

## HiPIMS technique used for fusion related mixed materials

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In this contribution, preliminary results are presented on HiPIMS technique used for Be-W mixed layers deposition as plasma facing material for nuclear fusion reactor applications. The main purpose is to study the retention of deuterium in various Be-W combinations with D and N seeding gases. The co-deposited films obtained by presented technique is proposed for simulation of the re-deposition process within the ITER device as relevant mixed layers onto the first wall as well as the evolution of wall surface compositions.

### 1. Introduction

It is well known that among the so called secondary processes related to plasma – wall interaction in thermonuclear fusion reactors the sputtered atoms from the plasma facing-components (PFC) materials, as beryllium (Be) and tungsten (W) as well as plasma absorbed fuel can be re-deposited on other plasma-facing surfaces or remote areas [1]. Therefore, a mixture material of beryllium originating from the first wall and tungsten from the divertor at the bottom of the machine has to be considered. The formed materials as (Be<sub>x</sub>W<sub>y</sub>) will change the tritium retention/desorption behaviour of the original PFC [2], as well as their thermo-mechanical properties. Besides W and Be, and their mixed layers will contain nuclear fuel: deuterium (D) and tritium (T), as well as seeding or seeded elements like nitrogen (N) or noble gases (Ar, He) [3-5]. Consequently, it is important to understand the deuterium retention first in single elements used for PFCs, and then try to understand the retention in co-deposited layers which will very likely be made of mixed materials. In this study, deuterium retention was investigated first in W, Be and then in the W-Be and W-Be-N mixed materials deposited by dual-HiPIMS in Ar-D and Ar-D-N<sub>2</sub> gas mixture.

### 2. Experimental device, methods and techniques

Tungsten, beryllium and tungsten-beryllium mixed materials samples were deposited by high power impulse magnetron sputtering (HiPIMS) in a high vacuum chamber pumped down to a typical base pressure lower than 10<sup>-4</sup> Pa. High purity (99.95%) circular tungsten and beryllium targets (2 mm thickness and 50 mm diameter), mounted on

two identical balanced magnetrons were used. The magnetrons were synchronously driven by two high power pulse generators, able to deliver 1 kV peak voltage and 50 A peak current values at 1 kHz repetition frequency. A signal generator was used as trigger source for high power pulse generators. Both magnetrons were operated in multi-pulse HiPIMS (m-HiPIMS) mode, a new deposition technique which has been presented in a previous work [6]. For W-Be mixed material with D seeding, a mixture of argon and deuterium, introduced through mass flow controllers (ratio of 1:1) was used as process gas, while for W-Be with D and N seeding, nitrogen gas was also added to the sputtering process gas using another mass flow controller. During deposition, the total working gas pressure was 1 Pa. The mixed materials were deposited on polished graphite and silicon substrates (unintentionally heated) mounted on a sample holder facing the target at a distance of about 10 cm. During sputtering process, the deposition rate was monitored by a quartz crystal installed in the vicinity of the substrate.

The chemical compositional, structural properties and surface morphology of the obtained layers were analyzed by Rutherford Backscattering Spectroscopy (RBS), X-ray Photoelectron Spectroscopy (XPS), X-ray diffraction (XRD) and Atomic Force Microscopy (AFM), respectively. For RBS measurements a <sup>4</sup>He energy beam of 2.5 MeV was used. The beam's energy value was chosen taking into account the interaction cross section of <sup>4</sup>He and beryllium, in order to avoid the non-Rutherford interaction that may cause errors in the data interpretation.

Deuterium retention mechanisms in the co-deposited layers, as well as the deuterium desorption

kinetics were studied using Thermal Desorption Mass Spectrometry (TDS). The experiment consisted in heating the sample with a temperature ramp rate of 10 K/min, from room temperature to 1275 K. During the entire heating process, the amount of deuterium released from the films (as D<sub>2</sub> and HD molecules) was measured.

### 3. Results and discussion

#### 3.1. W-Be sputtering by HiPIMS

Tungsten, beryllium, W-Be and W-Be-N mixed materials as plasma facing materials for nuclear fusion applications were deposited by dual-HiPIMS operating in multi-pulse mode. The HiPIMS is a powerful deposition technique, capable to generate very dense plasma with large fraction of ionized sputtered metal and an enhanced sputtering yield. An important advantage of m-HiPIMS, with respect to dc magnetron or other deposition technique, is the large number of discharge control parameters like: peak voltage during the pulse, repetition frequency of a sequence, micro-pulse duration, delay between consecutive micro-pulses, and the number of micro-pulses within the sequence [6]. These parameters can be controlled separately for two individual magnetron discharges. They also have an important role in controlling the energy transferred to the film-forming species, enabling the fine-tuning of the film properties and deposition rate of each target. In this work, sequences consisting of 5 micro-pulses, each of 3  $\mu$ s duration, separated by 50  $\mu$ s were applied on each target (Be and W) magnetron with the same repetition frequency of 700 Hz. The delay between beryllium discharge and tungsten discharge was 25  $\mu$ s.

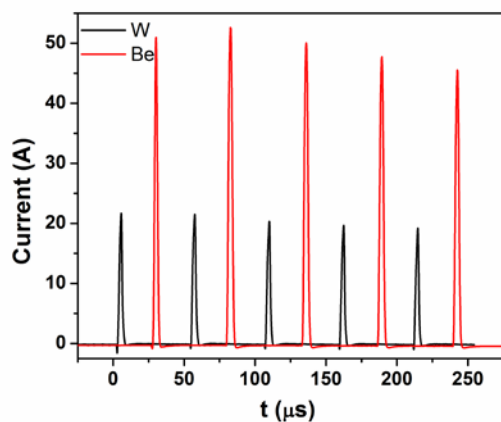


Fig. 1. Discharge current waveforms in m-HiPIMS mode. The multi-pulse is a sequence of 5 pulses of 3  $\mu$ s duration each and separated by 50  $\mu$ s.

The current waveforms for the discharges operating in multi-pulse mode in the Ar-D gas mixture at 1 Pa pressure are illustrated in Fig. 1. For tungsten discharge a peak current of about 20 A was obtained for an applied voltage of -1000 V on the target, while for beryllium discharge a peak current of about 50 A was reached for an applied voltage of only -500 V. For the pulse parameters mentioned above, the time-average power was 100 W for tungsten discharge and 50 W for beryllium discharge, while the peak power density on both targets was about the 4 kWcm<sup>-2</sup>.

#### 3.2. Structural and morphological analysis

The crystalline quality and crystal orientation of deposited Be, W and Be-W mixed films obtained by m-HiPIMS were investigated by X-ray diffraction. The Be-W films deposited in Ar-D mixture gas were found to be polycrystalline with preferential (110) orientation corresponding to tungsten cubic phase. Other identifiable peaks correspond to the intermetallic phase Be<sub>2</sub>W (103). The Be-W films, deposited in Ar-D-N<sub>2</sub> mixture gas, have a polycrystalline structure, revealing a mixed phase: two hexagonal metallic phases of tungsten with (110) and (200) crystal orientation, a hexagonal metallic phase of beryllium with (100) orientation, an intermetallic phase corresponding to Be<sub>2</sub>W (205) and an oxide phase for Be with (200) orientation.

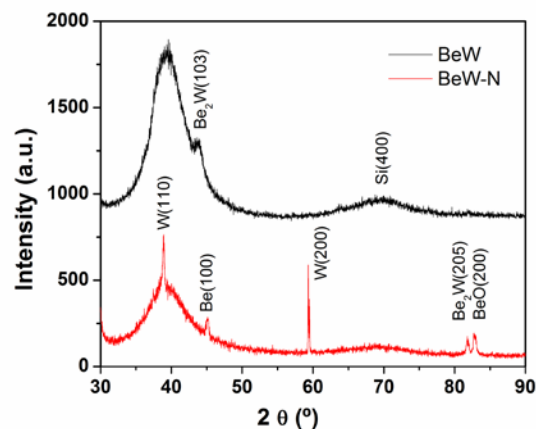


Fig. 2. XRD patterns of Be-W and nitrogen containing Be-W films.

Atomic force microscopy revealed that the small cone-shaped blisters with diameters of several micrometers and height of up to 300 nm are formed on W and Be-W film's surface. In contrast, no visible blistering occurred for the Be or BeW-N films.

### 3.3. Thermal desorption spectroscopy

The TDS spectra for the Be, W and Be-W layers with D and N seeding are presented in Fig. 3. Characteristic desorption peaks appear in TDS spectra at temperatures of about 455 K, 550 K, 625 K and 675 K. These release temperatures are in good agreement with the previously reported trapping states in Be and W, i.e. as supersaturated D for low energy trapping states, as well as deuterite, BeO [7] or WO<sub>x</sub> - related trapping respectively. Although D trapping natures are common, quantitative contribution of each trap for D retention varied with the deposition conditions and chemical composition of the deposited layers. For Be-W with N seeding, a distinctive peak appears at 550 K. This peak is present only in this sample and corresponds very well to the nitrogen desorption peak position.

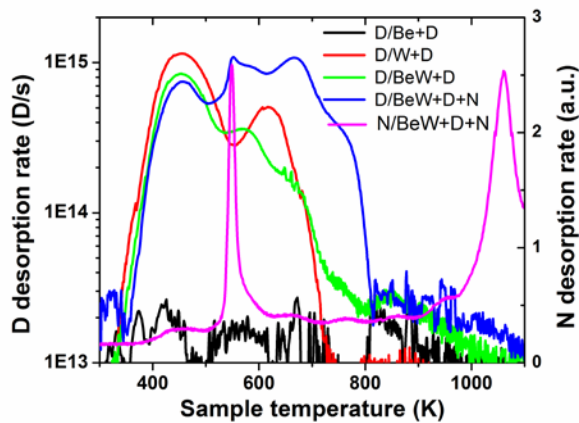


Fig. 3. Thermal desorption spectra of deuterium and nitrogen from the Be-W mixed materials obtained by HiPIMS.

The tungsten quantity in the samples also influences the D release processes. At 675 K a sharp peak appears for the W-Be with nitrogen seeding. This may be due to the Be trapping states attributed to BeD<sub>2</sub> and not to W related states because even the D quantity released from Be-D sample is low, this peak can also be observed.

We can argue that nitrogen acts as a deuterium trapping state barrier, the D release being well synchronised with N release, their bonding energies being connected. After the trapping states of N at 550 K are no longer occupied by its desorption, D immediately starts to outgas, this release temperature being attributed to Be:D with D ratio up to 1 [7]. No peaks were observed for D desorption at temperatures higher than 800 K, which implies that high energy trapping states related to defects are not occupied.

In Table 1 are given the values for the: thickness of the samples (profilometry technique), chemical composition (RBS) and released D amount (TDS). The nitrogen concentration in Be-W film deposited in Ar-D-N mixture gas is 11.4 at.%.

Table 1. Thickness, chemical composition and the D amount released from the samples measured by TDS.

| Sample  | <i>t</i><br>(nm) | Be<br>$10^{18} \text{ cm}^{-2}$ | W<br>$10^{18} \text{ cm}^{-2}$ | D<br>$10^{17} \text{ cm}^{-2}$ | D<br>(%) |
|---------|------------------|---------------------------------|--------------------------------|--------------------------------|----------|
| Be+D    | 200              | 2.4                             | 0                              | 0.11                           | 0.40     |
| W+D     | 290              | 0                               | 1.83                           | 1.82                           | 9.20     |
| BeW+D   | 485              | 0.32                            | 3.2                            | 1.39                           | 3.93     |
| BeW+D+N | 553              | 2.29                            | 2.29                           | 2.88                           | 6.00     |

### 4. Conclusions

HiPIMS is a promising and a versatile deposition technique for fusion related mixed materials, because the chemical composition and properties of the coatings can be fine tuned by changing the pulse parameters like: applied voltage, pulse length, delay between micro-pulses and sequences, repetition frequency.

For the first time in PFC materials research, Be-W containing layers have been obtained with 6 at.% D and 11.4 at.% N inclusions during the co-deposition process. TDS measurements showed that even the same bonding energies exist for the D trapped inside the samples, the quantitative contribution depends on the chemical composition of the deposited layers. Nitrogen trapped inside the layers acts as a D trapping state barrier so that the desorption of it highly depends on the release of N. The deuterium desorption efficiency decreases for Be-W layers with N seeding.

### 5. References

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