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Structural studies of deposited layers on JET MkII-SRP inner divertor tiles

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Abstract

Deposited layers formed on JET inner divertor tiles during 1998–2004 and 2001–2004 campaigns have been investigated using secondary ion mass spectrometry (SIMS), Rutherford Backscattering (RBS) and optical microscopy. The thickness of the deposit decreases from the top of vertical tile 1 to the bottom and then increases on vertical tile 3 reaching ∼60 μm. There are even thicker deposits on the small sloping section of the floor tile 4 that can be accessed by the plasma at the inner divertor legs. Deposited films on divertor inner wall tiles are enriched in Be indicating chemical erosion of C and a multi-step transport of C to the shadowed area on floor tile 4. The films have generally a layered and globular structure in the areas with plasma contact.
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1. Introduction

Heavy material deposition is found at the inner JET divertor. Detailed studies after each operation campaigns have shown that material is eroded from outer divertor and vessel walls [1–6]. Eroded material consisting mainly of carbon and beryllium flows around the scrape-off layer (SOL) from outboard to inboard and deposits in the inner divertor together with hydrogen isotopes from the plasma. Carbon is subsequently chemically sputtered from the inner divertor deposits, leaving behind films rich in beryllium and other metals that cannot be chemically sputtered, and is transported to regions shadowed from the plasma, such as the inner louvres, where large amounts of hydrogen isotopes are trapped in the deposits [1].

In the period 2001–2004 JET operated with the MkII-SRP (septum replacement plate) divertor replacing the septum structure of the MkII-GB

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divertor with a simple carbon fibre plate. A set of W-coated divertor tiles for erosion and deposition studies was installed prior to the 2001–2004 experimental campaigns. In this work W-coated vertical divertor tiles 1 and 3, and floor tile 4 have been analysed using various surface analytical techniques. The structure of the deposited films is compared with that of the deposited layers observed at JT-60U tokamak.

2. Experimental

JET is operated with plasma facing components (PFC) made of carbon fibre composite (Concept I manufactured by Dunlop Ltd.). During the shutdown in 2001 a poloidal set of divertor tiles was removed and replaced with a set of tiles that were coated with a W-marker layer of 3 μm thickness (prepared by DIARC Technology Inc.). The special marker tiles were retrieved for analysis during the 2004 shutdown (period I). Second poloidal set of divertor tiles which was exposed from 1998 to 2004 (period II) also became available for surface analyses. In this work analysed vertical tile 1 and floor tile 4 are from period I and vertical tile 3 from period II, respectively (Fig. 1). The results presented here are based on campaign-averaged data.

Depth profiles were taken at several poloidal positions of the analysed samples shown in Fig. 1. RBS measurements were carried out using the 3 MeV Van de Graaff accelerator of the University of Sussex. SIMS analysis of the samples was made

with a double focussing magnetic sector instrument (VG Ionex IX-70 S) at VTT. Layer thicknesses were calculated using sputter rates determined with a profilometer from redeposited layers on each tile 1, 3 and 4, separately. Cross-sectional samples for optical microscopy were prepared by cutting the core sample poloidally and placing it into cold mounting epoxy (Epofix, Struers). Polishing was made using Tegrasystem (Struers) with pre-programmed preparation method and automatic dosing system.

3. Results

SIMS depth profiling has been made from a number of samples on inner divertor tiles 1, 3 and 4. Figs. 2 and 3 show typical depth profiles from tiles which were exposed during periods I and II at JET, respectively. For profiles from vertical tile 1, the tungsten containing marker layers are clearly visible at the interface between a deposited film and the CFC substrate. Experimental RBS spectra were simulated using SIMNRA [7]. The best agreement
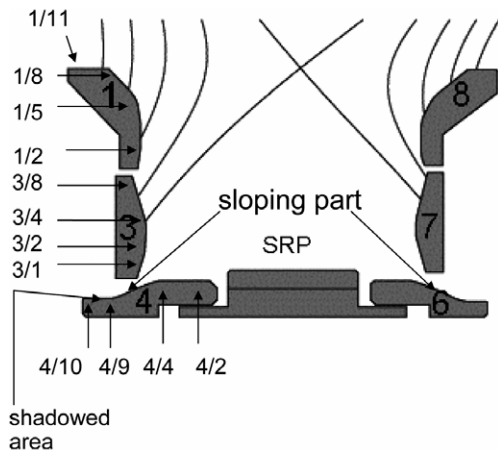
Fig. 1. The JET MkII-SRP divertor tile set. The samples for SIMS and IBA measurements are indicated with numbers. First number in the sample code refers to the tile and the second one to the poloidal location.
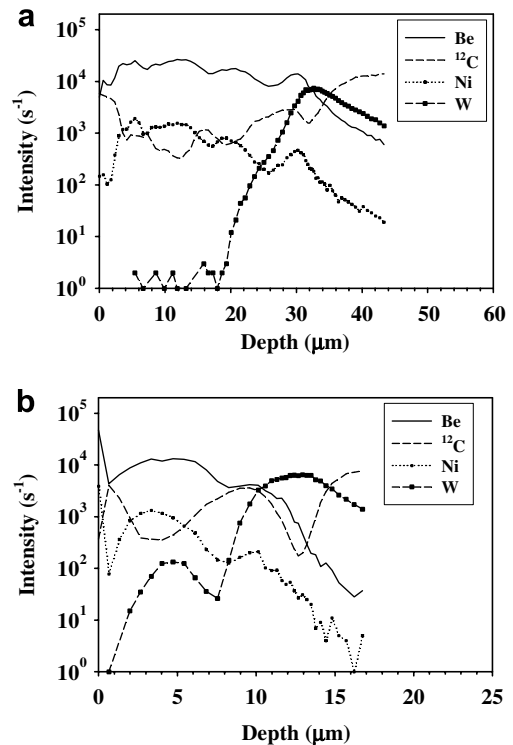
Fig. 2. SIMS depth profiles of Be, C, Ni and W from (a) from top of tile 1 (sample 1/8) and (b) from bottom of tile 1 (sample 1/2) (exposed in 2001–2004).
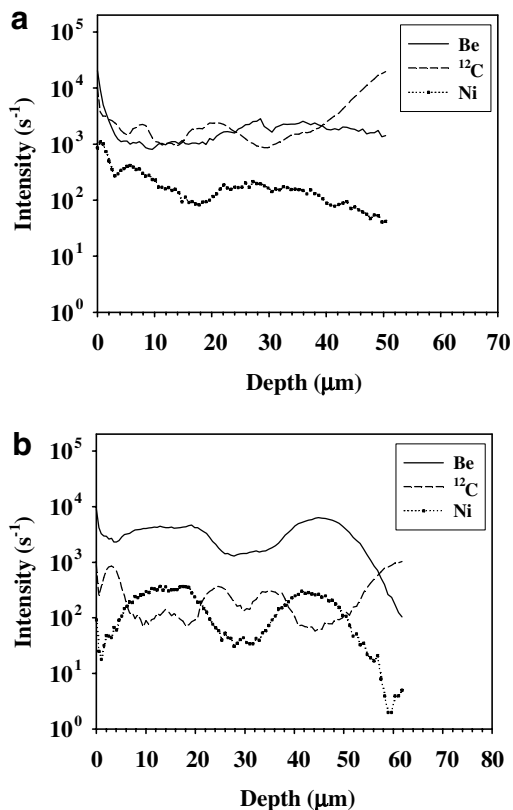
Fig. 3. SIMS depth profiles of Be, C and Ni from (a) from bottom of tile 3 (sample 3/2) and (b) from top of tile 3 (sample 3/8) (exposed in 1998–2004).

Table 2
Composition of the deposit at the bottom of tile 3 (sample 3/2) exposed in 1998–2004 measured with RBS

Layer	D, at.%	Be, at.%	^{12}C, at.%	^{13}C, at.%	O, at.%	Cr, at.%
1	12	3	61	6	17	0
2	5	2	75	0	17	1

high Be/C ratio as the inner part of the 2001 films, with just a small modification at the very surface. The original source of the Be is the Be evaporation performed approximately once a week in the main chamber. Other metallic elements such as Ni, Cr and Fe (from Inconel components in the main chamber) behave in the same way as Be. At the bottom of tile 1 (sample 1/2, see Fig. 2(b)) there is a thinner deposited layer (thickness ~10 μm) with varying Be/C ratio. According to the RBS results the Be/C ratio is 0.4 near the surface. On average the Be/C ratio near the surface is higher on tile 1 removed in 2004 than on tile 1 removed in 2001 [8]. Be evaporation has been done in a similar way during 2001–2004 operations as in 1999–2001. It thus appears that more C was being chemically eroded from the films in 2001–2004 than in 1999–2001. Films on tile 3 are thicker than on tile 1 (~40 μm at the bottom and ~60 μm at the top). Tile 3 was, however, exposed from 1998 to 2004 whereas tile 1 was exposed a shorter period in 2001–2004. The Be/C ratio varies as a function of depth in both depth profiles (see Fig. 3) and at the upper part of the tile (Fig. 3(b)) the Be/C ratio is clearly higher than at the bottom of the tile (Fig. 3(a)). Be and C profiles indicate a layer structure in the deposited film. Ion beam analyses show that Be concentrations on tile 3 removed in 2004 are higher than in the outer layer of tiles removed in 2001, and are comparable with the levels seen in the inner layer formed in 1999–2001 or in earlier campaigns [8]. It is clear, therefore, that chemical erosion of C at the inner divertor returned for the majority of the 2001–2004 operations from the reduced rate at the end of the 1999–2001 campaign to its earlier value, despite the similar JET wall temperature at the end of the 1999–2001 campaign and during 2001–2004 campaign.

The film on the private flux region on floor tile 4 has high C concentration, with some D and O. Be content in the deposit is very low. The film is ~7 μm thick. Deposit on the sloping part (see Fig. 1) of floor tile 4 is much thicker (thickness ~200 μm), but there is a much smaller D content

with the experimental spectrum was obtained by using two different layers in the simulations [6]. Typical composition of the deposited film on tile 1 and 3 measured with RBS is given in Tables 1 and 2. Deposited films are mainly formed of C with varying amounts of D, Be and O. Since the information depth with RBS is few micrometers, in case of thick films only the outer part of the film can be detected.

The deposited film on the top of tile 1 (sample 1/8, Fig. 2(a)) is mainly C with some D, Be and O of ~30 μm thickness. According to the RBS results Be/C ratio is about 0.2 near the surface and SIMS depth profiles show that it varies as a function of depth. The majority of the profile shows the same

Table 1
Composition of the deposit at the bottom of tile 1 (sample 1/2) exposed in 2001–2004 measured with RBS

Layer	D, at.%	Be, at.%	^{12}C, at.%	O, at.%	Cr, at.%
1	14	20	47	18	1
2	13	18	42	24	3

than on the private flux region area [8]. Film on floor tile 4 under vertical tile 3 is also very thick (thickness ~150 μm) with a similar composition to that previously found for the taking deposits at the inner louvres. The D/C ratio varies from 0.2 in the private flux region to 1 in the region shadowed by tile 3.

Figs. 4–6 show poloidal cross-sectional images for samples from tiles 1, 3 and 4. In Fig. 4 the original W marker layer is covered by deposited layer, and the marker layer has survived the plasma operations without any erosion. Deposited film has a layer structure in both analysed samples. Sample 1/1 from the bottom of tile 1 (Fig. 4(a)) has a continuous film on top of the W marker and the outer part seems to be more porous. On top part of tile 1 (sample 1/9, Fig. 4(b)) the film has a lamellar structure on top of the W-marker and individual layers can be observed. On top of the lamellar structure the film is quite porous and consists of globules. The outer surface layer seems to be continuous.

Fig. 5(a) shows a deposited film from the lower part of tile 3 that is porous with globular structure and thicker than on tile 1. The film consists mainly of globules with various sizes. The structure of the films on the centre part of tile 3 is similar to that in Fig. 5(a) even though the centre of tile 3 is the main strike point area. There are only some discharges with strike point on the lower part of tile 3 [6]. The mean temperature of tile 3 before and after a day of pulsing is about 140 °C, but during each plasma discharge (in the divertor configuration) power is deposited on the tile surfaces; the

Fig. 5. Cross-sectional optical microscope image from bottom of tile 3 (sample 3/2) (a) and from top of tile 3 (sample 3/7) (b).

Fig. 6. Cross-sectional optical microscope image from sloping part of tile 4 (sample 4/7) (a) and from shadowed area (sample 4/9) (b).

strike point region reaches regularly temperatures above 1000 °C and the overall energy deposited in the divertor may be 10 MJ. The deposited layer on sample 3/7 (Fig. 5(b)) from near the top of the tile is formed of compact lamellar layers.

Films on the floor tile 4 (see Fig. 6) have different thicknesses, and the films are thinnest on the private flux region and get thicker towards the sloping and shadowed part. In the private flux region the deposited film has a globular structure. The film on the sloping part (Fig. 6(a)) is porous and consists mainly of globules. There seems to be more or less continuous film on the surface. The surface layer and single globules seem to have a lamellar structure with layer thickness of about few microns. The deposited film in the shadowed area (see Fig. 6(b)) has completely different structure than anywhere
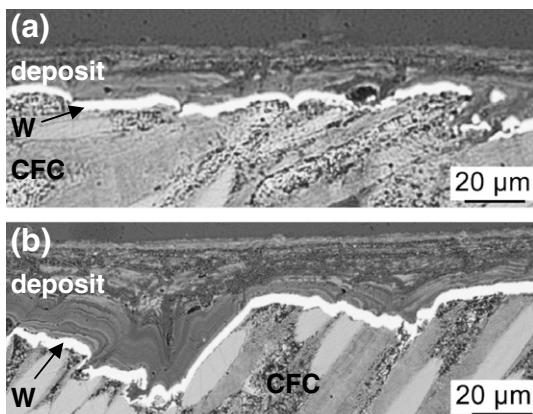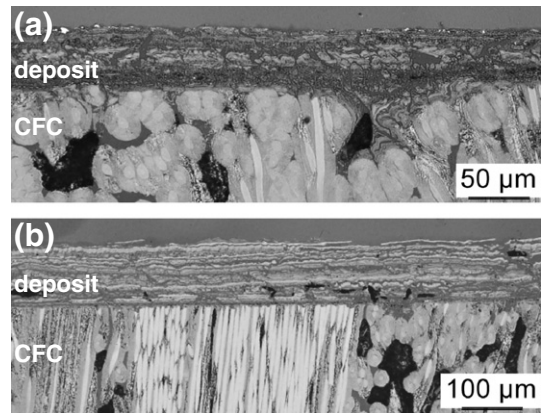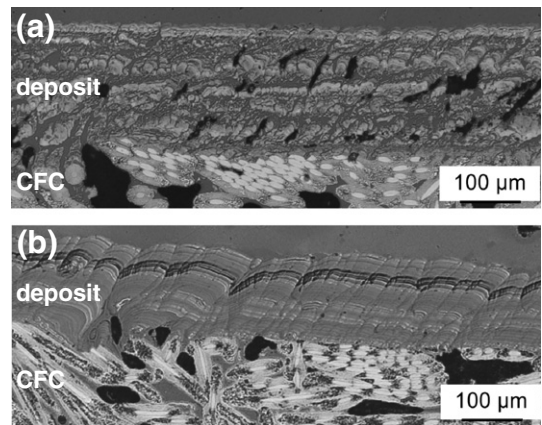

Fig. 4. Cross-sectional optical microscope image from bottom of tile 1 (sample 1/1) (a) and from top of tile 1 (sample 1/9) (b). Deposited film, W-marker layer and substrate marked in the figures.

else at the inner divertor. Fig. 6(b) shows a columnar structure each column having an internal laminated structure. The deposited film has been nucleated continuously as new nucleation centres have constantly been formed throughout the thickness of the film. The laminated structure in Fig. 6(b) has earlier been observed at JET in the deposits from the inner leg of divertor [9].

4. Discussion

A poloidal set of MkII-SRP inner divertor tiles exposed in JET for the 2001–2004 and 1998–2004 operations have been characterised to assess erosion/deposition process. It was found that deposited films at the inner wall of the divertor contain beryllium and other metals for the most part, while carbon is chemically sputtered from these tiles and transported to shadowed regions of the inner divertor. The Be/C ratio of the deposits on MkII-SRP tiles is similar to that on MkII-A tiles, but different from that on MkII-GB tiles exposed in 1999–2001, which had low Be/C ratio on the surface. It has been speculated whether the duplex nature of deposits and the low Be/C ratio on the surface of MkII-GB tiles was due to temperature decrease from 300 °C to 200 °C of the vessel in 2001. The vessel wall temperature throughout 2001–2004 operations was 200 °C indicating that the temperature decrease was not the reason for the duplex nature. The current hypothesis for the low Be/C ratio could be the He-fuelling campaign for one month towards the end of C4 campaign in 2001. The films on tile 3 exposed in period II should be a composite of the film found on the tiles exposed in period I on top of the film deposited during the 1998–2001 campaign.

Gotoh et al. have been investigating systematically cross-sectional samples from JT-60U tokamak with various divertor configurations using SEM and TEM [10,11]. The analysed tiles were exposed in 1988 [11] and in 1997–1998 [10]. Columnar structures were observed for all the re-deposition layers at the inner divertor, while lamellar structures were only observed in the strike point area at shallower depths in the thicker layers as lamellar/columnar-layered structures. They speculated that the lamellar structures could be due to the higher deposition temperatures during high heat fluxes in the strike point zone. At higher temperatures, re-deposition layers may become more graphitic because of the higher probability in C–D bond breaking and the

higher mobility of carbon atoms on the surface. On the other hand, columnar growth-structure with open void boundaries is formed at relatively lower deposition temperatures due to low ad-atom mobility [12]. Columnar growth was explained by ballistic aggregates, i.e. particles moving in straight lines are added to a structure whenever they touch a previously added particle.

The columnar structures shown in Fig. 6(b) are not oriented perpendicular to the tile surface, and the column axis is about 40 °C from the surface normal. This indicates that there can be some correlation between the deposition of particles and the magnetic field lines (see Fig. 1) and the part of the particles that are ionised. There is an empirical relation between the column axis β (from surface normal) and incidence angle α of the incoming particles [13]

$$\tan \alpha = 2 \times \tan \beta.$$

The column axis in Fig. 6(b) is around 40 °C and the formula above gives 60 °C (from surface normal) for the angle of incident particles. This value seems to be somewhat bigger than the poloidal angle of the magnetic field lines. The relation above has been observed to be relatively accurate for the films at the inner divertor of JT-60U. At JET the gap between tiles 3 and 4 is small, about 3 cm, which affects the impurity transport to the shadowed areas. Carbon undergoes a multi-step erosion/re-deposition at the inner divertor and it is finally deposited on the sloping part of tile 4. The final step in the transport into the shadowed areas around the louvres is perhaps by occasional intense bursts of carbon particles when the strike point is placed in the corner of the divertor [6]. Part of this eroded material is neutral particles which do not follow the magnetic field lines. It is thus quite obvious that the tangential relation above is not applicable.

Films formed on the plasma exposed areas at the inner divertor of JET have different structure from the films at JT-60U. JET films have a layer structure which consists mainly of globules, and their size is bigger than at JT-60U. However, globules on tile 4, have an internal lamellar structure which was not observed at JT-60U. Columnar structures in the JET films were observed only in the shadowed region, but not in the areas with direct plasma contact. Different film structures at JET and JT-60U can be attributed, e.g. to different vacuum vessel temperature, to different heat loads and particle fluxes at the inner divertor. Moreover, JET films

contain quite high amounts of Be and O, whereas JT-60U films are mainly C with metallic impurity content <5 at.%. Boron is the main metallic impurity at JT-60U and the source of B is boronisation which was done twice during the experimental campaign [10]. JT-60U was operated at vacuum vessel temperature of 300 °C, whereas tiles 1 and 4 from JET divertor were exposed at vessel wall temperature of 200 °C. JET tile 3 investigated in this work was exposed at first at 300 °C during 1998–2001 plasma operations and after that at 200 °C. It is, however, not possible to correlate this temperature decrease with the structure of the films (see Fig. 6). One cross sectional sample from the shadowed area of tile 4 (exposed in 1999–2001) was also prepared and the structure of the deposited film was similar to that in Fig. 6(b). This indicates that the temperature of the vessel wall does not play a major role in the formation of deposited layers in remote areas where there is no direct plasma contact. Cross-sectional samples from tiles 1 and 4 were made both from the W-coated and uncoated areas and no differences between the deposited layers were observed. This indicates that the substrate material does not influence the growth of the film.

5. Conclusions

Deposited layers formed on inner divertor tiles during 1998–2004 and 2001–2004 campaigns have been investigated using SIMS, RBS and optical microscopy. Heavy deposition was found both on the inner divertor wall and floor tile. Deposited films on divertor inner plasma facing tiles are enriched in Be and O. The films have generally a layered and globular structure in the areas with plasma contact. The structure of the films was compared with redeposition layers formed at JT-60U. Films formed on the plasma exposed areas at the inner divertor of JET have different structure from the films at JT-60U. Columnar structures were observed for all the re-deposition layers at the inner divertor of JT-60U, while lamellar structures were only observed in the strike point area. JET films have a layer structure which consists mainly of globules, and their size is bigger than at JT-60U. Columnar structures in the JET films were observed only in the shadowed region, but not in the areas with direct plasma contact. The reason for different film structure at JET and JT-60U is unclear.

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