Surface morphology influence on Deuterium retention in beryllium films prepared by thermionic vacuum arc method

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Abstract

In a plasma-confinement device, material eroded from plasma facing components will be transported and re-deposited at other locations inside the reaction chamber. Since beryllium from the first wall of the ITER fusion reactor will be eroded, ionized in the scrape-off layer plasma and finally re-deposited on divertor surfaces flowing along the magnetic field, it is important to study the properties of divertor armour materials (C, W) coated with beryllium.

By applying different bias voltages (-200 V to +700 V) to the substrates during deposition, the morphology of the obtained films was modified. The films' morphology

was characterized by means of AFM and SEM, and it was found that the coatings prepared using negative bias voltage at the substrate during deposition are more compact and have a smoother surface compared to the samples prepared with positive bias voltage. The thickness and composition of each film were measured using Rutherford Backscattering Spectrometry (RBS). A study of deuterium implantation and retention into the prepared films was performed at IPP Garching in the High Current Ion Source.

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1. Introduction

In a plasma-confinement device, material from plasma facing components will be eroded, transported and redeposited at other locations inside the reaction chamber. The first wall of the ITER machine is planned to be from beryllium, while the divertor region will consist of tungsten and carbon [1-2]. Beryllium (Be) from the first wall will be eroded and then ionized within the scrape-off layer plasma and finally flowing along the magnetic field will be redeposited on the divertor region. Consequently, studying the properties of divertor materials (C, W) coated with beryllium is an important issue.

In this work Be films were prepared using thermionic vacuum arc technology developed at NILPRP Bucharest [3]. Deposition was made onto biased graphite substrates. Both composition and morphology of the beryllium films versus substrate bias (in the range from -200 V to +700 V) were investigated. The film analysis was characterized by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) techniques. It was found that the coatings done using negative bias applied on the substrate during the deposition are more compact (meaning free of holes) and smooth having an average roughness of 7 nm.

Also, the thickness and composition of each film were confirmed by Rutherford Backscattering Spectrometry (RBS) and deuterium implantation was performed at IPP Garching in the High Current Ion Source. After the implantation, the amount of D retained in the films was determined by Nuclear Reaction Analysis (NRA) using ^{3}He ion beam and the obtained α particle spectrum was converted to D depth profile using SIMNRA code [4-6].

2. Experimental set-up

Beryllium film deposition was performed using the thermionic vacuum arc (TVA) technique. TVA technique is characterized by producing plasma in the pure vapors of the metal to be deposited without using any buffer gas [1]. The evaporation of the metal takes place in high vacuum conditions (about 10^{-5} Torr). An externally heated cathode consisting in a tungsten filament produces thermally emitted electrons at a current of about 100 mA. These electrons are focused by a Wehnelt cylinder on the anode which consists of the material to be deposited and is biased to high positive voltage (1 – 6 kV). The electron bombardment creates metal vapors above the anode at a local pressure of about 133 Pa. The TVA principle scheme is presented in Figure 1 where by I_f is designated an AC power supply providing the current for heating the cathode filament (0-120 A, at 0-24 V) and by HV is nominated the power supply able to provide high voltage (0-6 kV, at 0-5 A).

Using this technique deposition rates in the range of 4-20 nm/s can be obtained with the advantage that the high energy metal ions bombardment during deposition ensures high density layers, compared with those of the bulk material, good structure and high adhesion to the substrate. To study the influence of the energetic ions on both the structure and properties of the deposited films the substrate was biased during the coating process at voltages in the range from – 200 V to + 700 V, respectively. The positive bias of the substrate is to reject the ions and select only the neutral atoms for deposition. On contrary, in the case of negative bias of substrate the ions are accelerated impinging the deposited layer with relatively high kinetic energy.

Being a comparative study, all the depositions were characterized by the same discharge parameters (I_f = 45 A, I_{dis} = 1,5 A, U_{dis} = 800 V), the only difference being the bias applied on the substrate during the coating.

Also, the thickness of the films was controlled using an in situ Quartz Balance Monitor (QBM).

3. Results and discussion

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

After film deposition, the thickness and composition of each film were confirmed by Rutherford Backscattering Spectrometry (RBS). The chemical structure was analyzed by X-ray diffraction (XRD) and the surface morphology was observed by Scanning Electron Microscopy (SEM).

The thickness of coated films was basically around 700 nm, values in good agreement with the ones obtained from the QBM results. RBS measurements were performed using 2.6 MeV ^{4}He ion beam. In order to obtain the film composition from RBS experimental data, the SIMNRA code developed at IPP Garching was used. As one can see the only major impurities found was oxygen from the BeO layer formed on the film's surface due to air exposure. Figure 2 shows an example of RBS spectra while

Figure 3 shows examples of each film composition obtained by RBS for the case of graphite substrate.

From the SEM images analysis one can see that the morphology of the films grown using positive bias applied on substrate are similar with the films grown by thermal evaporation. For exemplification, in figure 4, SEM images of beryllium films grown using negative bias (a) and positive bias (b) applied on the substrate during the deposition process are presented. As one can see, by applying a negative bias on the substrate during the deposition the films present a lamellar structure. Also, the SEM images revealed that the films grown using positive bias showed some cracks on the surface meaning that they are more brittle.

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. The average R_{ms} roughness of the films grown under negative bias was 7nm in contrast with the large R_{ms} roughness (19 nm) of the films prepared under positive bias.

In order to study the influence of the bias applied on the films' properties from the point of vie of fuel retention, deuterium implantation was performed at IPP Garching 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 x 10^{22} D/m^2 for each sample. After the implantation, the amount of D retained in the films was determined by Nuclear Reaction Analysis (NRA) using ^{3}He ion beam. The concentration of D in the films was measured by means of D (^{3}He, α)p reaction with the ^{3}He 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^{o}. The obtained α particle spectrum was converted to D depth profile using SIMNRA code.

Figure 5 presents the Deuterium distribution in the Be films prepared using: a) -200V bias on substrate and b) +700 V bias on substrate.

The amount of deuterium retained in the films, estimated from the NRA measurements, was of about 7 x 10^{22} D/m^2. As one can see from the deuterium depth profiles presented in figure 5 the implanted deuterium didn't reach the Be/C interface zone. Analyzing the deuterium depth profiles, presented in figure 6, one can observe that they depend on the bias applied on the substrate. For the negatively biased samples, higher amount of deuterium is retained in the area near the surface while for the positively biased ones higher amount of deuterium is present in deeper area. This behavior can be related to the structure of the films.

4. Conclusions

As a result, 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 the scratching test. Also, the SEM and AFM measurements revealed that applying a negative bias on the substrate during the deposition were obtained adherent, compact and smoother beryllium films. From the fuel retention point of view, it seems that there is a correspondence between the bias conditions and deuterium depth profile but further investigations need to be done for a confirmation of this supposition.

References:

[1] ITER Physics Expert Group, ITER physics basis, Nucl. Fusion 39 (1999) 2137.

[2] C. Grisolia, G. Counsell, G. Dinescu, A. Semerok, N. Bekris, P. Coad, C. Hopf, J. Roth, M. Rubel, A. Widdowson, E. Tsitrone and JET EFDA Contributors, Fusion Engineering and Design, 82 (2007) 2390.

[3] 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, Phys. Scr. T128 (2007) 157.

[4] J. Roth, V. Kh. Alimov, A. V. Golubeva, R. P. Doerner, J. Hanna, E. Tsitrone, Ch. Brosset, V. Rohde, A. Herrmann, M. Mayer, Journal of Nuclear Materials, 363-365 (2007) 822.

[5] R. Pugno, K. Schmid, M.J. Baldwin, R. Doerner, J. Hanna, D. Nishijima, V. Rohde, J. Roth Journal of Nuclear Materials, 375 (2008) 168.

[6] M. Mayer, SIMNRA User's Guide, Tech. Report IPP 9/113, Max-Plank-Institut fur Plasmaphysik.

Figure captions

Figure 1. TVA principle scheme.

Figure 2. RBS spectrum for Be film on graphite substrate.

Figure 3. Composition depth profiles of Be films prepared using -100V and +700V bias voltages, obtained by RBS.

Figure 4. SEM images of Be films grown using negative (a) and positive (b) bias.

Figure 5. D depth profile into the Be films grown using negative (-100 V) and positive (+700 V) bias obtained from the α spectrum using SIMNRA code.

Figure 6. D depth profiles for Be films grown on graphite by applying -200V, -100V and +700V biases on substrates.

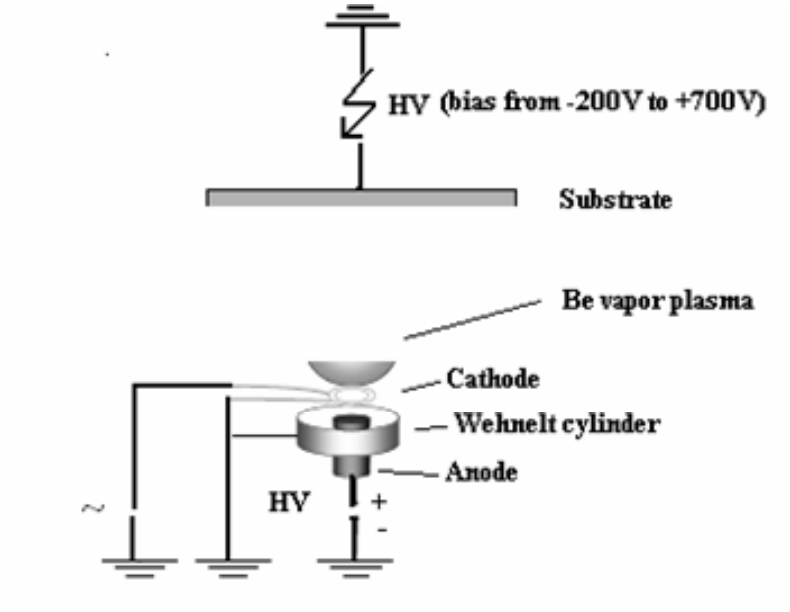
HV (bias from -200V to +700V)
Substrate
Be vapor plasma
Cathode
Wehnelt cylinder
Anode
HV
+
-
~

Figure 1

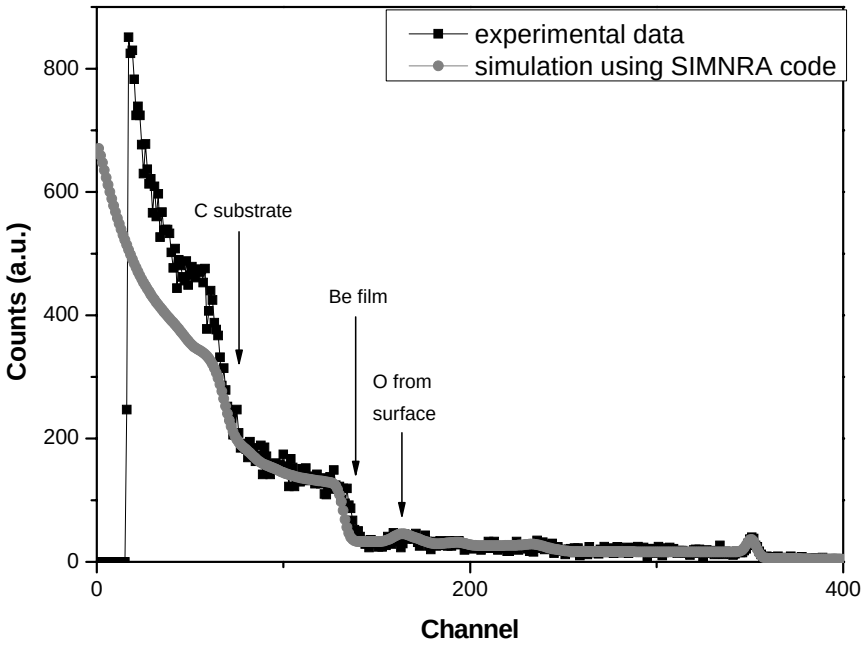
experimental data
simulation using SIMNRA code
C substrate
Be film
O from
surface
Counts (a.u.)
Channel
0
200
400
600
800
0
200
400

Figure 2

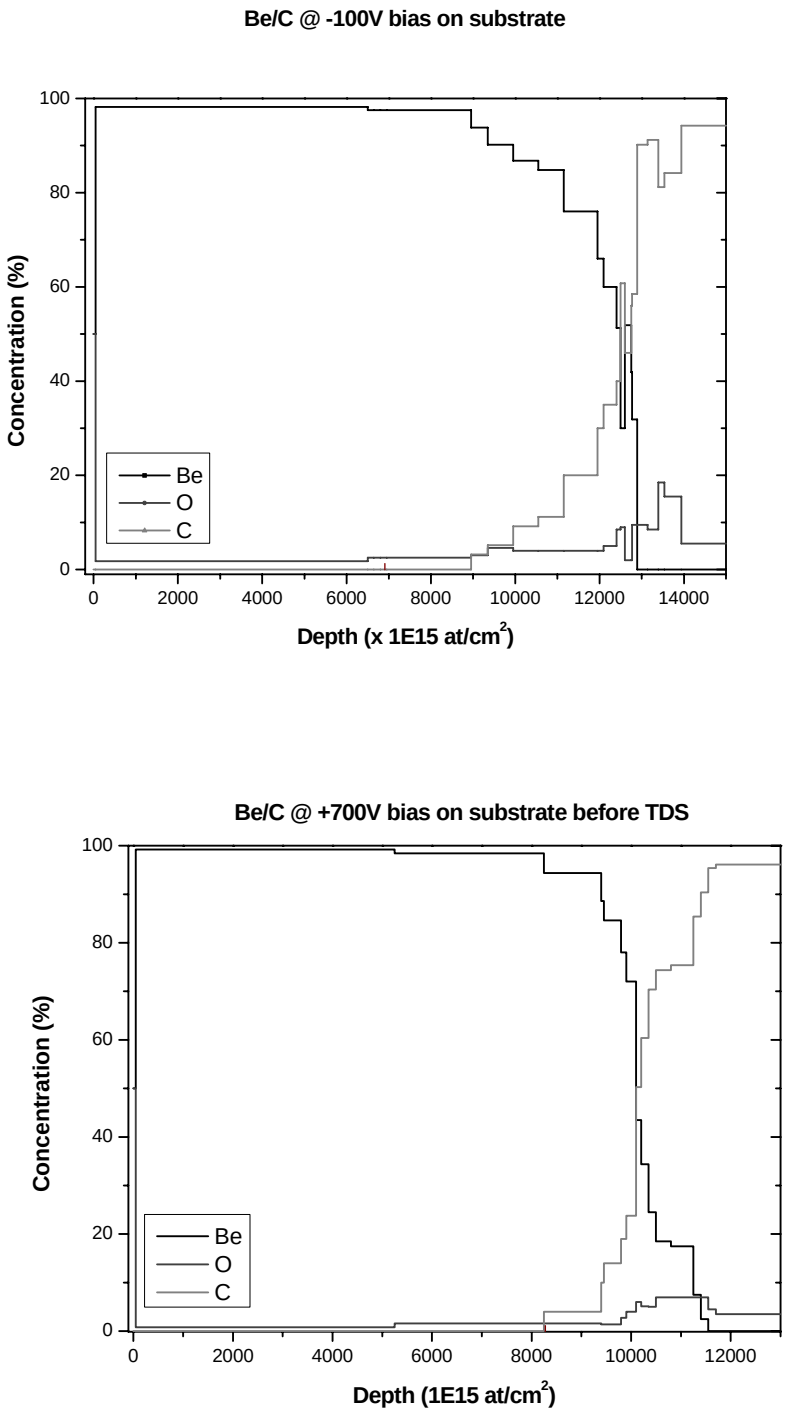
Be/C @ -100V bias on substrate
Concentration (%)
Depth (x 1E15 at/cm^2)
Be
O
C
Be/C @ +700V bias on substrate before TDS
Concentration (%)
Depth (1E15 at/cm^2)
Be
O
C

Figure 3

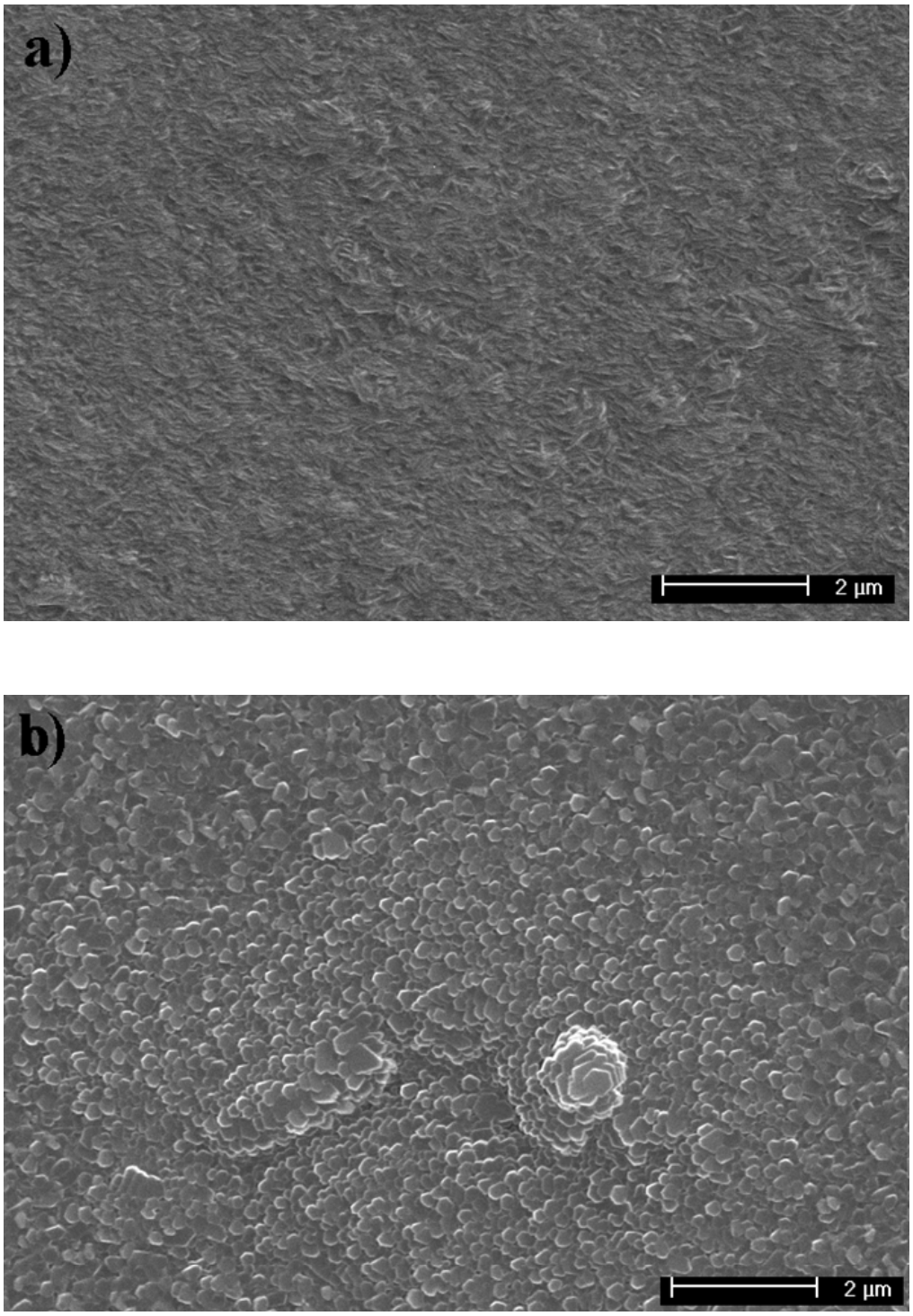

Figure 4

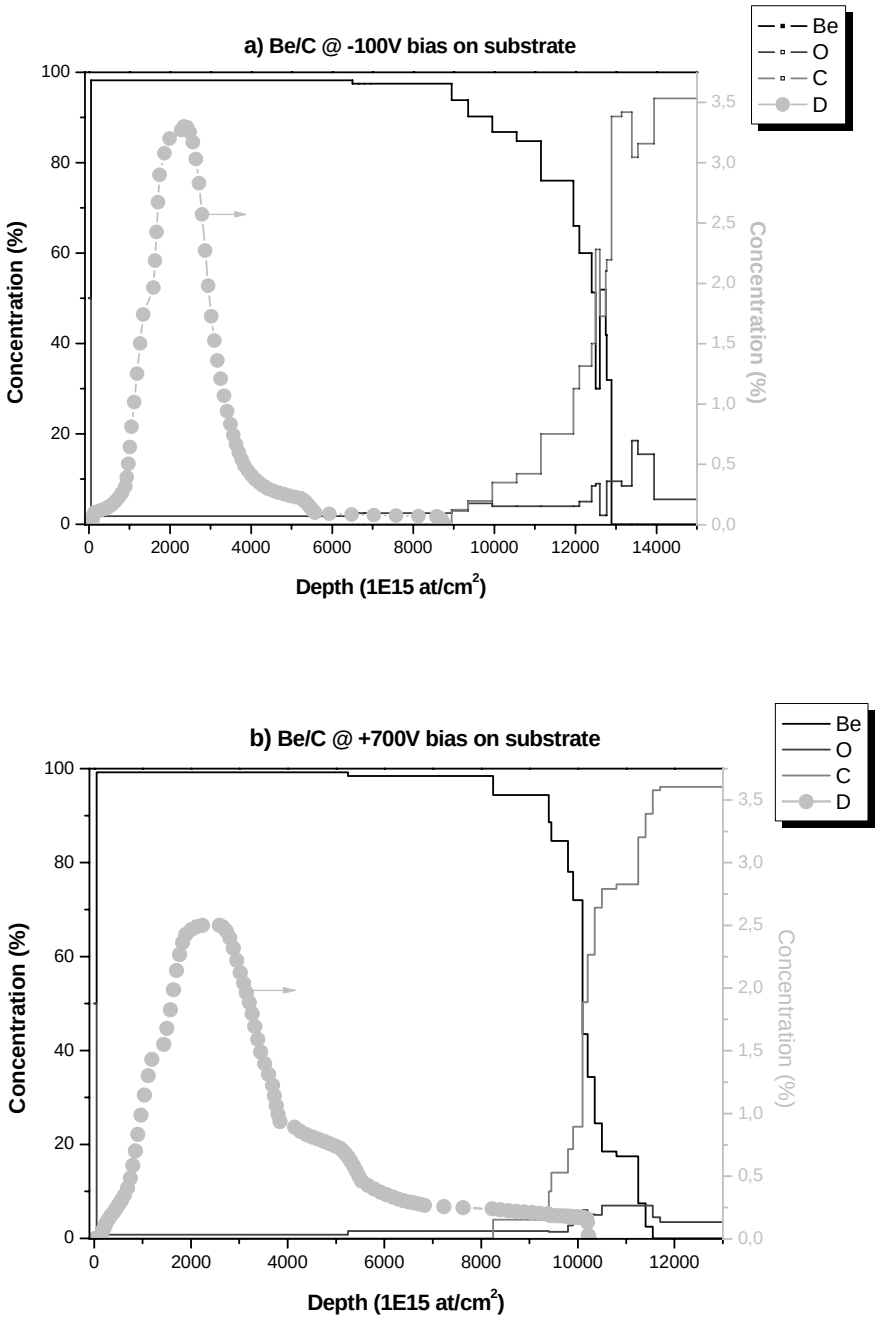
a) Be/C @ -100V bias on substrate
Be
O
C
D
Concentration (%)
Concentration (%)
Depth (1E15 at/cm²)
b) Be/C @ +700V bias on substrate
Be
O
C
D
Concentration (%)
Concentration (%)
Depth (1E15 at/cm²)

Figure 5

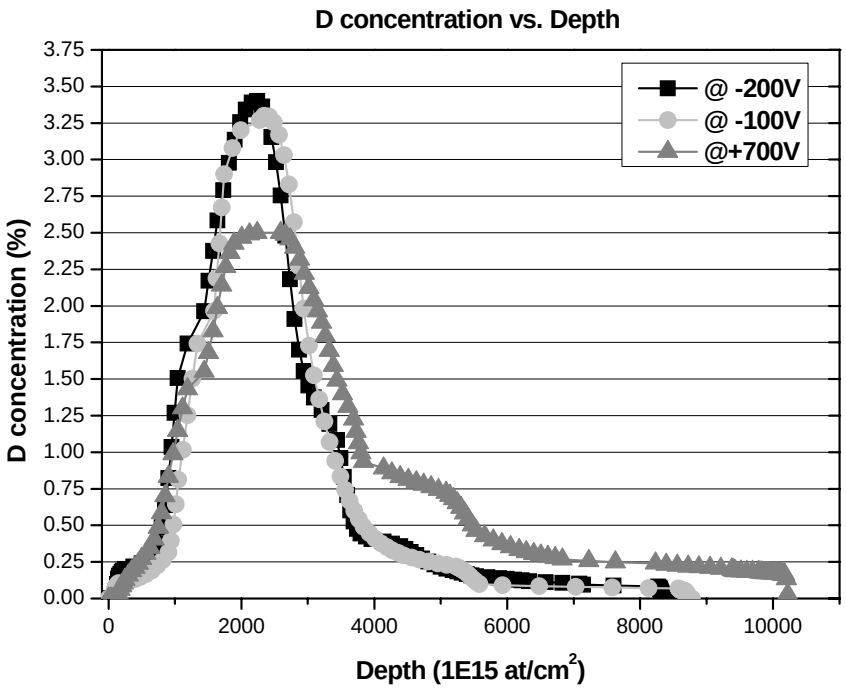
D concentration vs. Depth
D concentration (%)
Depth (1E15 at/cm2)
@ -200V
@ -100V
@+700V
0.00
0.25
0.50
0.75
1.00
1.25
1.50
1.75
2.00
2.25
2.50
2.75
3.00
3.25
3.50
3.75
0
2000
4000
6000
8000
10000

Figure 6