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Three batches of pure beryllium (Be), pure tungsten (W) and mixed Be:W (50:50) coatings were deposited by using the thermionic vacuum arc (TVA) technique. Different superficial roughness and morphologies were obtained by adjusting the bias voltages in the substrate holder. The coatings were irradiated afterwards with 15 keV D^+ ions up to a fluence of 5e17 ion/cm^2 with the purpose to investigate the influence of the roughness parameters on the retention of deuterium in Be-W deposits in the absence of other co-deposited impurities. Current densities lower than 0.2 W/cm^2 were used to avoid the release of deuterium caused by heating loads. The results of the experiment point to the negligible effect of rugosity on the retention behaviour under energetic ion bombardment.

1. Introduction

The erosion of plasma facing components (PFC) and the migration of impurities during the operation of the International Thermonuclear Experimental Reactor (ITER) will result in the formation of co-deposits on the surface of PFC with properties distinct from those ones of the original materials. These issues have motivated the research community to investigate the properties of co-deposited Be:W coatings, since in ITER the deposits will present a beryllium (Be) and tungsten (W) matrix. Recent investigations of tiles exposed in the Joint European Torus ITER-like wall (JET-ILW) have shown that the fuel retention in co-deposits highly depend on their roughness and morphologies [1]. Beyond beryllium and tungsten, other impurities as carbon are commonly identified as co-deposits, and this chemical effect can hinder the individual role of the superficial morphologies on the fuel retention mechanisms in mixed Be:W layers.

The tailoring of the topography of Be and W coatings will be a demand in the next future retention experiments. The goal of the present document is to report the activity in the deposition process and to investigate the influence of superficial topography on the retention behaviour of Be:W coatings in the absence of other impurities than small oxygen (O) amounts, due to the affinity of O to react with Be.

2. Experiment

Three different batches composed by pure Be, pure W and mixed Be:W (50:50) coatings were deposited on mirror quality Si plates by using the thermionic vacuum arc (TVA) method, a plasma deposition technique that avoids the use of any buffer gas [2,3]. The TVA electric diagram used in multi-elemental depositions is described in Fig. 1. The container of each elemental source is biased with a positive voltage and surrounded by a Wehnelt cylinder that focuses the electron beam produced by a circular heating cathode (a tungsten filament). A brief description of the discharge parameters useful to assure plasma ignition are presented elsewhere [2,3]. The elemental deposition rates imposed by plasma exposure on the sampler holder are controlled with quartz balance monitors (QBM). The deposition process is obviously enhanced by biasing the substrate holder with a negative voltage, which increase the energy of the incident ions, leading to more compact topographies. In opposition, a positive voltage will reject most of the negative ions and the growth of the deposited layers will be carried out preferentially by neutral atoms. Earlier experiments revealed changes in the final superficial morphology of pure Be coatings by simply adjusting the bias voltage of the sampler holder and maintaining all the other deposition parameters [2]. The calibration of the final morphologies involving Be and other elements is under development. In this experiment three bias voltages were used to deposit Be, W and mixed Be:W coatings: -700 V, 0 V and +400 V. At the end, the topography of nine different surfaces (three within each batch) were evaluated by atomic force (AFM) and scanning electron (SEM) microscopies.

The elemental depth compositions of the as-deposited and as-implanted coatings were quantified with ion beam analysis, namely by using elastic (EBS) and Rutherford backscattering spectroscopies (RBS) with incident 1600 keV H^+ and 2000 keV $^4He^+$ ion beams, respectively, for thickness and elemental quantification. Experiments involving the use of energetic ions are important in fusion research since abnormal plasma instabilities will easily accelerate hydrogen ions towards the divertor [5]. In this case, the retention behaviour of deuterium on the surfaces was investigated by implanting 15 keV $^2H^+$ ions with fluences of 1e17 ion/cm^2 and 5e17 ion/cm^2. The depth

range (R_p) and straggling (ΔR_p) of the incident ions on pure Be, pure W and Be:W (50:50) layers were evaluated with the SRIM code [4] to be about 255±47 nm, 140±66 nm and 87±44 nm, respectively, pointing to the final retention of the incident ions inside the coating's depth. During implantation, the ionic current densities were maintained lower than 0.2 W/cm^2 in order to avoid heat loads on the targets. The retained ^{2}H amounts were evaluated along 48 h after the implantation campaign by nuclear reaction analysis (NRA) using incident 1000 and 1500 keV ^{3}He$^+$ ion beams. The analytical procedure is described in some previous works [6-10].
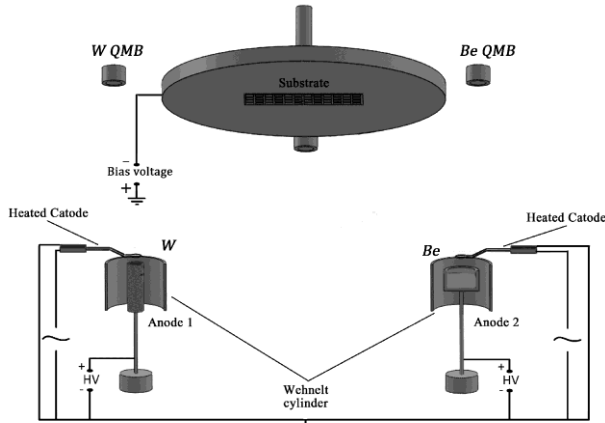

Fig. 1. Electric diagram of the TVA facility.

3. Results and discussion

As an example of the analysis performed to the topography of the coatings surface, Fig. 2 presents two-dimensional AFM maps collected from the as-deposited Be samples. The average roughness evaluated for each surface is presented in Table 1. Despite the similarity between the average values measured from the Be/-700 V (fig. 2a) and from the Be/0 V (fig. 2b) coatings, it is observed an increment of the surface sharpness by increasing the bias voltage of the sample holder during the deposition procedure. The result is consistent with the decrease of the incident energy and implanting behaviour of Be ions from -700 V to 0 V, and to the gradual increment of higher deposition rates of neutral atoms towards the substrate. The same trend is visualised from the corresponding SEM maps for pure Be and from the AFM and SEM maps of the Be:W and W coatings.

Fig. 3 presents some of the spectra collected with ion beam techniques from Be/-700 V coatings before (a) (b) and after ^{2}H$^+$ implantation with a fluence of 5e17 ion/cm^2 (c). The fit lines related to the chemical analysis are also included in the figure. Equivalent spectra and fit lines for Be:W/-700 V coatings are also presented (d) (e) (f). The three vertical arrows in the EBS spectra of Fig. 3a identify the energies corresponding to presence of Be on the superficial layer. They are related to the elastic scattering of protons by ^{9}Be, ^{9}Be(p,p$_0$)^{9}Be, which is also the main nuclear interaction, and to the emission of deuterium and α particles from the ^{9}Be(p,d$_0$)^{8}Be and ^{9}Be(p,α$_0$)^{6}Li nuclear reactions. All the spectrum is easily

resolved with a fit line considering the presence of a pure Be coating deposited on a Si plate. The existence of O as impurity was not considered since the corresponding ^{16}O(p,p$_0$)^{16}O yield will appear superimposed to the ^{9}Be(p,d$_0$)^{8}Be barrier (see fig. 3d). Differences between the measured yields and fit lines are explained by the rugosity of superficial layers. The energy loss caused by the coating material to the impinging ions is higher for ^{4}He$^+$ than for H$^+$ and the thickness of the coatings are commonly evaluated by RBS using ^{4}He$^+$ ion beams. As example of this procedure, the recoil of the Si yield (present in the substrate) caused by the energy loss imposed by the Be coating to the incident ions, and the corresponding fit line are shown in Fig. 3b. Finally, fig. 3c shows simultaneously the proton yields emitted from the ^{9}Be(^{3}He,p$_i$)^{11}B and ^{2}H(^{3}He,p$_0$)^{4}He nuclear reactions, which lead us to the final Be and ^{2}H depth contents on the coating after ^{2}H$^+$ implantation. Similar spectra were collected from the Be/0 V and Be/+400 V samples.
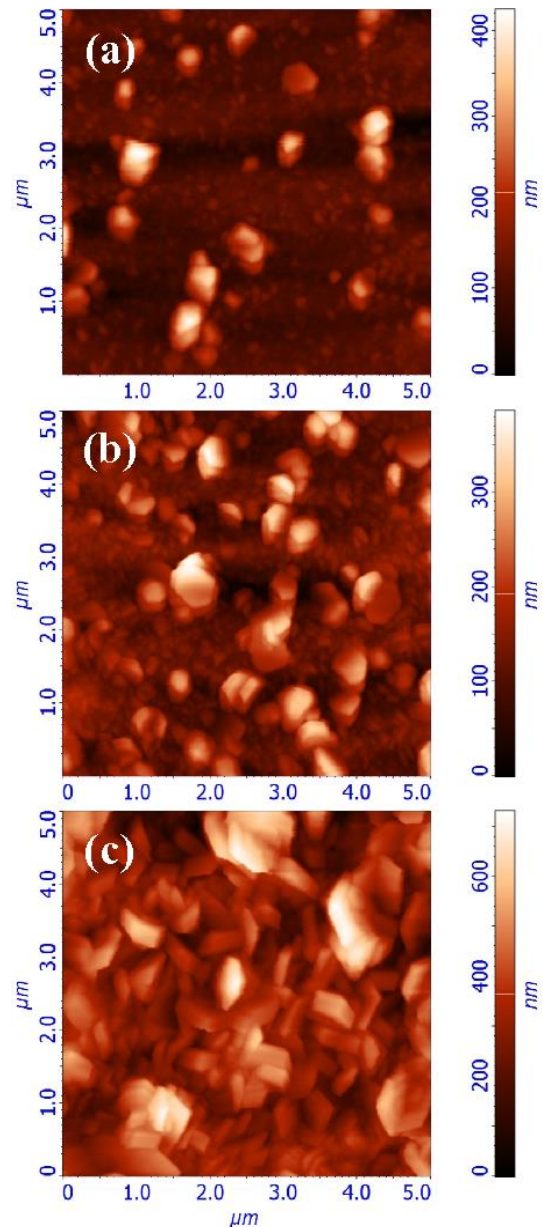

Fig. 2. Two-dimensional AFM maps and topography of the as-deposited Be/-700 V (a), Be/0 V (b) and Be/+400 V coatings (c).

Significant oxygen contents are observed from the EBS spectra of the Be:W coatings. As a consequence, the energy related to presence of O at the surficial layer is identified in Fig 3d. The O amount is reduced along the implantation depth zone for 15 keV ^{2}H$^+$ ions (the corresponding elemental contents in the implantation zones are shown in Table 1) and it is higher at deeper depths. From the RBS spectra (Fig. 3e) we quantify the thickness of the coatings, the depth profiles for W and

Be:W elemental depth ratios close to (50:50), which relates to the Be contents evaluated from the proton yields induced by the ^{9}Be(^{3}He,p_i)^{11}B in the NRA spectra (Fig. 3f). Only the presence of W and ^{2}H was identified from the analysis of the remaining W coatings. Additional EBS and RBS spectra collected from the as-implanted samples evidenced negligible erosion effects caused by the irradiation campaign.
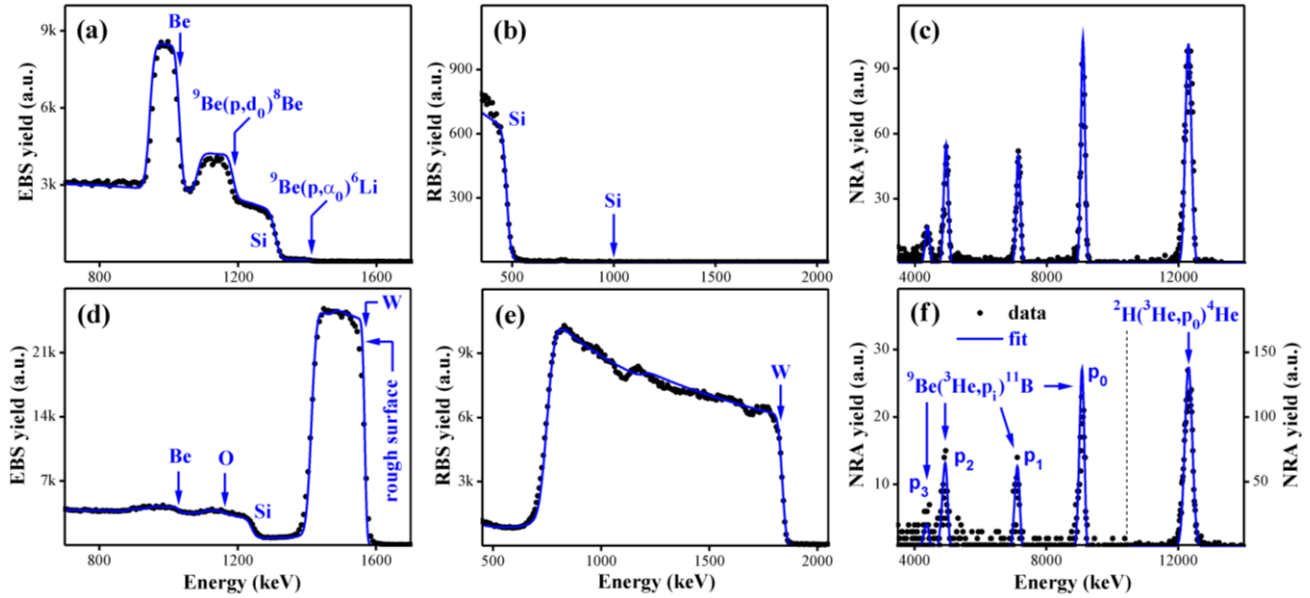

Fig. 3. EBS (a), RBS (b) and NRA (c) spectra collected from the Be / -700 V coatings and corresponding fit lines involving the chemical analysis; corresponding results for the Be:W / -700 V coatings at (d), (e) and (f).

Table 1. Averaged roughness (aver. rough.), thickness and elemental contents (elem. cont.) of the as-deposited samples; retained D in the as-implanted samples (estimated errors of 5 % for the EBS/RBS and of 15 % for the NRA results).

as-deposited coating	aver. rough. (nm)	thickness (1e17 at/cm^2)	elem. cont. Be	O	W (at.%)	D content after 1e17 D$^+$/cm^2 1.0 MeV ^{3}He$^+$ (1e17 at/cm^2)	1.5 MeV ^{3}He$^+$ (1e17 at/cm^2)	D content after 5e17 D$^+$/cm^2 1.0 MeV ^{3}He$^+$ (1e17 at/cm^2)	1.5 MeV ^{3}He$^+$ (1e17 at/cm^2)
Be/-700 V	52	170	100	~ 0	-	0.78	0.78	4.43	4.00
Be/0 V	56	175	100	~ 0	-	0.77	0.81	4.68	4.64
Be/+400 V	88	160	100	~ 0	-	0.81	0.76	4.65	4.55
Be:W/-700 V	3.1	73	45	~ 0	55	0.60	0.71	3.62	3.52
Be:W/0 V	0.3	74	40	5	55	0.76	0.81	2.61	2.83
Be:W/+400 V	3.0	69	33	7	60	0.72	0.71	3.97	3.87
W/-700 V	5.0	76	-	-	100	0.35	-	1.57	-
W/0 V	3.4	79	-	-	100	0.37	-	0.98	-
W/+400 V	9.0	79	-	-	100	0.35	-	1.23	-

Huge amounts of deuterium were found in all the as-implanted coatings along depth range two times higher than those ones predicted by the SRIM code [5], especially in the Be and Be:W samples. It is observed an evident decrease of the retained amounts by decreasing the Be content. The results are in agreement with the experiments of Chernikov et al. [11] involving the irradiation of Be by energetic ^{2}H$^+$ ions in the range from 3 to 10 keV under fluences up to 8e17 ion/cm^2. In this work, the main retention mechanism was observed at

fluences lower than 1e17 ^{2}H$^+$/cm^2. It is explained by the formation of small cavities in the nm scale with high volume density acting as recombination sites for molecular (^{2}H$_2$) in the implantation zone. The medium size of the cavities increase and an open porosity (microchannels) is generated in the near surface region by increasing the fluence [5,11]. Changes in the incident ion energies lead to the same retention behaviour [11]. Commonly, the retention of ^{2}H is much lower in W. Nevertheless, W exhibits very high recombination rates

and hydrogen diffusivity may even be enhanced in the implantation zone under high irradiation regimes [12]. The behaviour explains the blistering and collapse occurred in the W/0 V and W/+400 V coatings loaded with 5e17 ^{2}H$^+$/cm^2 after the irradiation campaign. In opposition, the W/-700 V coating irradiated at the same fluence preserved the initial mirror like look. As a consequence, the last three samples were not considered for the final analysis. Furthermore, and as it is expected from pure W, the morphological changes induced under irradiation point to a huge retention nearby the superficial layers. It is not necessary to perform exhaustive depth quantification for ^{2}H and the NRA analysis was carried out only with a unique energy for the incident ^{3}He$^+$ ion beams.

Table 1 resumes the main results of the chemical analysis. Apart the deviations observed in the retained amounts in the quite flat Be:W surface after implantation of 5e17 ion/cm^2, they do not evidence a trend in the retained contents by changing the morphology of the superficial layers, enhancing the influence of other co-deposits in PFC as the main retention sources under operative plasma scenarios.

4. Summary

Three batches of thick and pure Be, pure W and mixed Be:W (50:50) coatings were prepared by TVA. Within each batch the coatings presented similar compositions and distinct morphologies at the surface, being the changes in the topography obtained by adjusting the bias voltage of the substrate holder during the deposition procedure. The calibration of the deposition parameters of the method is under development.

Apparently and within the morphological changes observed in the present as-deposition samples, the retention amounts follow the Be contents and it is not observed a trend in the retained deuterium content by changing the topography of the surfaces after irradiating the coatings with energetic 15 keV ^{2}H$^+$ ions up to fluences of 5e17 ion/cm^2 and under low power density regimes. The results seam to enhance the main influence of the composition of the co-deposits on the final retention rates. The irradiation experiments should be complemented with other distinct coatings morphologies and at lower incident energies.

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