6. Neutron irradiation effects-1 (Properties of Beryllium)

6.1 Carbon and Tungsten Effect on Characteristics of Sputtered and Re-deposited Beryllium Target Layers under Deuteron Bombardment

L.S. Danelyan[1], N.G. Flistratov[2], V.M. Gureev[1], M.I. Guseva[1], B.N. Kolbasov[1], V.S. Kulikauskas[3], V.G. Stolyarova[1], N.N. Vasiliev[1], V.V. Zatekin[3], A.M. Zimin[2]

[1]*RRC «Kurchatov Institute», Nuclear Fusion Institute, Kurchatov sq. 1, 123182 Moscow, Russia*
[2]*N.E. Bauman Moscow State Technical University, 2nd Bauman str. 5, 105005 Moscow, Russia*
[3]*Institute of Nuclear Physics, M.V. Lomonosov Moscow State University, Vorob'yovy gory, 119899 Moscow, Russia*

Abstract

The behavior of the plasma facing Be-elements in the International thermonuclear experimental reactor ITER will be affected by the re-deposition of other eroded plasma facing materials. The effect of carbon- and tungsten-additions on the microstructure, chemical composition and hydrogen isotope accumulation in the sputtered and re-deposited layers of beryllium TGP-56 at its interaction with 200-300-eV hydrogen isotope ions was studied in the MAGRAS facility.

1 INTRODUCTION

The protection of plasma facing components is an important issue at designing the International Thermonuclear Experimental Reactor (ITER) [1,2]. The protective armour will coat the plasma facing elements of the first wall, the divertor, and the limiter. The materials of this armour will be subjected to combined plasma effects [3].

To model possible patterns of plasma interaction with the first wall armour material under expected ITER conditions, we used a MAGRAS test facility [4] equipped with a magnetron sputtering system (MSS) in which different targets are exposed to a flux of 200-300-eV hydrogen isotope ions.

Currently, beryllium is being considered as the candidate material for fabricating the ITER first wall protective armour. The necessity of modelling the beryllium sputtering and erosion product deposition stems from the lack of adequate information regarding the transport of sputtered plasma facing beryllium and hydrogen isotope accumulation in it under ITER conditions. Inside the ITER vacuum vessel, sputtered erosion products will be re-deposited onto the armour made of different materials which covers the first wall and other PFC components significantly affecting its surface condition and properties.

The key benefits offered by MAGRAS are the ability to adjust the ion flux over wide energy and density ranges and the possibility to obtain required irradiation doses much quicker than they actually will occur within ITER, thus shortening the time of testing. It is also important that the experiments are low in cost, and the facility is simple in structure.

2 EXPERIMENTAL CONDITIONS AND DIAGNOSTIC TECHNIQUES

MSS with the permanent magnets operates under relatively high H and D pressure within the chamber and produces an H^+ and D^+ ion flux onto the investigated target. We used the compound target consisted of four 2 mm thick polished central sectors, pressed to the magnetron cathode by a beryllium ring. The sectors were either all-beryllium or made of different materials. We studied the targets composed of beryllium and tungsten (another PFC armour material). In this case, the targets were differently combined in terms of Be:W area ratios (Fig. 1 (a-c)) and used for studies of mutual re-deposition of the erosion products. Due to high pressure within the

chamber, the cathode erosion products scatter on the ambient gas and predominantly return back to the target surface. In cases of mixed-material targets, we examined the re-deposition patterns. The average irradiation dose in the experiments was $4x10^{25}$ m^3 , and the temperature of exposed specimens was 400 ± 20 K. The sectors made of different materials were exposed to ion fluxes of the same energy (200-300 eV). Due to azimuth drift of electrons in MSS, the discharge was homogeneous even when the cathodes were made of materials with significantly different properties [5].

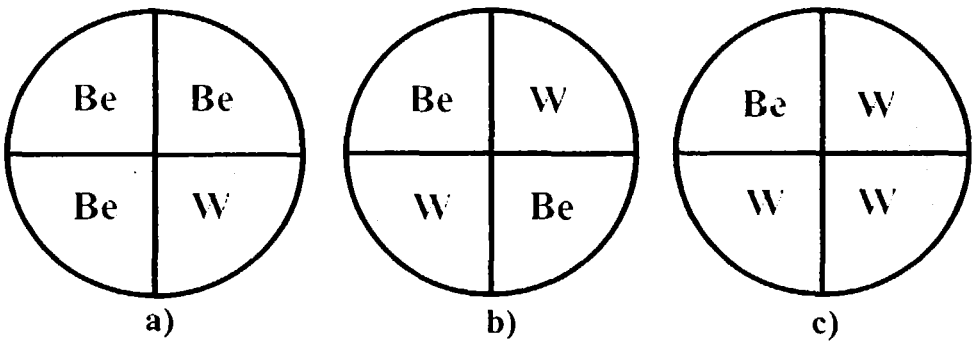

FIGURE 1. Compound sector targets with different Be to W area ratios.

Effect of carbon, which is one of the candidate materials for high-heat-flux divertor elements in ITER, on the Be-behaviour was studied by placing different C-based materials at about 30 mm from Be-targets. The targets CT-1 and CT-2 were exposed to D^+-ion doses of 810^{24} and 210^{25} m^{-2} with temperatures (controlled by a thermocouple) of 350 and 420 K, respectively.

Apart from the temperatures of targets and erosion product collectors, we registered the discharge electrical characteristics (current and voltage) and its outward appearance as well as the pressure of the plasma-producing gas. A set of diagnostic techniques was used to analyze the eroded areas and re-deposited layers, including:
- gravimetric analysis – to measure the weight change of tested targets with an accuracy of 10^{-4} g;
- scanning electron microphotography (microscope JEOL) – to examine the microstructure of materials subjected to H^+ and D^+ ion bombardment and the topography of re-deposited layers;
- profilometry (profilometer STYLUS) of targets – to measure the erosion depth and thickness of layers re-deposited on the target;
- X-ray diffraction and electron-diffraction analyses – to examine the phase composition of eroded surface and re-deposited layers;
- elastic recoil detection method – to determine the distribution of hydrogen isotope atoms in the depth of the surface layers. In this method, 1.8-MeV helium ions accelerated in a Van de Graaff accelerator bombarded the target surface at an angle of 15°. The recoil atoms were registered at an angle of 30° to the initial incident He-ion beam;
- Rutherford backscattering of 1.5-MeV helium ions in the Van de Graaff accelerator at a scattering angle of 160° – to study chemical composition of different target zones and erosion products.

3 RESULTS AND DISCUSSION

Fig. 2 shows schematically the post-irradiation structure of the target sector. There are fairly well-defined annular centrally symmetric zones. Deposition of sputtered material took place in zone 1. Zone 2 is a zone where Be-sputtering and re-deposition of the sputtered atoms occurred.

It is separated from the re-deposition zone 4 by a narrow dark zone 3 adjoining the discharge boundary. During the ion bombardment, the exterior ring of sector 5 was shielded by a screen.

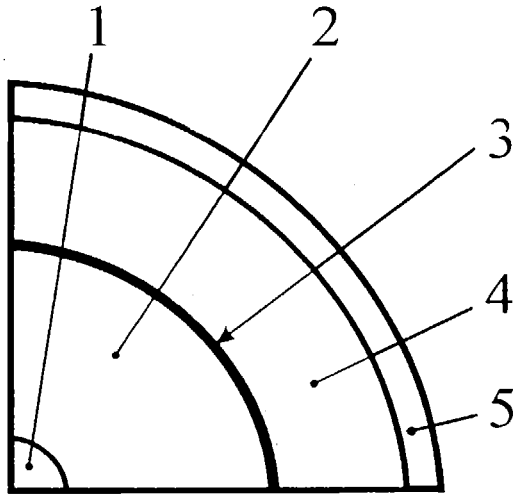

FIGURE 2. Post-irradiation structure of a target sector: (1) deposition zone; (2) zone of Be-sputtering and sputtered atoms re-deposition; (3) dark zone; (4) re-deposition zone; (5) exterior sector ring shielded by a screen during ion bombardment.

Effect of Carbon on Be-Behaviour

We measured geometric characteristics of sputtering and re-deposition zones. Zone 2, which is predominantly a sputtering area (Fig.2), thinned in the CT-1 target by 3.75 μm, according to profilometry measurements. The deposit thickness, *d*, in zone 4, which is a beryllium re-deposition area, increases with growth of irradiation dose and reaches ~80 nm and ~170 nm on average for CT-1 and CT-2 targets respectively. It varies radially and reaches around 230 nm within the space of 1 mm in zone 4 of the CT-2 target.

The microstructure analysis of the irradiated target surfaces revealed that blistering had also occurred in the sputtering zones. The blisters, unlike those found on surfaces unaffected by carbon impurities, were 0.5 - 5.0 μm across. Small blisters typical for hydrogen isotope ion irradiation were found both on blister caps and target surfaces. Some of the larger blisters were broken. We believe that the generation of the larger blisters is due to the presence of C and H atoms in the near-surface parts of the targets and the formation of CH_n (CD_n) molecules. In other words, larger blisters may result from ~~the~~ accumulation of hydrocarbon gas molecules.

The analysis of recoil atom spectra revealed thin C- and D-enriched surface layers presence in each zone of the target [6]. Deuterium was found not only in the zones subjected to ion bombardment, but also in the deposits unaffected by ion fluxes. Fig.3 shows the distribution of chemical elements in the re-deposition zones of CT-1 (*a*) and CT-2 (*b*) targets. This, especially D-accumulation profile, was a subject of inquiry for this series of experiments. Table I summarizes the results of the analysis of D and O accumulation in the targets.

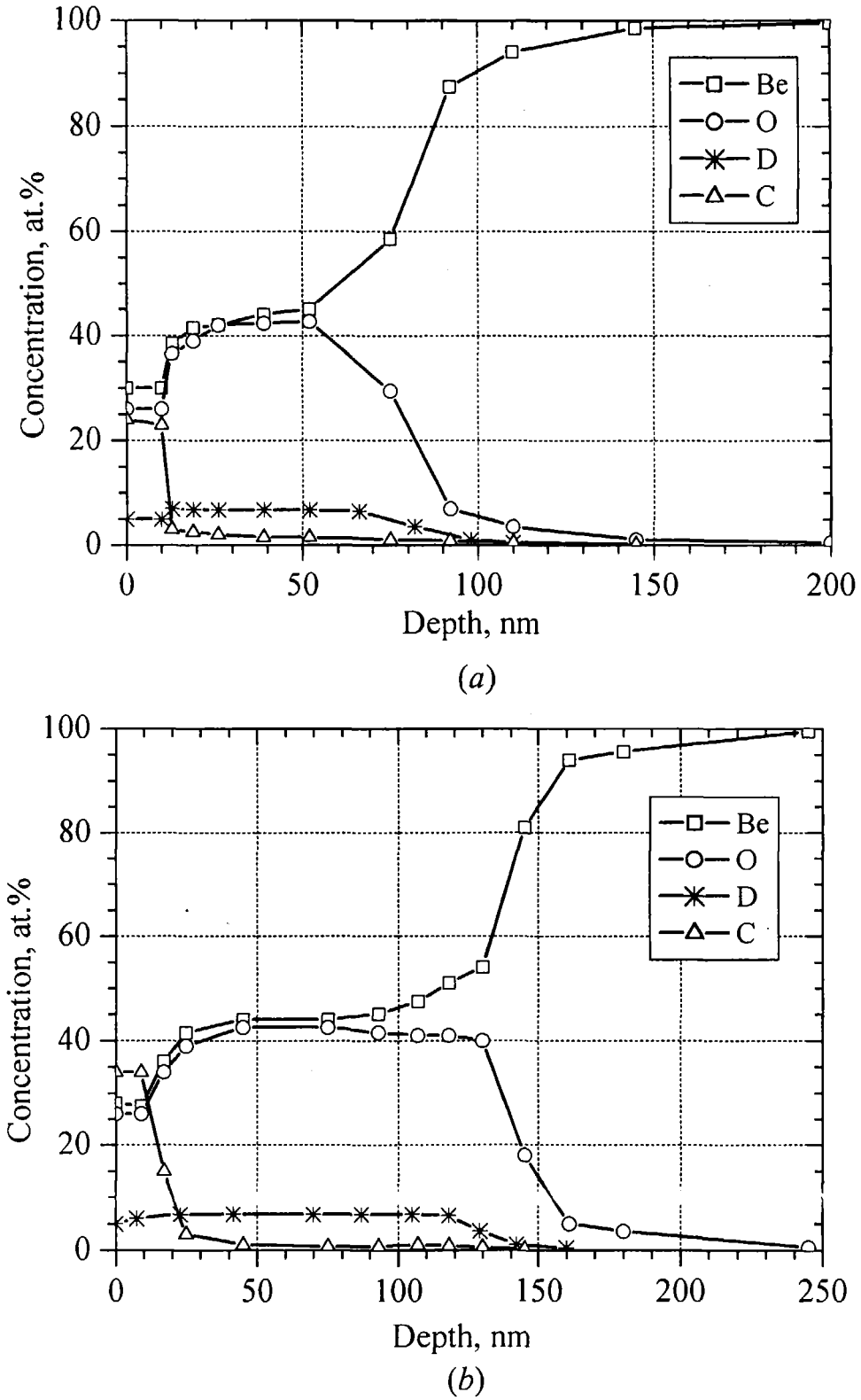

FIGURE 3. Depth distributions of chemical elements in re-deposition zones of CT-1 (*a*) and CT-2 (*b*) targets.

Table I
Characteristics of the Be-based layers of CT-1 and CT-2 targets subjected to D^+-ion bombardment

Target	T, K	Dose, m^{-2}	d, nm	O/Be	C_D, m^{-2}	D/BeO	O/Be	C_D, m^{-2}	D/Be
			Re-deposited			layer	Sputtering		zone
CT-1	350	8.10^{24}	80	1.0	$8.50 \cdot 10^{20}$	0.16	0.10	$3.00 \cdot 10^{20}$	0.07
CT-2	420	2.10^{25}	170	1.0	$1.83 \cdot 10^{21}$	0.16	0.15	$1.63 \cdot 10^{20}$	0.04

D-atoms distribution profiles for sputtering zones of CT-1 and CT-2 targets have a table-like shape like the case of pure beryllium. For the CT-2 target (exposed at a higher temperature), it was slightly blurred. The integral D-concentration, C_D in the sputtering zone decreases with temperature growth, and the D/Be atomic ratio is reduced from 0.07 at 350 K to 0.04 at 420 K. These results are close to results of measurements made for co-deposited films by R.Causey *et al.* controlling oxygen content in co-deposited films [7]. This may be due to re-emission of deuterium from the target driven by opening of large blisters. The D- distribution in the sputtered parts of the target is confined to the narrow near-surface area.

In the re-deposition zone, where Be atoms accumulate, integral D-concentration increases with increasing thickness of the deposited Be-layer. Evidently, in this experimental conditions, Be works as a getter. In each of the targets, D-distribution in the re-deposited layers is fairly uniform, although has different extent in depth. Generally, in the presence of carbon the D-concentration in both sputtering zone and re-deposited Be-based layers is much higher than that in its absence.

In the re-deposition area along most of the deposit depth atomic ratio is O/Be ≈ 1. This ratio in bombardment-cleaned sputtering zones is much lower (0.1 and 0.15 for CT-1 and CT-2 targets respectively), but it is essentially higher than that without carbon. Apparently, C-impurities promote not only deuterium but also oxygen concentration increase. The O-distribution profile has maximum in narrow, 10-15 nm-deep, near-surface layers. Carbon concentration in sputtering zones is also lower - 10-12 at.% in comparison with 24-35 at.% in re-deposition zones.

Co-sputtering and Co-deposition of Beryllium and Tungsten

As mentioned above, some of our experiments were carried out with circular CT-2 targets composed of Be and W-sectors differently combined in terms of Be:W area ratios (see Fig.1). The experiments have shown that W-content in the re-deposited layers is practically independent of the Be:W area ratio .The following are some of experimental results for a compound target with the Be:W area ratio of 1:3 (see Fig. 1c). A ~340 nm-deep near-surface layer of the Be-sector has low (< 1 at.%) concentration of tungsten, which is connected with relatively low sputtering and return rates of W. Its sputtering yield is 100 times as low as that of Be exposed to ~~the~~ a deuteron flux of the same energy, and the return to the target surface is hampered by low efficiency of its heavy particles' thermalization on light D-gas.

But for the presence of ~1 at.% W in layers re-deposited on Be-sectors of compound targets, the chemical composition of such layers is very similar to that of homogeneous all-Be targets. The O/Be atomic ratio of ~0.8 persists across most of the re-deposited layer thickness. These facts suggest that sputtered Be-atoms, during their re-deposition onto the target surface, pick up oxygen contained in the residual gas.

The D-distribution in the re-deposited layers on both Be and W sectors is almost uniform. The integral D-concentration in beryllium within these layers is ~ $2 \cdot 10^{21}$ m^{-2}. The D/BeO ratio there

is ~0.12 (slightly greater than that for an all-Be target). It is associated with a higher O-content in the re-deposited Be-layers in comparison with that found in initial material. The thickness of a layer co-deposited with deuterium on either Be or W sectors of the target is within 250-350 nm. It grows with increasing irradiation dose and relative portion of Be-surface area on the target. Apparently, beryllium plays the dominant role in D-accumulating in re-deposited layers when Be, W and D are located within the same vacuum volume.

The study of chemical element distributions across the depth of the near-surface layers in the Be-sector sputtering zones of compound targets showed that O-concentration was ~3 at.%, and that oxygen could be found only in a narrow (several tens of nanometers) near-surface layer. The integral W-concentration in the same layers did not exceed $10^{19}m^{-2}$.

Depth distribution profiles of D-atom concentration in the sputtering zones on the Be-sectors of compound targets had the same table-like shape as in homogeneous all-beryllium targets. The D-concentration there (2.5-4.0 at.%) was essentially lower than in the re-deposited layers. At the same time, D-concentration in the sputtering zones of the W-sectors was 0.2-0.3 at.%, that is, by 1-2 orders of magnitude lower than in the re-deposited layers.

Surface topography of the sputtering zones on the Be-sectors of the compound target is similar to that of an all-Be target and bears evidence of surface blistering. However, separate cones observed on all-Be targets and attributable to different sputtering rates of Be and BeO are absent on Be-sectors of compound targets.

Because of low rate of W-sputtering by D^+ ions with a 200-300 eV energy, close to the W-sputtering energy threshold, the surface microstructure of the sputtering erosion and re-deposition zones on the W-sectors is practically identical with the initial one. Even at higher ion energies, changes in the all-W target surface microstructure are insignificant. The only difference is that the grain boundaries in the sputtering zone of such a target are more clearly distinguishable.

Similar to the case of carbon, the presence of W causes an increase, although to a lesser extent (by ~20%), of the D-content in the re-deposited layers.

4 CONCLUSIONS

1. A series of experiments has been carried out using the MAGRAS test facility equipped with the magnetron sputtering system as a hydrogen isotope ion source to study the sputtering of beryllium targets bombarded by hydrogen isotope ions and the re-deposition of the sputtered material. The phase and chemical composition of the sputtered surface and deposited and re-deposited layers, their microstructure and accumulation of hydrogen and deuterium atoms in them under beryllium irradiation by hydrogen isotope ions have been examined. The layers re-deposited on Be-sectors, under intensive re-deposition, contain ~50 at.% of beryllium and ~40 at.% of oxygen, which may be attributed to oxygen capture by thermalised Be-atoms depositing onto the target surface. The D/BeO ratio in the re-deposition zone is ~0.1, while the O and D content in the sputtering zones on the target is several times lower than in the re-deposited layers.

2. The mutual effect of beryllium, carbon and tungsten (candidate armour materials for plasma facing components of fusion reactors) on characteristics of the sputtered surface and re-deposited layers was studied under conditions of intensive re-deposition. The mutual re-deposition was investigated using compound targets fabricated from different candidate materials. To this end, an azimuthally uniform discharge providing irradiation of different cathodes-targets by D^+-ions

of the same energy was ignited within MSS with heterogeneous cathodes. It was shown that admixture of other materials promote the higher hydrogen isotope content in re-deposited layers. Such an effect may be undesirable in operation of a fusion reactor. Deuterium accumulation in the layers re-deposited on the W-sectors of the target is due to re-depositing Be-atoms.

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