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ABSTRACT
Elemental mapping of cross sections of deposited layers on inboard tiles in the JET divertor after exposure to plasma operations with carbon wall are presented. The study was made using microbeam ion beam analysis methods in combination with optical microscopy and SEM. The surfaces had been exposed to plasma through different periods of operation (1998-2007, 2007-2009 and 1998-2009). The texture and composition of the layers are non-uniform. The physical structures include columnar, lamellar and disordered globular appearances. The distribution of trapped deuterium was frequently found to be lamellar, with well-defined sub layers with higher deuterium concentration. However, 3D regions with dimensions of about 100μm with enhanced deuterium content were also found, both at the layer surfaces and in the layer cross sections. The distributions of beryllium and Inconel components were lamellar but did not otherwise show large non uniformity on the same scale length as the deuterium.

1. INTRODUCTION
The choice of plasma facing materials in ITER is beryllium for the entire main chamber wall and either tungsten or a combination of tungsten and carbon CFC for the divertor. Besides the requirement of acceptable core impurity concentrations, the most critical plasma surface interactions issues for ITER and other future big fusion devices are the lifetime of the plasma facing components, dust production and tritium inventory in the vacuum vessel [1,2]. The net erosion rate at some plasma facing surfaces limits the the component life time, while the net deposition at other surfaces leads to the formation of thick layers, which can contribute both to dust formation, when they break up, and to building up the tritium inventory by co-deposition of fuel [1]. The mixing of different materials at plasma facing surfaces makes it more difficult to predict both the erosion properties and the fuel retention [2]. JET was operated between 1994 and 2009 with carbon, mainly Carbon Fibre Composite (CFC) at the most exposed plasma facing surfaces. Some materials migration and mixing occurred due to frequent vapour deposition of small amounts of beryllium at the main chamber wall. From 2011 JET is operated with beryllium as plasma facing material in the main chamber and tungsten in the divertor, an ITER-Like Wall (ILW) [3,4].

The trapping of hydrogen isotopes at tokamak surfaces in areas with net deposition due to co-deposition with migrating wall material was first demonstrated at the sides of the poloidal limiters in JET [5]. Post-mortem analysis of plasma exposed surfaces at JET has been performed routinely following every major shut down, in particular with Ion Beam Analysis (IBA) methods. Thick deposits, which incorporate large quantities of fuel, have consistently been found especially in the shadowed areas of the divertor [6-10]. A proper understanding of how the layers grow and evolve is essential in order to predict the materials migration phenomena in new conditions. While IBA depth profiling of deuterium is possible for layers thinner than about 10μm [5], thicker layers have to be investigated by other means. By cutting through the layers and polishing, the layer cross sections can readily be characterized by optical microscopy [11]. Microbeam IBA (μ-IBA) of layer cross sections is a useful method that was employed already 20 years ago [12]. The microscopic variations in fuel retention in fusion devices have attracted more interest in recent years and μ-IBA has been used both to study layer cross sections and more generally to investigate microscopic inhomogeneities in fuel retention at plasma facing surfaces in fusion devices [13-23]. Microanalysis of layers deposited at the floor of the divertor (tiles 4 and 6) in JET up to 2007 was presented in [20]. In the present report the analysis is extended to tiles 1 and 3 of the inner divertor and includes layers deposited up to 2009.The fuel retention at surfaces is expected to be much reduced with the ILW compared to carbon wall, since beryllium typically traps only a fifth of the hydrogen concentration that can be retained in carbon [1]. One of the goals with the ILW is to verify that prediction [4] and an additional aim of this report is to prepare for the comparison by providing information about the small scale structure of the fuel retention and materials mixing in JET with carbon wall immediately before the change of plasma facing materials.

2. EXPERIMENTAL

Figure 1 shows a poloidal cross section of the inboard tiles of the JET divertor. Samples were cut out of tiles that had been removed during the 2007 and 2009 JET shut downs. The original surfaces and layer cross sections of one set of samples were investigated with Scanning Electron Microscopy (SEM) and with μ-IBA. Another set of samples was used to produce polished cross sections of the deposited layers, which could be studied microscopically [11] and with μ-IBA. The cutting, polishing and optical microscopy was carried out at VTT in Otaniemi. The SEM was made with a Zeiss Ultra 55 instrument at KTH, Stockholm. In the following, the term lamellar will be used for layer structures with visible stratified sub layers parallel to the surface, columnar will be used for structures with prominent features that are nearly perpendicular to the layer surface and globular will be used for all layer structures that look more disordered, but often shows more or less circular spots [11]. The microbeam set up at the Tandem laboratory in Uppsala and the quantification method for the Nuclear Reaction Analysis (NRA) are described in [13]. A beam of 3MeV $^{3}He^{+}$ was used, the Be/C concentration ratio was determined from ratio of the the $^{3}He(^{9}Be,p_0)^{11}B$ and $^{3}He(^{12}C,p_0)^{14}N$ proton yields and the deuterium content from the $^{3}He(D,p)^{4}He$ reaction. Calibration was made using thick elemental samples [13]. Where there is much deuterium, there is some background in the region of the Be p_0 peak in the proton spectrum and where there is much beryllium the Be p_3 peak contributes a background in the region of the C p_0. For depth profile plots the experimental spectra were used for background subtraction in these cases [20]. For 2D maps a simplified and less accurate background subtraction is made. Beam induced release of deuterium [16] and removal of Be and D due to the sample polishing [19] are not believed to influence the results significantly. Particle Induced X-ray Emission (PIXE) was used to study the distribution of medium Z impurities. For layer cross section analysis the ion beam spot size and hence the lateral resolution was typically adjusted to about 15μm in the direction perpendicular to the layer surface and 20-100μm in the parallel direction. For calculating the local composition it is assumed that it is approximately constant over the accessible depth in the direction of the beam, about 5-10μm.

3. RESULTS

Figure 2 shows maps of D and Be from a polished cross section of tile 1, position 10, together with a SEM image from an adjacent area. The tile had been exposed through 2007-2009. Optically this layer is 50-70μm thick and has a lamellar coarse structure, with 8-10 distinct sub layers. SEM shows sub layers with a clearly columnar structure. The Be distribution is lamellar but does not extend to the interface. The deuterium is found largely near the interface and has a globular structure. Figure 3 shows a map of the D distribution at the surface of tile 1, position 8. The sample was exposed 1998-2007. The distribution of D is non uniform, largely localized to irregular spots which are 100-200μm wide and contain at least four times higher D concentrations than the surrounding areas. In this case the D rich spots cover about 14% of the total area. Similar D rich spots were found at the surface of tile 1, position 10, exposed 2007-2009 (~5% of the area) and at tile 3, position 2, exposed (2004-2007) (30% of the area). Figure 4 shows cross section maps of D, Be and Ni for tile 3, position 2, exposed 1998-2009, together with an optical image of the cross section. Also shown are the profiles of D and Be through the layer. The layer is 70-80μm thick and once again optically appears coarsely lamellar, but with globular structure especially in the top sub layer. The bright band at the centre of the layer coincides with the region with high D concentration but lower Be and Ni content. Figure 5 shows profiles of D and Be in layers on tiles 1, 3and 4. Figure 6 shows a comparison of optical images of layers in the shadowed area on tile 4, position 10, from different exposure periods, 1998-2009, 1998-2004, 2001-2004 and 1998-2007. There is a prominent line in all photographs, which helps orienting them. Approximately below that line the layers look lamellar, whereas above it they are globular. The layers deposited 2007-2009 on fresh CFC are optically globular as well. Figure 7 shows D maps for layer cross sections from the same region, for the periods 1998-2007, 2001-2004 and 1998-2007. Once again there is a consistent line of separation between lamellar and globular structure, which can be identified with the shut down in 2004. Figure 8 shows cross section maps of D and Be from the sloping part of tile 4, position 7a, exposed 1998-2009. Optically the layer is 200μm with a lamellar coarse structure, but globular structure in the sub layers. The deuterium is found mainly near the interface with the CFC but also at the layer surface. At position 4/1 exposed 2007-2009 the layer is about 25μm thick and the inferred D/C is about 0.1. The Inconel components Cr, Fe and Ni generally appear together with beryllium in the layers. The concentration is about 1.6×10^{-3} times the carbon concentration at position 1/10 and one order of magnitude less on tile 4. At position 1/10 the composition Cr:Fe:Ni is about 0.17:0.13:0.7.

4. DISCUSSION

The general understanding of materials mixing in JET with carbon limiters and frequent Be evaporation in the main chamber is that C, Be and Inconel components are eroded in the main chamber and migrate in the SOL to the inner divertor, where they are deposited mainly on tiles 1 and 3 [10,24-25]. In the divertor carbon is eroded preferentially due to chemical sputtering and migrates to shadowed areas on tiles 4 and 6, where thick layers are formed, which incorporate large amounts of deuterium [9-10]. However, microanalysis reveals a rich variation in both elemental composition and layer texture in the deposited layers [11,20]. These variations may depend on the local conditions at the time of deposition, in which case a study of the layer cross sections can be interpreted in an archeological fashion. This kind of argument was used in JT-60U, based on SEM studies of layer cross sections [24] and has been used at JET with SIMS depth profiles at divertor tile 3 [7] and with μ-IBA layer profiles from the sloping part of tile 6 [20]. It is also possible that the layer structure changes with time, e.g. due to plasma exposure and heating after they have been deposited. Thus it was suggested for the D distributions on the sloping part of tile 4 exposed 1998-2007 that D may have become mobile when the layer was heated up and trapped at the surface and at the interface with the CFC substrate [20].

A mixture of lamellar and columnar film structures has previously been observed in JT-60U [26]. Columnar layers have been seen also in TEXTOR [27] and inTore Supra [28] and are usually explained in the framework of the structure zone model as ballistic aggregation in case of low surface mobility of the deposited material, which is assumed to arrive preferentially from some direction [29]. The archeologic hypothesis is most likely to be valid in the shadowed region 4/10, where the surface temperature has not been very high and erosion is expected to be small. Indeed, the optical images in Figure 6 as well as the D distributions in Figure 7 fit quite well with the interpretation that the more lamellar structures were deposited up to the shut down in 2004, whereas the globular structures closer to the surface have been deposited after 2004. There is a clear layer sequence also at position 1/1. As the layer deposited in 2007-2009 remains thinner than the previously reported limiting thickness where it starts to peel of at that position [9], it seems likely that the layer structure can be interpreted archeologically. The beryllium and metals at 1/10 are found mostly close to the surface, whereas at position 4/7a the Be concentration is highest near the CFC substrate. This proves that the Be concentration is determined not by the arrival rate of Be from the main chamber, but by the local materials migration in the divertor. The compositional structure of the layer at 3/2 is very similar to the SIMS profiles at position 3/8 after shut down in 2004 [7]. It was interpreted archeologically there, with the conclusion that the chemical erosion of carbon in early 2001 had been reduced due to a He-fuelling campaign, leaving more carbon and hence lower Be/C concentration behind in the central part of the layer. At the sloping part of tile 4, deuterium in the layer deposited 1998-2007 was found predominantly at the interface with the CFC and at the surface [20]. This is true also for the layer deposited in 1998-2009, so the situation is at least not purely archeological. It seems unlikely that this pattern can be associated with layer heating, as the D distribution is reversed at position 3/2, where the inner strike point is often found and the layer is likely to have been frequently heated up. More likely the surface D concentration can be related to the final day of operations before shut down.

Local surface regions with higher D concentration than the surrounding areas were found at several locations in the JET divertor. Similar features have been found at surfaces in the toroidal pump limiter in Tore Supra [14, 21-23] and at CFC surfaces after plasma exposure in PISCES [18] and in TEXTOR [15,18]. The sizes of the features with high fuel retention in these earlier reports range from about 10μm [14] to about 500μm [23] and the local enhancement in fuel concentration has been reported as between a factor two and a factor 80. A plausible explanation [14,22-23] for the local D trapping enhancement is that pores and pits in the CFC substrate provide regions that are partially protected from plasma exposure and where the deposition conditions may favor high fuel retention. This is supported with observed correlations between high D retention and the surface topography that can be observed with SEM [14,22]. This explanation may be applicable to the D rich spots at position 1/8 (Fig.3), since the deposited layer is thin there. Maybe also to the spots with high D concentration at the interface at 1/10 (Fig.1). The argument does not apply as easily to the spots on the surface of the thick layer at position 1/10, or to the blobs with enhanced D content that can be seen near the surface in the cross sections of the layers at position 4/10 (Fig.6). One possibility in these cases is that there has been a local peeling off of a top surface layer, so that a pit in the layer was created where deuterium could more easily be retained. Another hypothesis is that flakes or particles from layers in regions with high fuel content can be transported to surfaces where conditions favor lower fuel content in the deposit, that the particles stick to the surface and are finally buried in the deposited layer.

CONCLUSIONS

The microstructure and the microscopic distribution of deuterium, beryllium and Inconel components in layers deposited on inboard divertor tiles in JET have been studied, for layers deposited up to 2009. Lamellar, columnar and globular structures are observed. A clearly archeologic structure is observed at least in the shadowed region of tile 4, where the parts of layers that were deposited beforec 2004 have lamellar structure, whereas the layers deposited later are globular. The distribution of Be and Inconel components is predominantly lamellar. The deuterium distribution is frequently lamellar as well, but globular distributions are also seen and local spots with 2-5 times higher D concentration are observed both at the surfaces of tiles 1 and 3 and in the layer cross sections in the regions on tiles 1 and 4 which have least plasma exposure.

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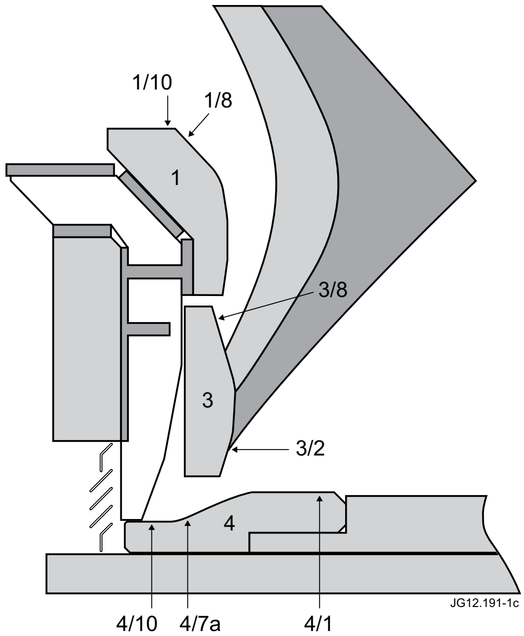

Figure 1. Poloidal cross section of the inboard leg of the JET divertor. Samples from the shown positions on tiles 1, 3 and 4 have been investigated.

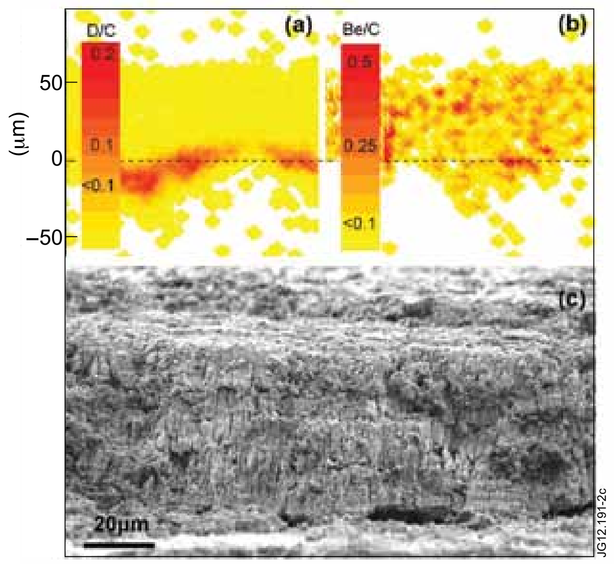

Figure 2. Maps of (a) deuterium and (b) beryllium on a polished cross section of tile 1, position 10, exposed through 1998-2009. (c) SEM image from an adjacent, non polished area of the cross section, showing characteristic columnar structure within lamellar sub layers.

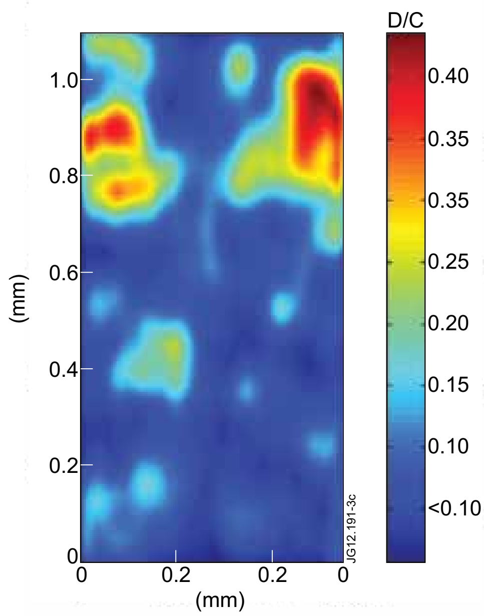

Figure 3. Map of the deuterium concentration at the surface of tile 1, position 8, exposed through 1998-2007. The deuterium is largely localized to blobs with a size of 100-200 µm. Similar distributions are found in several places at tiles 1 and 3.

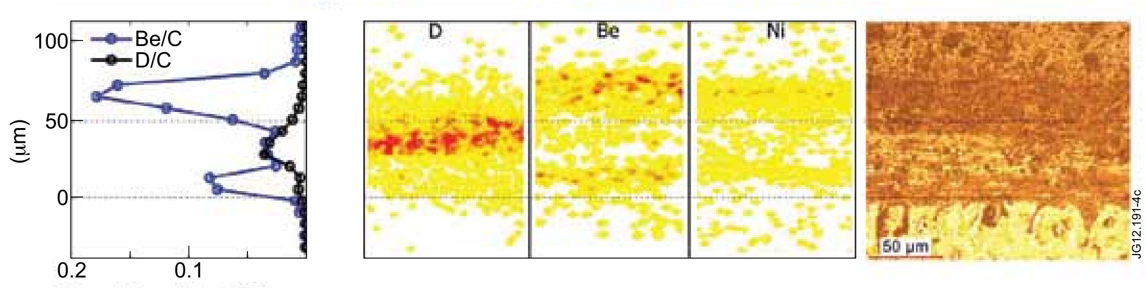

Figure 4. Maps of D, Be and Ni through the cross section of the layer deposited at tile 3, position 2. Exposed 1998-2009. In the optical image from the same region (not exactly the same position) the layer looks lamellar in particular near the interface with CFC, whereas the top layer is internally globular. The bright band at the centre coincides with high D concentration and low Be and Ni.

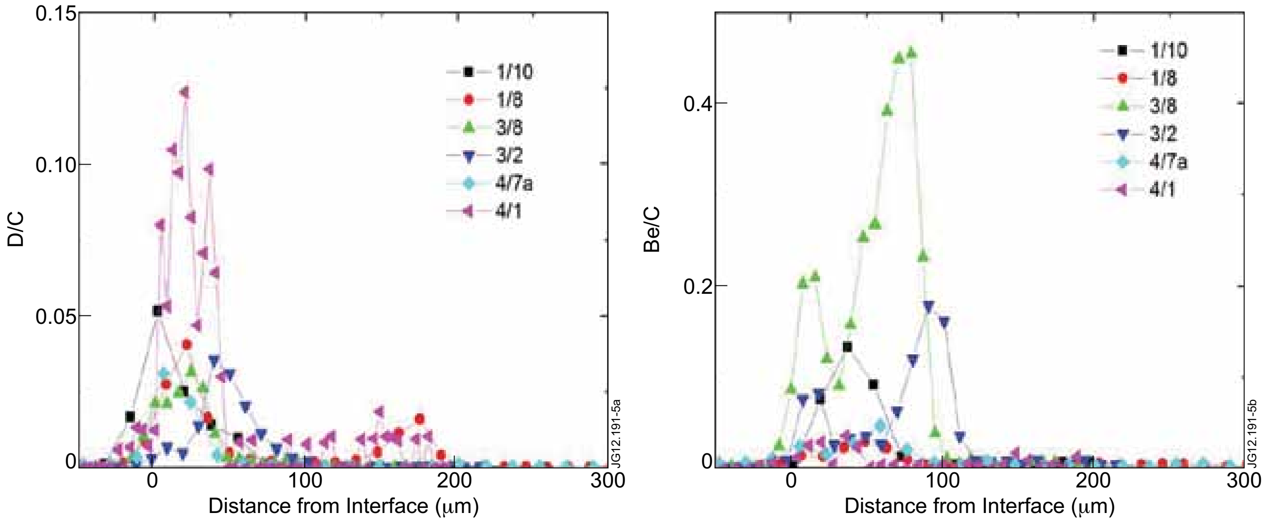

Figure 5. Profiles of D and Be through layers on tiles 1, 3 and 4.

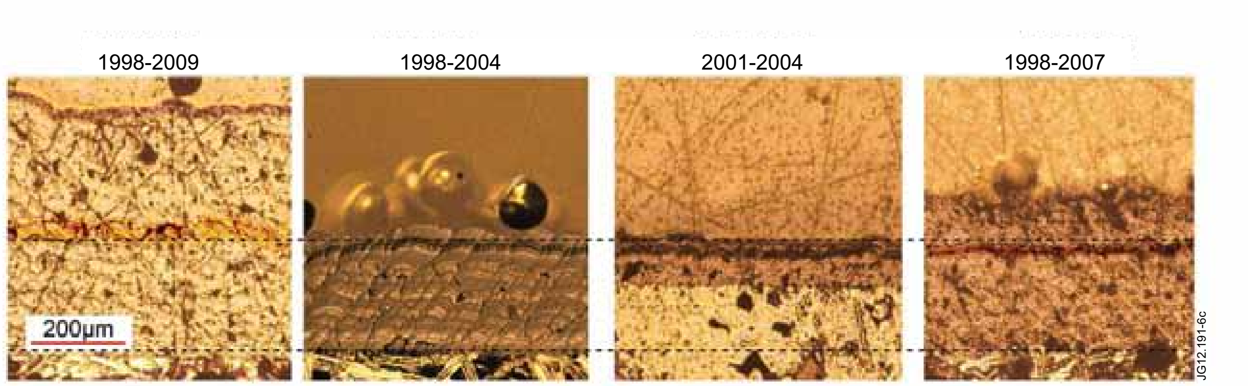

Figure 6. Optical images of layers at tile 4, position 10. The layers were deposited in 1998-2009, 1998-2004, 2001-2004 and 1998-2007 respectively. In all cases there is a thin sub layer, about 20 µm thick, which stands out. Below that line, the layer structure is mostly lamellar. Above the line it is globular and disordered. The line corresponds in time approximately with the shut down in 2004. Layers deposited in 2007-2009 at this position are likewise globular.

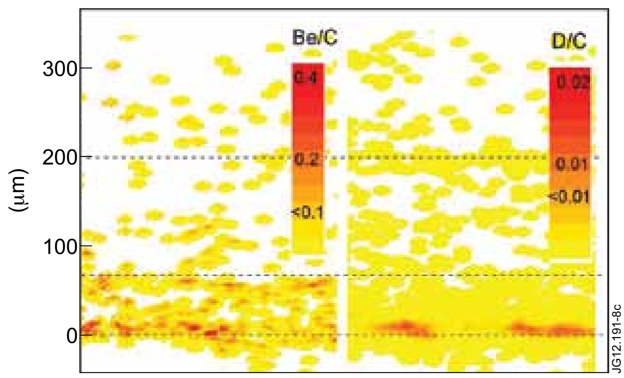

Figure 7. Maps of deuterium at cross sections of layers on tile 4, position 10. The layers were deposited in 1998-2009, 1998-2007 and 2001-2004 respectively. There is a transition between lamellar structure near the interface and globular structure towards the surface. The transition occurs likely in 2004, corresponding to the opticasl images in Figure 6.

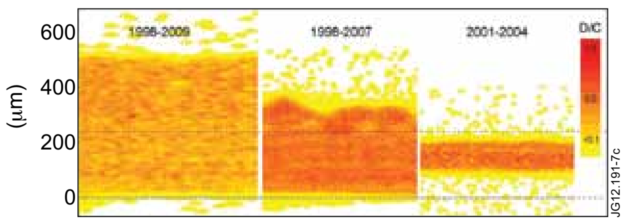

Figure 8. M aps of Be and D for a cross section of the layer at tile 4, position 7a. The layer was formed in 1998-2009. The Be is found in two lamellar layers close to the interface, while the Dis found at the interface and at the surface. The layer extends between 0 and 200µm.