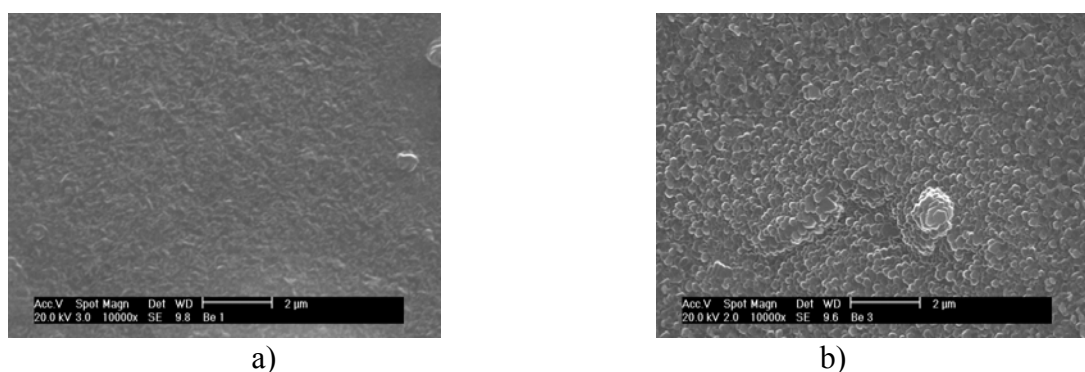


Figure 5. SEM images of Be films deposited on graphite using a negative bias (a) and a positive bias (b) applied during the deposition

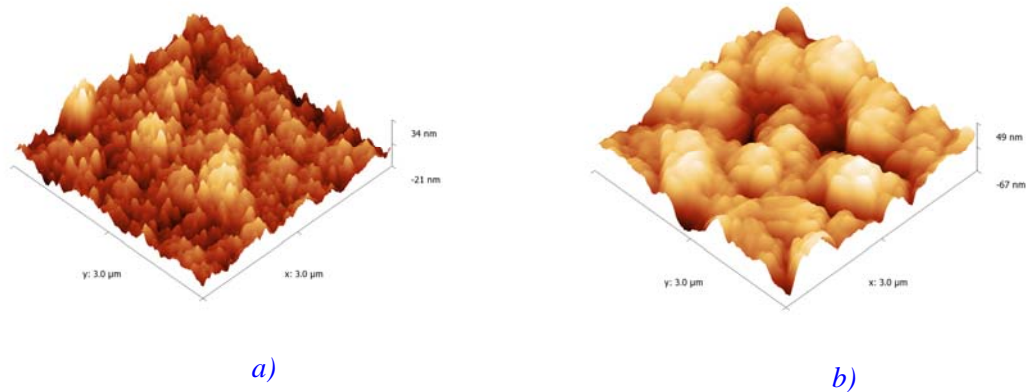


Figure 6. 3D AFM for negatively biased (a) and positively biased (b) beryllium films deposited on Si substrate

2. Deuterium implantation and NRA analysis

Deuterium implantation was performed at IPP Garching, Germany, during a collaborative action, in the High Current Ion Source. The energy of D ion beam was 600 eV D_3^+ (meaning 200 eV/D) at an incident direction normal to the target surface at room temperature. The fluence was 5×10^{22} D/m² for each sample. After the implantation, the amount of D retained in the films was determined by Nuclear Reaction Analysis (NRA) using ^3He ion beam. The concentration of D in the films was measured by means of $D(^3\text{He}, \alpha)p$ reaction with the ^3He energy of 0.69 MeV. The α particles generated by the nuclear reaction were energy analyzed with a small angle surface barrier detector at laboratory angle of 102°. The obtained α particle spectrum was converted to D depth profile using SIMNRA code.

Figure 7 presents the Deuterium distribution in the Be films prepared using: a) -200V bias on substrate, b) -100 V bias on substrate and c) +700 V bias on substrate.

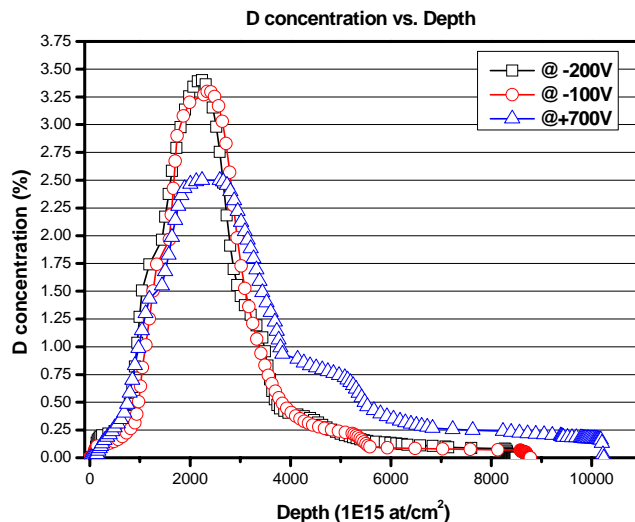


Figure 7. Deuterium distribution in the Be films prepared using: a) -200V b) -100 V and c) +700 V bias on substrates.

3. Thermal desorption analysis

Thermal desorption characteristics were obtained by placing samples in a quartz tube attached to a vacuum chamber pumped by a turbo-molecular pump. A programmable infrared furnace encloses the quartz tube to heat the sample uniformly. The temperature of the sample was monitored using a thermocouple in contact with the quartz tube. The partial pressures of the

evolving gas species were measured with a quadrupole mass spectrometer (QMS). The base pressure in the vacuum chamber was kept below 10^{-8} torr.

Once the samples have been inserted into the bake-out chamber and the base pressure has fallen below 10^{-8} torr, the infrared furnace was programmed to linearly increase the sample temperature at a rate of 15 K per minute up to 1033 K. Following the temperature ramp, the sample temperature was maintained at 1033 K for an additional 5 min and then cooled to room temperature over the following 60 min.

From RBS measurements done after the TDS was observed that the pure Be films was transformed into a mixture of Be, BeO and Be₂C after heating the sample. This behavior was expected since it is known that Be₂C phase appears at 500°C.

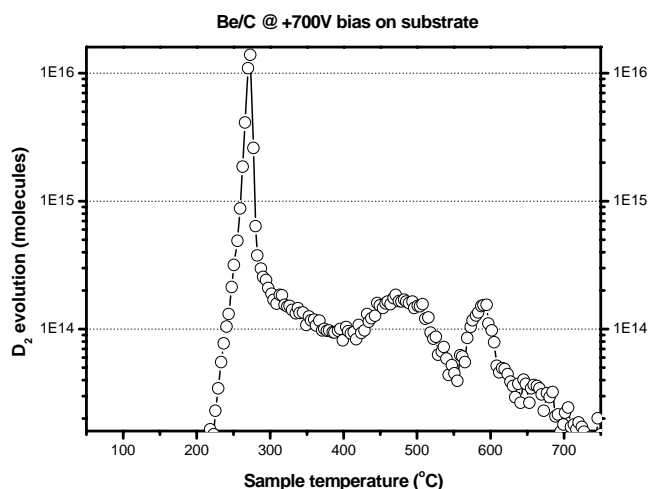


Figure 8. D₂ molecules desorbed evolution during heating the sample obtained from TDS measurements

Fig. 8 represents the TDS spectrum of the D₂ molecules desorbed from the sample. As can be observed, three deuterium molecules peaks were observed at sample temperatures of 270°C, 475°C and 585°C. The first peak, being also the highest one, corresponds to the deuterium desorbed from the area near the surface while the other two come from the deuterium trapped in deeper areas during implantation. Calculating the amount of deuterium desorbed from the sample, we found out that all the implanted deuterium was desorbed during the TDS measurements. This result was confirmed by NRA analysis after TDS by means that no α peak was observed.

4. Conclusions

Using thermionic vacuum arc method, were prepared high quality beryllium films on tungsten, graphite and CFC substrates. The prepared specimens were sent to IPP Garching for evaluation of the film quality and deuterium retention. During a collaborative action the specimens prepared using bias voltages were analyzed too.

In the case of C or Be coating, D retention property was predominantly determined by the film, but was observed almost no influence from the substrate. This was caused that D diffusion in C or Be at a room temperature could be almost negligible, i.e. most of D was retained around implantation range. Eventually, D retention in film was saturated at a certain amount. In both C and D films, the D saturation level for 200eV D implantation was $\sim 7.0 \times 10^{20}$ D/m².

The D retention in W coated samples did not reach the saturation in this experimental fluence range. On the other hand, D bulk accumulation was inhibited because D diffusion in the substrate (Be or C) is much lower than that in W at a room temperature, hence total retention decreases compared to poly-crystalline bulk tungsten.

References

- [1] K. Sugiyama, K. Krieger, C.P. Lungu, J. Roth *Hydrogen retention in ITER relevant mixed material layers*, sent to: Fusion Engineering and Design, 2008
- [2] 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*, Physica Scripta T128 (2007) 157–161.