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Abstract

Retention of D was investigated in Be-based co-deposited layers. The D inventory increased with the thickness or decreasing amount of O in the deposits, and the highest concentrations (up to 40-50 at.%) were measured for mixed Be-C-O-D layers with O concentrations below 5 at.%. Increasing roughness enhanced D accumulation but only by a factor of 1.5 when roughness changed by two orders of magnitude. Of the studied samples, Be-C-O-D layers were the closest proxies to the samples extracted from JET-ILW tiles in terms of their retention and fuel-release characteristics.

Keywords: Beryllium, Deuterium, Fuel retention, Surface roughness, Co-deposition, Implantation

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1. Introduction

Beryllium (Be) will be used as a plasma-facing material in the main chamber of ITER [1]. To lay the basis for efficient operation of ITER in the late 2020s, JET has been equipped with an ITER-Like Wall (ILW), consisting of a Be main chamber and a tungsten (W) divertor [2, 3]. Two important aims of the ILW project are estimating material migration and fuel retention in an ITER-relevant environment, and during the last couple of years the following conclusions have been made [4-10]:

- Compared to operations in the carbon phase, fuel retention in JET-ILW is reduced by a factor of 10-20 and is dominated by co-deposition [4, 5].
- Co-deposited layers, up to 40 μm in thickness, have been predominantly observed on high-field side (inner) divertor plasma-facing components (PFCs) [6-9].
- These co-deposits are rich in Be with fuel (here, deuterium (D)) fractions of 5-10 at.%. The most noticeable impurities are carbon (C) from the PFCs and oxygen (O) ending up in the vessel between operational days. The relative abundances of these impurities are comparable to that of D [6-9].
- Retention is persistent in the deposits: even after baking at 350°C for several hours, hardly more than 10% of the retained D can be released [10].

To understand the physics behind the observations and to study how retention depends on the properties of the deposited layers, a large set of laboratory samples, mimicking the observed co-deposits on JET-ILW PFCs as much as possible, is needed. To this end, an extensive development project has been launched under the Work Package PFC of the EUROfusion Consortium, consisting of production and characterization of different Be-based laboratory samples.

Here, we will report the status of the project and review its main physics outcomes. The focus is put on Be-C-O-D and Be-O-D co-deposits that resemble those found on the corner region (so-called Tile 1) between the inner divertor and the main chamber of JET-ILW. For comparison, pure Be-D layers as well as different mixtures of Be-W-D are investigated; together with Be-O-D, such layers may be encountered on selected PFC regions in ITER.

2. Production and analyses of the co-deposited layers

All the co-deposits were produced in the facilities of the National Institute for Laser, Plasma and Radiation Physics (NILPRP) in Romania using their High Power Impulse Magnetron Sputtering (HiPIMS) and Direct Current (DC) magnetron sputtering devices [11] as well as Thermo-Vacuum Arc (TVA) [12] deposition systems. Three different types of samples were produced: Be-D, Be-O-D, and Be-C-O-D, with the target values for the D content being in the range of 1-10 at.% and the O and C concentrations within 5-10 at.%. In addition, first batches of Be samples doped with nitrogen (N) - simplest examples of deposits that could be formed as a result of seeding experiments in ITER - were produced with the nominal composition of Be(90%)-N(5%)-D(5%).

As substrates, bulk W was primarily used but silicon (Si) was also used, especially for the thinnest coatings. The nominal thicknesses of the samples ranged from 0.4 to 15 μm and all the depositions were made at room temperature. The nominal compositions and thicknesses of the co-deposits are collected in Table 1. A set of 0.4-μm thick Be, Be-O, and Be-C-O coatings without D inclusions were also prepared for implantation experiments with D^+ ions at Instituto Superior Técnico in Lisbon. Here, the applied ion energy was 5 keV and the fluence was fixed to 2.0×10^{17} D^+ cm^{-2}.

Finally, a number of Be-W coatings, both with (magnetron sputtering) and without (TVA) deuterium was produced at different levels of sample bias voltage to alter their average roughness. The Be:W ratios of the films were 1:0, 2:1, 1:1, 1:2, and 0:1. Again the D-free samples were implanted using otherwise similar parameters for the D^+ ions as listed above but the fluence was varied from 1.0 to 5.0×10^{17} D^+ cm^{-2}.

After the production, the samples were analyzed in different contributing laboratories for their true composition, the relative and absolute D content of each co-deposited layer, as well as roughness and modifications of their surfaces. For these purposes, Rutherford Backscattering Spectrometry (RBS), Nuclear Reaction Analysis (NRA), Time-of-flight Elastic Recoil Detection Analysis (TOF-ERDA), Secondary Ion Mass Spectrometry (SIMS), Thermal Desorption Spectroscopy (TDS), and Raman spectroscopy were used. Complementary information was provided by Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) analyses.

For RBS, either 1.0-1.8 MeV protons or 2.0-MeV $^4He^+$ ions at 165° were used to determine the Be, C, and O contents of the samples. The D levels were extracted by NRA, using protons formed in the reaction $D(^3He,p)^4He$ and an energy of 1.5 or 2.2 MeV for the incoming ^3He beam. The analysis depth was generally < 5 μm. The TOF-ERDA measurements provided the absolute concentrations of all elements on the samples but only from their topmost 0.5-2 μm thick surface layer. The analyses were carried out either using a 20-MeV $^{127}I^{6+}$ beam at 20° (analysis depth ~0.5 μm) or a 50-MeV $^{35}Cl^{9+}$ beam at 40° (analysis depth 1-2 μm). The depth profiles of selected elements (here H, D, Be, C, O, and N) were provided by SIMS. These measurements were performed using a 5-keV O_2^+ primary ion beam at a typical sputtering rate of 0.5-0.7 nm/s of the coating material. The TDS measurements were performed by heating the sample up to 700°C to avoid peeling of the deposited layer. Recorded signals corresponded to ion masses 2 (H_2), 3 (HD), and 4 (D_2).

3. Results

3.1 Effect of sample thickness and impurity content on retention

As the thickness of the produced samples exceeds 1 μm, their D levels increase by a factor of 5-10 compared to thinner variants of the same sample type. This is illustrated in Figure 1 where TOF-ERDA depth profiles of H, D, Be, C, O, and N for a ~0.4 μm (part a) and ~5.5 μm thick sample (part b, data from the 0.5 μm thick surface layer) are shown. Both samples have the same target composition of Be(79%)-O(8%)-C(8%)-D(5%), i.e., the deposition parameters were identical during the production phase.

The drastic difference in the amount of D retained in the “thin” and “thick” coatings could be connected with the deposition method: only when the growing film exceeds a certain thickness, the structure becomes stable and the stoichiometry is preserved. The hypothesis is supported by SIMS depth profiles (see examples in Figure 2) which show alterations from the otherwise uniform behaviour only within the last micrometer before the substrate is reached. Chemistry seems to play a large role here since the oscillations visible in the D, Be, and O profiles in Figure 1a are much more moderate or even non-existent for samples without C additions.

No big differences are observed in the D concentrations or depth profiles of samples for thicknesses >1 μm. Yet, many of the “thick” coatings flake off when subjected to the analysis beams of TOF-ERDA or SIMS, either resulting from relief of stresses that have formed during the deposition or from cracking of the deposited layer upon exposure to the mechanical shock of the impinging ions. However, this observation cannot be purely attributed to the increasing layer thickness since coatings with large C:Be and D:Be ratios and thicknesses >5 μm are measured to be tightly adhered on their substrates.

The second important observation is that the relative D concentrations of the "thick" samples are typically in the range of 5-25 at.% but can climb to as high values as 40-50 at.%. Since the target level for all the samples was 5-10 at.%, it appears hard to reach exactly the desired composition by simply adjusting the process parameters during deposition. For instance, considering three samples with the same target composition of Be(79%)-C(8%)-O(8%)-D(5%) and comparable values for the film thickness (5-6 μm), the measured D levels vary from 18 to 38 at.%. However, when the structure of the compound becomes "simpler", the D levels can be controlled more accurately. As an example, the actual D concentrations of samples with target compositions of Be(99%)-D(1%), Be(95%)-D(5%), and Be(90%)-D(10%) are 1-7 at.%, 3-18 at.%, and 10-20 at.%, respectively.

The TOF-ERDA profiles in Figure 1 also show another interesting feature: the smaller the O content, the higher is the D retention in the coatings. This is most clearly visible in Figure 1a: once the O level shoots up, the D signal drops down and vice versa. Again in thicker samples the O levels seem to be much better under control: typically they remain within 0.5-4 at.%. In the thinner counterparts, the oxygen concentrations cover a huge range from 3 to 50 at.%, at least partly influenced by residual O in the deposition chamber. This we notice from the analysis of samples supposed to contain only Be and D but showing measurable amounts of O in their film matrix (0.5-1 at.%).

Adjusting the O levels of the coatings is thus difficult but it can be largely controlled by the fraction of C in the crystal lattice. One can conclude that the higher the C content, the smaller the O concentration and thereby the stronger the retention. In thin samples, inclusion of C by some 5-10 at.% results in the reduction of the O content by almost 50% and the fuel retention by a factor of 4-8 while in the thicker samples the changes in the O levels are not that dramatic (atomic fractions are already low): retention changes from around 20 at.% to >40 at.%. This is in line with the observations made in Ref. [10] where D retention was observed to be strongly dependent on the layer composition. However, in those studies the as-deposited samples were loaded with D at varying surface temperatures unlike the deposits discussed here.

Adding N in the lattice instead of O does not alter the retention characteristics but comparable D concentrations (20-25 at.%) are measured on Be-D and Be-N-D samples with otherwise identical properties. The N levels were close to the desired 5 at.% (measured value ~4 at.%) while the background nitrogen concentrations in non-doped samples were generally between 0.5 and 1 at.%. Interestingly, earlier results in Ref. [14] of N-containing Be samples indicate nitrogen to increase retention by a factor of 1.5-2 - but also for these samples the D exposure conditions have been different from what we have applied: in [14] the samples were exposed to a D atomic beam.

3.2 Retention and release characteristics of the produced samples

As mentioned in the previous section, the highest retention was observed for Be-C-O-D samples with as low O concentrations as possible. The data set is limited but results from a few samples with different C contents (4, 8, and 11 at.%) indicate that the closer the carbon concentrations are to 10 at.%, the larger will be the fraction of accumulated D: in the three examples mentioned above, the relative D fractions are 18, 23, and 38 at.%, respectively. All these films had roughly identical thicknesses and the same deposition parameters (target composition Be(79%)-O(8%)-C(8%)-D(5%)).

The differences in the retention characteristics of Be-C-O-D, Be-O-D, and Be-D samples can be seen in Figure 3a. Here, NRA data for the following samples is shown: Be(85%)-C(5%)-

O(5%)-D(5%), Be(85%)-O(10%)-D(5%), and Be(95%)-D(5%). The real thicknesses of the samples were 7, 10, and 6 μm, respectively. The carbon-containing sample is the richest in D (measured D concentration ~30 at.%) while no clear differences can be seen between the latter two deposits with and without oxygen inclusions (for both, the retention is ~20-25 at.%). The extracted O levels were ~1 at.% for Be(85%)-C(5%)-O(5%)-D(5%), 1.5 at.% for Be(85%)-O(10%)-D(5%), and ~0.5 at.% for Be(95%)-D(5%). The O level does not explain the differences in retention but especially at small concentrations (<5 at.%), more important will be the amount of C in the film structure.

The release properties of D from the samples discussed above is also dramatically different. This can be seen in Figure 3b, where the normalized D desorption flux of TDS measurements is shown. Be-D has a narrow peak at around 350°C, the Be-O-D samples show broader peaks at much higher temperatures (>400°C) - even though the differences in the real compositions of Be-D and Be-O-D are not that large - and in Be-C-O-D the release peak is structured and covers a large temperature range from 300 to 600°C. In addition to the samples from Figure 3a, also data from a Be(90%)-O(5%)-D(5%) layer (thickness ~2 μm, D concentration ~7 at.%, O concentration ~2 at.%) is reproduced here.

The differences can be, at least partly, explained by the contribution of strong C-D bonds that have been measured by Raman scattering in Be-C-O-D samples. An example from a ~7-μm thick Be(76%)-C(7%)-O(7%)-D(10%) coating can be seen in Figure 4a. The same figure shows also a group of weaker peaks that can be associated with O-D bonds and contribute to D retention both in Be-C-O-D and Be-O-D (and in Be-D whose residual O content is always >0.5 at.%). According to Figure 4b, no signs of Be-D bonds are visible: the Raman curves extracted from various beryllium-based coatings are drastically different from what would be expected for a stoichiometric BeD_2 compound [15]. The pedestal-like feature in the range 400-700 cm^{-1} and around 1000 cm^{-1} of the measured Raman shift, are due to various defects in the Be matrix [16] and they seem to be inherent for the deposition process, becomig more prominent as the D content increases. This leads one to conclude that, besides bonding with O and C, fuel is retained in various defects and traps of the crystal. An additional contribution may come from the formation of crystalline BeO where D is favourably bound via O-D bonding as evidenced by data from melt-affected Be tiles of JET-ILW [17].

3.3 Effect of surface roughness and morphology on retention

The produced Be-C-O-D, Be-O-D, and Be-D samples have generally a smooth surface, almost independent of their D content, and the irregularities on the surface reflect those of the substrate. This we can see in Figures 5a and b where SEM images of two Be-O samples with D contents of 12 at.% and 26 at.% are shown (O level ~2.5 at.%). No hills or valleys are present and compared to tokamak PFCs, the measured surface roughness of R_a<100 nm is orders of magnitudes smaller.

Detailed investigations of a set of Be-W-D samples with different Be:W ratios have, however, revealed that surface roughness has a moderate effect on retention: when the roughness increases by two orders of magnitude (from <1 nm to ~100 nm), retention increases but only by a factor of ~1.5. This we can see in Figure 5c. The two different curves are for sample types where a different bias voltage (0 V vs. -700 V) has been applied during the deposition [18], resulting in rougher or smoother surfaces on average and, consequently, enhanced or reduced fuel retention.

The average surface roughness is not the only critical parameter but the type of structures formed on the surface play also a role. Based on AFM images in Figure 5d, large irregularly-shaped features with heights >500 nm have been formed on the surface of pure Be (bias voltage

0 V) but also on pure W several needle-like structures have been formed - albeit they are considerably shallower (maximum height ~50 nm). Such spikes appear to favour accumulation of D in their vicinity as can be seen from the jump of fuel retention from 6 to 9×10^{16} at cm^{-2} (Figure 5c) when changing the sample stoichiometry from Be:W=1:1 to 0:1. The mixed coatings of Be-W show typically smooth surfaces, thus fuel retention remains low independent of the Be:W ratio.

3.4 Differences in retention between co-deposited and implanted coatings

Ion implantation was performed on selected 0.4-µm thick Be, Be-O, Be-O-C, and Be-W samples and the results suggest radical differences in their retention behaviour [19]. During the implantation phase, the fluence was adjusted such that the D concentration could be increased to several at.%. In the case of Be-C-O, the retention was the largest (7-8 at.%) while in the Be-O and Be layers it remained in the range of 1-4 at.%. The depth profiles of the measured coatings in Figure 1c reveal, quite expectedly, that the D peak resulting from implantation is concentrated at the film-substrate interface (depth 0.3-0.4 µm).

The data from implanted Be-W samples reveals that, in contrast to the results obtained from co-deposits (see section 3.3), fuel retention follows their Be content: the highest D surface densities were determined for pure Be samples [18]. In addition, the largest relative D concentrations were observed for the mixed Be:W films - despite them exhibiting the smoothest surfaces. Generally, surface roughness does not play a large role in retention resulting from implantation. All this indicates that implantation does not fully reproduce all the features obtained for co-deposited samples unless a wide energy spectrum is used to extend the D profile up to the surface.

3.5 Comparison to deposits obtained from JET-ILW

The deposits studied here were designed to resemble as much as possible the layers formed on the surface of the inner divertor Tile 1 of JET-ILW [6-8]. The main properties of these deposits are summarized in Table 2, together with the summary of the data extracted from the Be-C-O-D coatings reported in previous sections. One can notice that the O and C contents are relatively close to the target values while the Be and D fractions differ considerably between the two cases. Possible reason for the discrepancies could be that the JET-ILW samples have been exposed to numerous different plasma discharges, resulting also in heating of the surface, including transiently during ELMs. This may favour evaporation or outdiffusion of deuterium and thus reduce retention. Furthermore, the configuration of defects and bonding mechanisms within the produced layer may be altered resulting from temperature excursions.

In terms of the release of the retained D, the co-deposits studied here show many similarities to the TDS data extracted for samples originating from the JET-ILW Tile 1 [20]. In the JET-ILW case, the TDS curves contain two distinct peaks in the interval ~320-420°C and around 550°C, indicative of release from traps. These are to be compared with Figure 3b where a two-humped peak structure at ~350°C and ~470°C is visible for the Be-C-O-D sample. The other examples in Figure 3b do not resemble any of the JET-ILW data curves, proving that for fuel retention replicating the exact composition and chemical fingerprint are crucial. To conclude, the Be-C-O-D structure represents a step towards the desired direction but work is still to be done. The motivation for further development is also pointed out by the large fraction of D that remains in the JET-ILW tiles after the TDS cycle [10] while the laboratory-made co-deposits can be more easily emptied of their fuel inventory.

4. Conclusions

We have investigated retention of plasma fuel in beryllium-based co-deposits that would be proxies to the layers observed on JET-ILW or predicted for ITER. The prepared samples consisted of D-doped Be, Be-O, and Be-C-O layers, in addition to which D-containing Be-W deposits with varying Be:W ratios were produced. Moreover, for each sample type a series on non-doped films were prepared and implanted with D at a pre-determined energy and fluence.

The main results can be summarized as follows:

- The thicker the sample, the larger will be the amount of D retained in the growing film, apparently due to the sample structure becoming stabilized at thicknesses > 1 μm.
- The largest D levels were observed for Be-C-O-D samples, and the relative atomic fractions could be increased to 40-50 at.%. However, controlling the D amount by adjusting the deposition parameters could be done only for Be-D films.
- The retained fraction of D in the co-deposits increases with the decreasing O content which, for its part, can be controlled by adding more C in the lattice. In contrast, inclusion of N in the produced layer does not alter its retention characteristics.
- When the O levels are below ~5 at.%, only the C content of the films influences fuel retention. The role of defects and various traps as well as strong C-D and O-D bonds largely determines the D inventory.
- Increasing surface modifications or roughness enhances retention of plasma fuel but only by a factor of 1.5 following a roughness increase of some two orders of magnitude.
- In implanted samples, the retention follows their Be content up to the applied fluence, while roughness plays hardly any role.

Compared to the JET-ILW samples, the Be-C-O-D deposits show many similarities especially when it comes to their C and O contents and the characteristics of fuel release but the Be and D concentrations are way off from the target values. The discrepancies will be addressed by altering the surface temperature of the co-deposited layers during their production phase, to better reproduce realistic exposure conditions inside a fusion reactor. Analyses of laboratory samples, implanted with D at different temperatures, indicate that as temperature increases, retention in Be samples diminished but stays constant in Be-C [21]. In addition, higher roughnesses, of the order of several microns, may enhance retention: in these studies, the samples have generally been smoother than those removed from JET-ILW. Finally, our results indicate that the properties of complicated co-deposits on tokamak wall tiles can only be understood by varying a number of different parameters of the laboratory-made layers, not only the composition or thickness.

Acknowledgments

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Figure captions

Figure 1. TOF-ERDA depth profiles extracted for different Be-containing samples. (a, b) Be(79%)-C(8%)-O(8%)-D(5%) with sample thickness of (a) 0.4 μm and (b) 5 μm; (c) D-implanted Be(84%)-C(8%)-O(8%) with a thickness of 0.4 μm.

Figure 2. SIMS depth profiles measured for different Be-containing samples. (a) Be(81%)-O(9%)-D(10%) and (b) Be(76%)-C(7%)-O(7%)-D(10%). The measured thicknesses and compositions of the samples are: (a) d ~8 μm, D~25 at.%, Be~70 at.%, O~3 at.%; (b) d ~6.5 μm, D~45 at.%, Be~35 at.%, C~14 at.%, O~2 at.%.

Figure 3. (a) NRA and (b) TDS data extracted for the samples (i) Be(85%)-C(5%)-O(5%)-D(5%), (ii) Be(90%)-O(5%)-D(5%), (iii) Be(85%)-O(10%)-D(5%), and (iv) Be(95%)-D(5%). The measured thicknesses and compositions of the samples are: (i) d ~7 μm, D~30 at.%, Be~55 at.%, C~11 at.%, O~1 at.%; (ii) d ~2 μm, D~7 at.%, Be~91 at.%, O~2 at.%.; (iii) d ~10 μm, D~25 at.%, Be~70 at.%, O~1.5 at.%.; (iv) d ~6 μm, D~20 at.%, Be~78 at.%, O~0.5 at.%.

Figure 4. Raman spectral data extracted for (a) Be(76%)-C(7%)-O(7%)-D(10%) and (b) different Be-D samples, together with a typical spectrum of a BeD_2 structure.

Figure 5. (a, b) SEM images for the surfaces of Be-O-D samples with D contents of (a) 12 at.% and (b) 26 at.%. (c) Retention of deuterium in Be-W-D samples with different Be:W ratios and bias voltages during deposition. (c) Influence of the Be:W ratio and the bias voltage on the surface roughness and morphology of the produced Be-W-D coatings.

Table captions

Table 1. Target parameters (nominal composition and thickness as well as D content) of the produced Be-containing samples on W substrates and the range of measured values for the true composition of the produced samples.

Table 2. Comparison between the elemental composition of co-deposits measured on JET-ILW Tile 1 and the laboratory samples produced within the scope of this work.

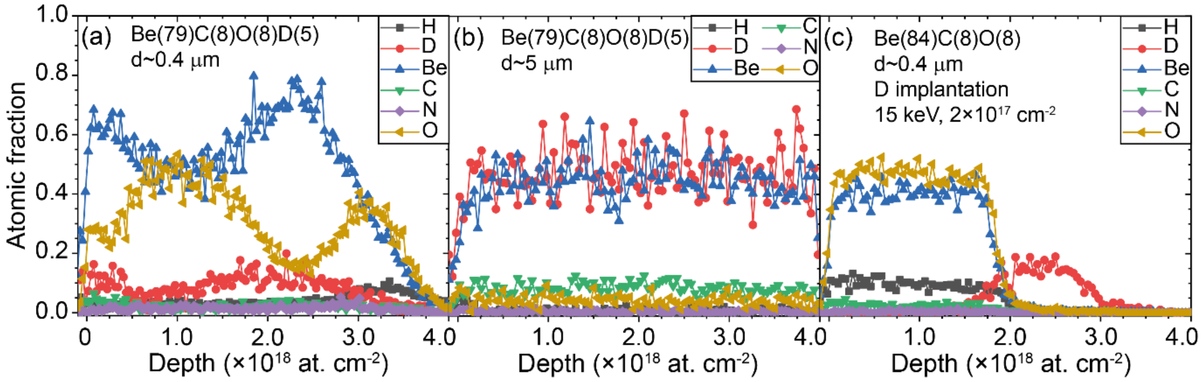


Figure 1.

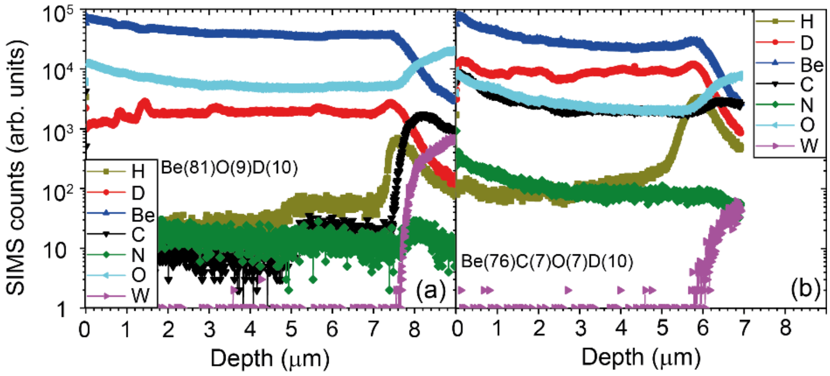


Figure 2.

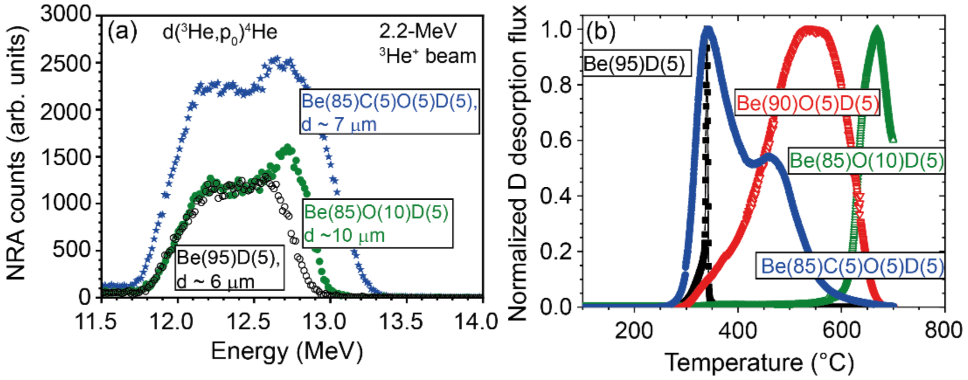


Figure 3.

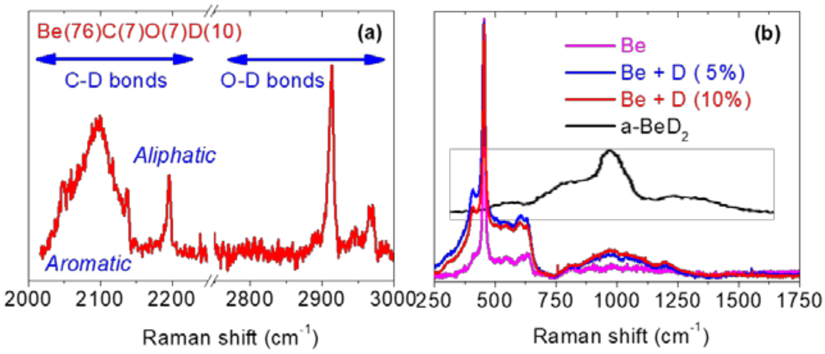
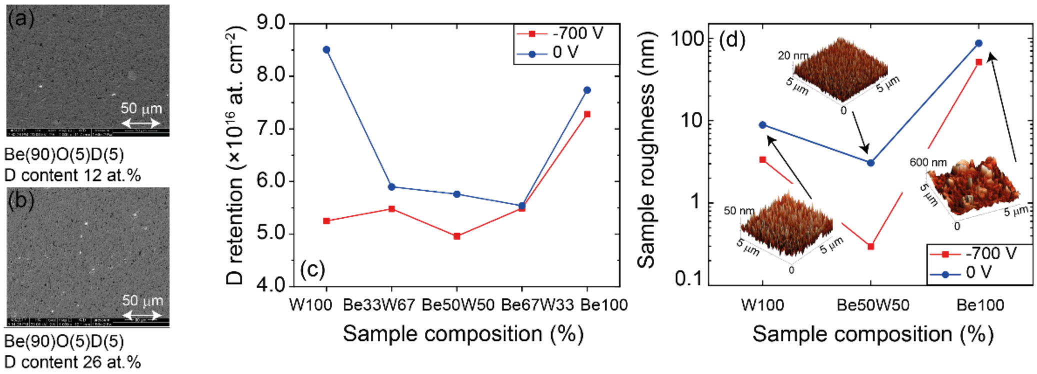


Figure 4.



Be(90)O(5)D(5)
D content 12 at.%

Be(90)O(5)D(5)
D content 26 at.%

Figure 5.

Table 1.

Target values			Measured values (at.%)			
Composition (at.%/at. ratio)	**Thickness (µm)**	**D (at %)**	**Be**	**C**	**O**	**D**
Be(85)-C(5)-O(5)	0.4-15	5	50-75	4-14	1-20	3-40
Be(79)-C(8)-O(8)	0.4-15	5	40-65	2-11	4-40	2-45
Be(76)-C(7)-O(7)	5	10	30-40	11-17	2-13	40-50
Be(90)-O(5)	0.4-5	5	38-90	<4	1-50	1-7
Be(85)-O(5)	0.4-5	10	40-50	<3	40-50	1-9
Be(85)-O(10)	5	5	80-85	<0.5	<2	14-19
Be(81)-O(9)	5	10	65-75	<0.5	<3	20-26
Be(99)	1-10	1	93-99	<0.5	<1.5	0.5-7
Be(95)	0.4-5	5	78-96	<0.5	0.5-3	2-20
Be(90)	1-10	10	73-90	<0.5	<1	10-20
Be:W (1:0, 2:1, 1:1, 1:2, 0:1)	1	>1				

Table 2.

Element	**JET-ILW (>15 µm) [5]**	**Co-deposits (>5 µm)**
Be	75-85 at. %	40-70 at.%
O	6-9 at.%	1-4 at.%
C	3-10 at.%	8-17 at.%
D	4-5 at.%	20-50 at.%