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Cross Sections of Deposited Layers Investigated by Micro Nuclear Reaction Analysis

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ABSTRACT.

Cross sections of deposited layers from the divertor of the Joint European Torus (JET) have been investigated, microscopically and by ion microbeam analysis. The thickness of these layers on the studied samples varies between about $50\mu\text{m}$ and $800\mu\text{m}$ depending on the exposure time and poloidal location of the sample. The three main constituents of the layers are C, D and Be. For most of the thicker layers laminar structure is observed. In some locations structure is also be observed in the direction parallel to the original surface, as well as more complex structures.

1. INTRODUCTION

Processes involving erosion and deposition in tokamaks and migration of materials are decisive for successful ITER operation and ultimately for the realization of a technically and economically feasible power producing fusion reactor. Three of the most critical plasma wall interactions issues for ITER are the lifetime of plasma facing components, the tritium inventory in the vacuum vessel and the production of dust [1]. The lifetime of plasma facing components is limited by the net erosion rate, which depends on the balance between erosion and deposition. The build-up of a tritium inventory at the plasma facing surfaces is largely due to co-deposition with the eroded wall material, particularly when carbon is used as a plasma facing material [2], and the disintegration of thick deposited layers is expected to be a major source of dust. Hence it is important to understand the dynamics of erosion and deposition in tokamaks and the properties of deposited layers in tokamaks.

The wall in JET in 1998-2007 has been entirely carbon, with periodic deposition of beryllium by evaporation. The deposited layers at the divertor surfaces have routinely been investigated by different techniques, including optical microscopy [3], ion beam analysis and Secondary Ion Mass Spectrometry (SIMS) [4-8].

Post mortem analyses of plasma facing surfaces give detailed information on the spatial distribution of net erosion and deposition in the divertor, but the resulting data is time integrated over an entire period of operations. The measurements of local deposition rates by quartz micro balances in the divertor are a valuable complement [9]. Another potential time resolution would be if it were possible to relate the stratified deposits at divertor surfaces to a time sequence in an archaeological manner. The thickest layers in the JET divertor are hundreds of micrometers thick. Ion beam analysis with nuclear microbeam for mapping the elemental distribution in cross sections of the deposited layers has been observed to be a suitable analysis technique in particular for the thick layers [10-12]. This report gives examples of layer cross sections from JET divertor floor surfaces, which have been exposed to plasma operation for selected time periods 1998-2007 and how they relate to their exposure history.

2. EXPERIMENTAL

Samples from tiles 4 (inboard floor tile, cf. Fig.1) and 6 (outboard floor tile) were investigated. The tiles had been exposed throughout the operational periods 1998-2001 (7278 plasma pulses with

toroidal current > 1MA), 2002-2004 (6797 pulses) and 1998-2007 (19257 pulses). Tiles 4 and 6 have had the same design during all three divertor configurations and the position of the samples on the tile are specified with sample numbers from 1 to 10, with the smaller numbers indicating locations close to the central septum and the higher numbers towards the louvres [4].

Cross-sectional samples for microscopy and microbeam analysis were cut out, placed into cold mounting epoxy and polished as described in [4,12], using a virtually water free diamond suspension. For simultaneous measurements of beryllium, carbon and deuterium, the nuclear reactions ${}^3\text{He}({}^9\text{Be},\text{p}_n){}^{11}\text{B}$, ${}^3\text{He}({}^{12}\text{C},\text{p}_n){}^{14}\text{N}$ and ${}^3\text{He}({}^2\text{D},\text{p}){}^4\text{He}$ were used, with 3MeV ${}^3\text{He}$ beam [11,12]. The ion beam analysis facility is set up at the 5 MV tandem pelletron accelerator at the Ångström Laboratory in Uppsala. The focused beam spot is scanned over the sample polished cross section of the sample [13]. Proton spectra were collected using two solid state detectors with 1500 μm depletion layer. The detectors were located at 130° relative to the incoming beam, with a total solid angle of 230msr. Typically the beam spot was 20-100 μm in the direction of the deposited layers but only 15 μm wide in the perpendicular direction. With that beam size the achievable beam current was in the range of 300pA. For the 2D elemental maps concentrations were calculated with the simplifying assumption that carbon is the dominant material in the deposited material and without background subtraction [12]. The ratios of Be and D to C for the profiles were calculated without these assumptions. For each depth interval the method described in [11] was utilized for calculating the ratios of deuterium and beryllium to carbon.

3. RESULTS

In Fig. 2 elemental maps for Be and D as well as depth profiles for a sample from tile 6 (exposed in 1998-2007, position 7) are shown and compared with an optical microscope image. This is from the sloping, plasma accessible part of the outer divertor floor. Two areas can be distinguished in the optical image. The lower part, close to the original CFC surface, shows a clearly layered, laminar structure, whereas the upper part shows a more broken up, two-dimensional structure. The laminar structure is clearly visible also in the elemental concentration maps, in particular in the Be distribution. Some of the 2D structure is carried over also in to the elemental distributions, especially close to the surface. There is very little deuterium in the region close to the CFC. The different peaks in the Be depth profile are quite narrow, suggesting that they were formed in a short time span or due to isolated events. In Fig.3, elemental maps and depth profiles for D from four different positions on tile 4 (exposed in 1998-2007) are shown. The innermost position (sample 10), in the plasma shadowed region, has a much higher deuterium concentration ($\text{D}/\text{C} \approx 0.54$) than the other ones (D/C from less than 1% to 7%). The deuterium distribution is mostly laminar, especially close to the interface. The thickness of the sample from position 7 (at the sloping part of the tile) is roughly the same, but the deuterium profile there is very different, with lower concentration, laminar structure and peaks near the interface and near the surface. As the layers get thinner towards the centre of the divertor the deuterium concentration in the peak close to the interface between CFC

and deposited layer grows higher as can be seen in Fig. 4, which shows the different profiles for layers corresponding to the plots in Fig.3.

In Fig.5 the depth profiles for position 6 on the sloping part of tile 6 shows a similar structure with 5 peaks as can be seen in Fig.3(d) for sample 7 of tile 6 that also is on the sloping part. Along with this plot there are also plots for samples at this position but for tiles exposed at different periods. In Table 1 the measured thicknesses are summarised for positions 6 on tile 4 for several different time periods.

DISCUSSION AND CONCLUSIONS

The elemental mapping with a microbeam of cross sections provides reliable absolute composition of the deposited layers and allows for depth profiling even of the thickest layers, which are more than 500 μm thick. This makes the method a valuable complement, on one hand to SIMS depth profiles, which are difficult to make quantitative and are impractical for the thickest layers, and on the other hand to ion beam analysis from the surface, where the accessible depth is limited to $<10\mu\text{m}$. Elemental mapping of cross section also provides information on small scale spatial variations.

Earlier investigations have shown comparatively very little deposition in the outboard channel of the divertor (tile 6), thick deposits in the inboard divertor channel (tile 4) and in particular high levels of deuterium retention in the thick deposits in shadowed areas of tile 4 [9,14]. This pattern is interpreted as the result of a redistribution of deposited carbon from around the strike point towards the shadowed areas of the inner louvre, where due to lower electron temperature and lower surface temperature the deposited layers can incorporate more hydrogen [1]. This pattern is seen in the series of profiles in Fig.3. The deuterium concentration is comparatively low everywhere except in the shadowed area of tile 4 (Figure 3(a)). There has been a narrower region with thick deposits on tile 6, in the plasma accessible sloping region [14].

The layer thicknesses, determined by optical microscopy, and elemental mapping of polished cross sections agree quite well, as expected. The untreated, non polished cross sections appear to be thicker. However, since the analysed areas are not in precisely the same position, the discrepancies can be due to spatial variations in layer thickness. The visual microscopic appearance of the layer structure is qualitatively well reproduced in the elemental distribution, in the areas where there is a clear modulation of the layer composition, such as in Fig.2(b) and (c). When comparing the layer thicknesses on samples exposed 1998-2001, 2002-2004 and the whole period 1998-2007 respectively the missing last period would have to show an increased net deposition from 4-8nm per pulse with plasma current greater than 1MA to about 15nm for position 6 on tile 4. Similarly, the deposition rate at position 6 on tile 6 increased from 10-20nm per pulse to 40nm per pulse. This increase was observed also in [8]. In order to find a closer correspondence it would be necessary to make more detailed statistics on types of pulses, in particular on pulses with different strike point positions [8,9,14].

The large scale spatial variations in layer composition were reported on in [8]. There was an

increasing trend in Be/C on the sloping part of tiles 4 and 6, going from the MkII-GB divertor to the MkII-HD divertor. The Be/C ratio on the sloping part of tile 6 based on ion beam analysis at the surface, with accessible depth about $7\mu\text{m}$, was given as 1-6% for 1998-2001, 10-24% for 2002-2004 and 5-80% for 2005-2007. In the present report the composition was measured throughout the layers and was observed to vary considerably in the range 0-7%, as shown in figures 2(b) and 5(a). The absolute numbers are not immediately comparable, due to the large spatial variation [8], but figure 2(d) also shows an increasing trend in Be/C towards the surface (more recently deposited) region.

Comparing Fig 3d and 5a the sample is thicker in pos 7 compared to position 6 but there are still 5 peaks in the beryllium concentration, although not as clearly visible as in 5a. Overall the maximum concentrations are less in pos 6 compared to pos 7. For beryllium there is a fair agreement in figure 5a for the different periods, suggesting that the archaeological interpretation of continuously growing layers is reasonable, whereas for deuterium no such agreement is observed. This can be understood if beryllium is stored up and more or less stays in place, whereas deuterium may easily be released thermally if the layers long after being deposited are heated up by subsequent plasma operation. For a more conclusive answer whether and for which elements an archaeological investigation is possible, a larger set of samples would have to be investigated.

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REFERENCES

- [1]. J. Roth, E. Tsitrone, A. Loarte et al., *Journal of Nuclear Materials* **390-391** (2009)1-9.
- [2]. T. Loarer, *Journal of Nuclear Materials* **390-391** (2009)20-28.
- [3]. J. Likonen, J. P. Coad, E. Vainonen-Ahlgren et al., *Journal of Nuclear Materials* **363-365** (2007) 190-195.
- [4]. J.P. Coad, P. Andrew, D.E. Hole et al., *Journal of Nuclear Materials* **313-316** (2003)419-423
- [5]. J. Likonen, E. Vainonen-Ahlgren, J.P. Coad et al., *Journal of Nuclear Materials* **337-339** (2005) 60-64
- [6]. J.P. Coad, P. Andrews, S.K. Erents et al., *Journal of Nuclear Materials* **363-365** (2007) 287-293
- [7]. J. Likonen, J.P. Coad, D.E. Hole et al., *Journal of Nuclear Materials* **390-391**(2009) 631-634
- [8]. A. Widdowson, S. Brezinsek, J. P. Coad et al., *Physica Scripta T138* (2009) 014005
- [9]. A. Kreter, S. Brezinsek, J.P. Coad et al., *Journal of Nuclear Materials* **390-391** (2009) 38-43
- [10]. D. Grambole et al., *Nuclear Instruments and Methods* **B158** (1999) 647-652
- [11]. H. Bergs aker, B. Emmoth, P. Petersson et al., *Journal of Nuclear Materials* **362** (2007) 215-221

- [12]. P. Petersson, H. Bergs aker, G. Possnert et al. Nuclear Instruments and Methods. B. accepted
- [13]. G.W. Grime, F. Watt, Beam Optics of Quadrupole Probe-Forming Systems, Adam Hilger Ltd., Bristol, 1984.
- [14]. J.P. Coad, J. Likonen, M. Rubel et al., Nuclear Fusion **46** (2006) 350-366

Time period	Thickness (μm)		
	Optical	Polished	Untreated
98-07	150 \pm 30	180	190
98-01	35 \pm 12	30	45
01-04	40 \pm 20	30	90

Table 1: Thickness of the deposited layers from position 6 on tile 4 measured both optically from the polished cross sections, where the uncertainty refers to variations in the thickness of the layer over the 10 mm wide sample, and by the microbeam on both the polished cross sections and on untreated samples.

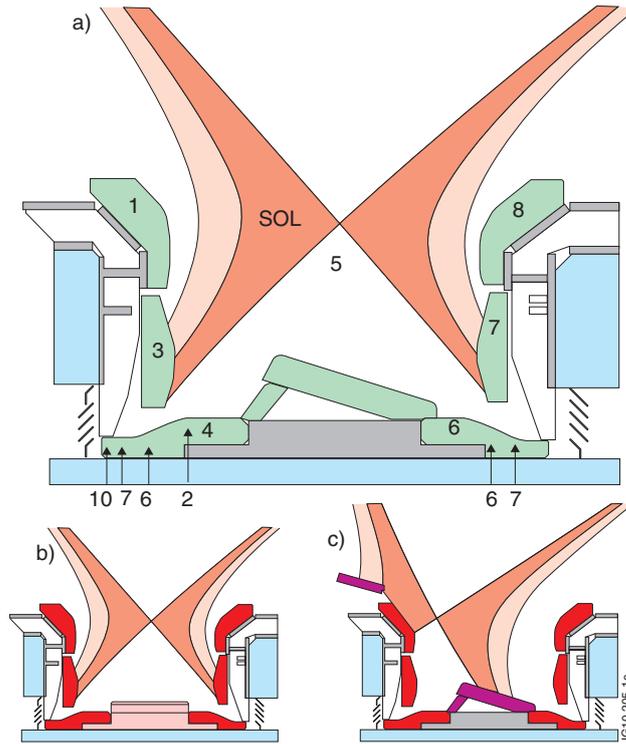


Figure 1: Three generations for the divertor used at JET: (a) MkII-GB (1998-2001), (b) MkII-SRP (2001-2004) and (c) MkII-HD (2004-2009). In (a) the different tile numbers and sample locations on the tiles are shown.

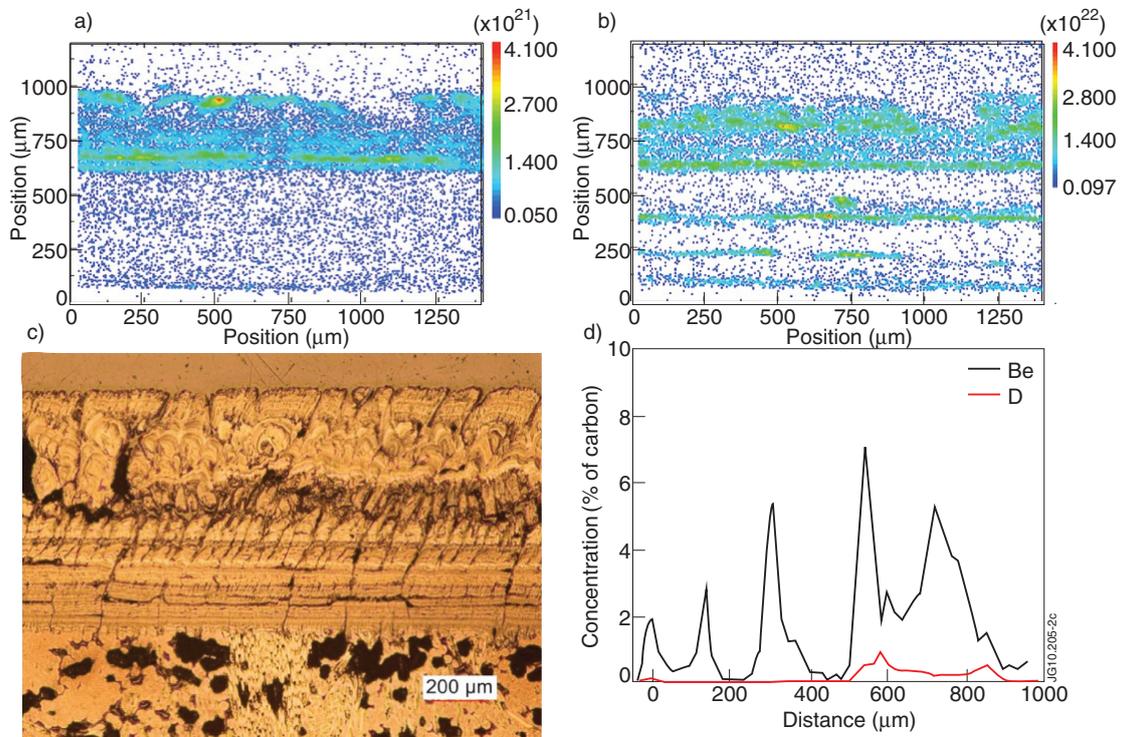


Figure 2: Mappings of deuterium (a) and beryllium (b) from position 7 on tile 6 (exposed in 1998-2007). The depth profiles are presented as a function of the distance from the interface between CFC and the deposits and are shown in (d). In (c) a microscopic image of the deposited layer is also shown.

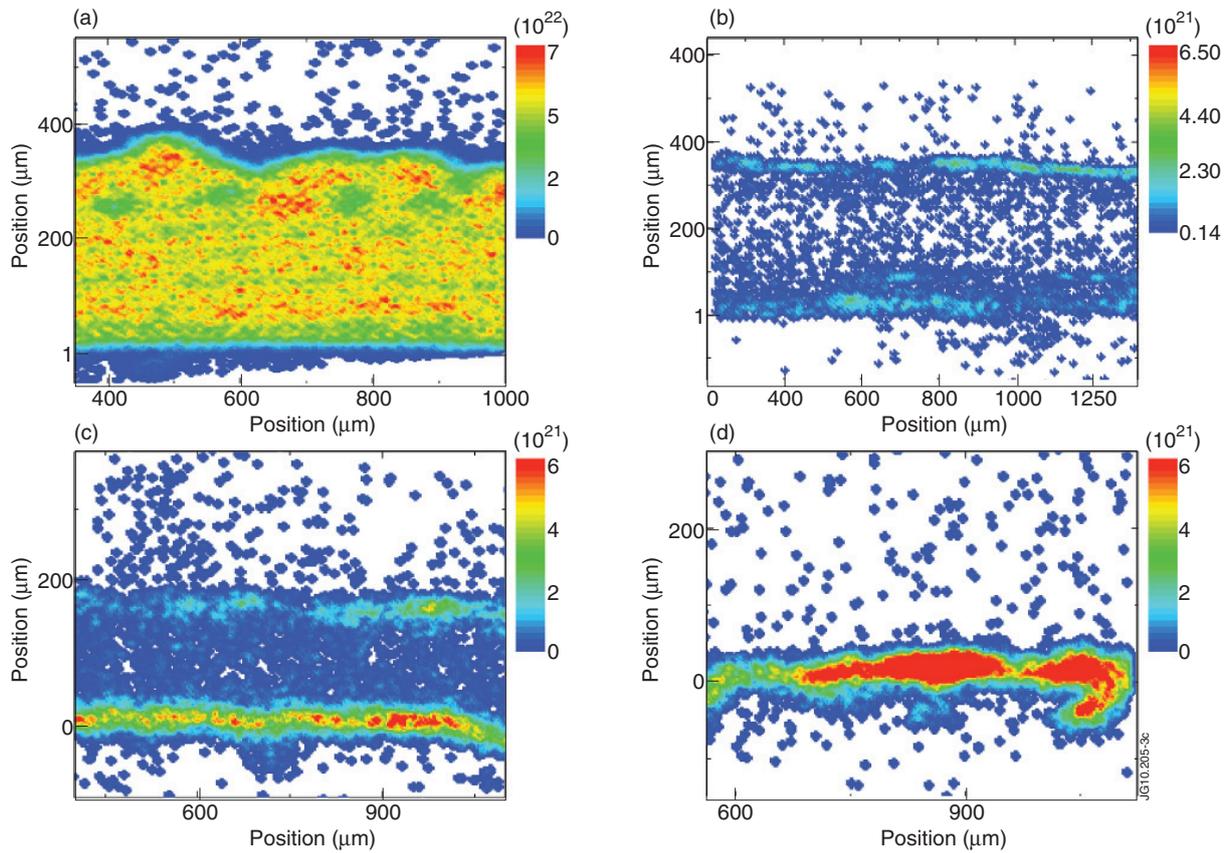


Figure 3: Mappings of deuterium from tile 4 (exposed in 1998-2007) and positions: (a) 10, (b) 7, (c) 6 and (d) 2. For positions see Figure 1.

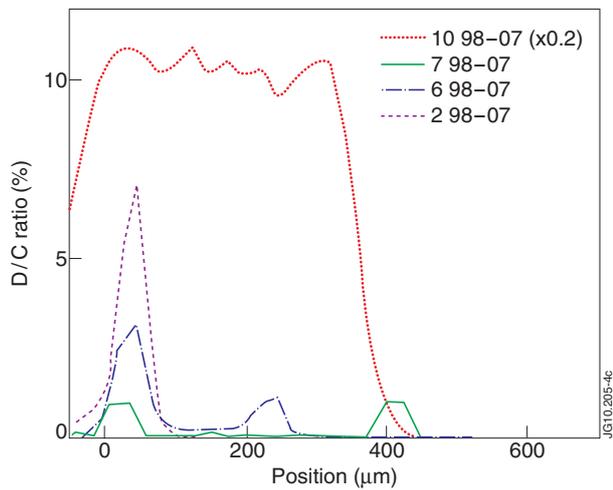


Figure 4: Depth profiles for tile 4, corresponding to the four plots shown in Fig 3. The depth profiles are presented as the distance from the interface between CFC and the depots.

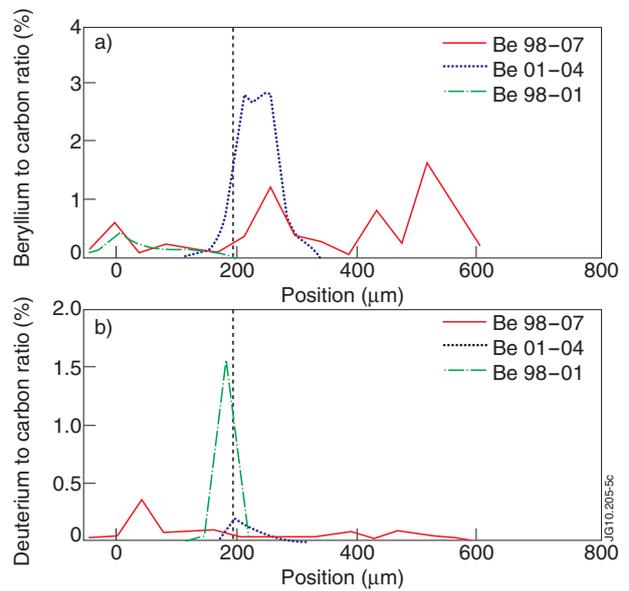


Figure 5: (a) Deuterium and (b) Beryllium depth profiles for position 6 on the sloping part of tile 6 for three different time periods. Both 1998-2007 and 1998-2001 start at zero representing the interface. For the sample from 2001-2004 the profile starts at 195 μm (marked by the dashed line) the thickness of the layer from the sample exposed in 1998-2001 at this position.