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# Co-deposited Layers in the Divertor Region of JET-ILW

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## **ABSTRACT**

Tungsten-coated carbon tiles from a poloidal cross-section of the divertor and several types of erosion-deposition probes from the shadowed areas in the divertor were studied using heavy ion elastic recoil detection to obtain quantitative and depth-resolved deposition patterns. Deuterium, beryllium, carbon, nitrogen and oxygen along with tungsten and Inconel components are the main species detected in the studied surface region. The top of Tile 1 in the inner divertor is the main deposition area where the greatest amounts of deposited species are measured. Beryllium and tungsten-containing deposits on the probes (test mirrors and quartz microbalance) indicate that both low-Z and high-Z metals are transported to remote areas. Deposition of nitrogen-15 tracer used for edge cooling only at the end of experimental campaigns in 2012 was also detected giving evidence that nitrogen is effectively retained in wall components.

## **1. INTRODUCTION**

The determination of fuel retention and material migration leading to the buildup of co-deposits – especially beryllium erosion and transport – belong to key research areas in operation of the JET tokamak with the ITER-Like Wall (JET-ILW) [1-3]. Spectroscopy [4-6] and gas balance studies [7] together with early surface analysis data [8-11] have shown a reduction of carbon concentration and deuterium retention by a factor of ten or more in comparison to JET with carbon walls (JET-C) [4]. As a positive consequence, also the thickness of co-deposits on plasma-facing components (PFC) has been vastly reduced. The only exception with a relatively thick layer from JET-ILW is the apron of the inner divertor on top of Tile 1 with beryllium-rich films of up to 15 mm [9,11]. For comparison in JET-C flaking layers reaching even hundreds of micrometers were identified [12].

The analysis of PFC from a campaign which started in 2011 with clean tile surfaces has given a unique opportunity of looking for a material migration and deposition history. This also includes tracing of the plasma operation and the history of heating power increase throughout the campaign: starting from limiter plasmas and then followed by the divertor operation with a gradually increased heating. During the latter operation period impurity species (e.g. nitrogen) were seeded to cool the plasma edge [13]. The retention of nitrogen needs to be addressed. The focus of the paper is on the search for a correlation between the operation and the deposition accompanied by material mixing. This has required application of proper analysis methods for quantitative depth profiling of surfaces of representative divertor components.

## **2. EXPERIMENTAL**

The study was carried out for the tungsten-coated carbon fibre composite (W-CFC) divertor plates and for erosion-deposition probes (EDP) retrieved from the JET vessel after the 2011-2012 campaigns which lasted totally for about 18.9h including 13h of X-point operation. Specimens were obtained by coring the inner (Tile 1, 3 and 4) and the outer divertor (Tile 6, 7, 8) plates. In total nineteen samples have been examined. The study of wall probes was done for test mirrors made of polycrystalline molybdenum and for a stainless steel cover of the quartz microbalance (QMB);

details about the structure and location of wall probes are in [14]. It has already been known [8-11] that the thickness of co-deposited layers in most positions does not exceed 1 mm. To study the impurity concentrations in these layers time-of-flight heavy ion elastic recoil detection analysis (ToF-HIERDA) was used. It is based on the surface irradiation with energetic high-Z ions: 36MeV  $^{127}\text{I}^{9+}$  beam 1 mm in diameter that impinges on the sample at the shallow angle of  $22.5^\circ$  and recoils from the sample are then detected at  $45^\circ$  with respect to the direction of the incoming beam. Details of the technique, signal formation, data recording and data analysis have been presented elsewhere [15]. In short, the reversed flight time and the energy gives banana-shaped traces for each recoiled mass from which the depth distribution can be calculated. For data analysis the Contes [16] code is used. The main advantages of ToF-HIERDA are: (a) high sensitivity, (b) good separation of light isotopes up to neon, (c) depth profiling with high resolution for about  $0.5\mu\text{m}$  (i.e. information depth) from the sample surface and (d) quantitative measurements for different elements. However, as both the impinging ion and the recoiled target ion are leaving the target at shallow angles the depth profiles are very sensitive to the sample roughness which consequently deteriorates depth resolution. For samples where the roughness is larger than the analyzed depth it is not possible to determine a quantitative depth profile. Instead the ratio of different elements in the analyzed layer were calculated, which is less sensitive to the roughness. This feature was used in the examination of W-CFC cored samples from the divertor.

### 3. RESULTS AND DISCUSSION

#### 3.1 DIVERTOR

A representative HIERDA spectrum for a divertor target (Tile 4 in this case) is shown in Figure 1. Hydrogen, deuterium, beryllium, carbon, nitrogen and oxygen along with tungsten, steel or Inconel components are the main species detected in most locations. In addition, molybdenum has been found in several locations. It originates both from the Mo-W marker layers on some tiles [14] and the erosion of Inconel 625 parts of the radio frequency antenna. An important feature is a clear trace of nitrogen thus proving that gases for plasma edge cooling are efficiently retained in co-deposits on PFC. This confirms results of works dedicated to the determination of nitrogen retention in TEXTOR [17-21] and ASDEX-Upgrade [20,22].

An overview of the poloidal deposition pattern of Be, C, N and O is shown Figure 2. The divertor cross-section with the so-called S-coordinate is also shown as an insert in Figure 2. The data are given in the form of concentration ratios with respect to beryllium, deposited as a result of erosion from limiters in the main chamber: O/Be, N/Be and C/Be. In this notation low values correspond to points with a high relative beryllium content. This particularly applies to the top part (apron) on Tile 1 where thick Be deposits were formed, as demonstrated in Figure 3, from [11] which presents distribution and absolute Be and D contents along the tile. A clear maximum occurs on the flat apron. Except that Be-rich region, in the majority of other analyzed areas, a strong signal from the tungsten, or molybdenum for Tile 3 that has a special marker coating, substrate could be detected thus indicating that the entire deposited layer was probed. For a uniform layer of Be on top of W,

under the current analysis conditions, a clear tungsten signal should be seen as long as the layer thickness was below  $2 \times 10^{19}$  Be cm<sup>-2</sup>. For most of the points the Be signal that was detected should correspond to less than 10 % of this as the W signal is not strongly affected. For the high field side of Tile 4 the layer is possibly thicker but should be less than 50%. Also on the upper part of Tile 6 the W signal is affected but should be less than 25%. In [23] the total amount of Be was found to be below  $10^{18}$  at./cm<sup>2</sup> on Tile 4 and 6 with higher amounts found locally in valleys formed by the CFC structure, this indicates indeed that the estimates above is an upper limit.

Using the data presented in Fig. 2 and 3 and the estimate above it is possible to estimate how much carbon, oxygen and nitrogen is found in the layer. Figure 4 shows the amounts of detected C, N and O impurities. This is rather a crude estimate but it indicates that the biggest portion of impurity species and Be in the divertor is found on Tile 1. For other divertor regions such calculation procedure may lead to an overestimated amount of Be and, consequently, of other impurities, but the tendency is reflected correctly. This statement is justified by the fact that full tile analysis has shown only small deposition of Be and C on Tiles 3-8 [9,24].

From the histogram in Fig.2 one infers that the atomic concentration of nitrogen is between 3% on top of Tile 1 and 15 % on Tile 3 in the inner divertor. However, when absolute numbers are considered, one perceives that the retention of nitrogen is the largest in the deposit on Tile 1. The same is true for oxygen and carbon. The obtained pattern is a result of the last period of operation with 151 pulses with the same position of strike points. Carbon migrates by erosion and re-deposition and it is transported also to Tile 4 where ratio changes from 0.49 on the sloping part to 0.93 in the corner under the protection plate of Tile 5. It is a result of a multi-step process, as determined earlier in marker experiments with <sup>13</sup>C-labelled methane [12,25-27]. However, the most important point is that the absolute carbon concentrations are small reaching only a level ( $10^{19}$  cm<sup>-2</sup>), even in the thickest layers.

The chemical form of deposited species could not be studied. The formation of BeO by gettering of oxygen impurities can be assumed with high probability. Indeed, oxygen is the most common impurity but in most locations O/Be < 1. Therefore, the presence of other forms of Be in the divertor should be considered. While the formation of a certain amount of stoichiometric compounds, e.g. beryllium nitride (Be<sub>3</sub>N<sub>2</sub>) or carbide (Be<sub>2</sub>C), under tokamak operation cannot be fully eliminated, the dynamic conditions of erosion and deposition suggest rather a mixture of compounds containing also chemically bound nitrogen. The formation of mixed oxides, nitrides, borides and carbides has been detected using X-ray methods on the very surface layer and in thicker films on components from TEXTOR [17,21,24,28] and, also on test mirrors from JET [10].

### **3.2 EROSION – DEPOSITION PROBES**

On the divertor-inserted probes with smooth surfaces (quartz microbalance and test mirrors from the First Mirror Test [10,14]) quantitative depth profiling could be accomplished. Plots in Figure 5 (a) reveal the deposition history on a molybdenum mirror from the outer divertor. The total deposit thickness is around 180 nm. The lack of a sharp mirror-deposit interface could be a result of both

material mixing and variations in the layer thickness, other effects such as the mirror roughness should be smaller. Beryllium is a dominant constituent in the deposited layer and its profile is nearly constant with O/Be, N/Be and C/Be are 0.3; 0.04 and 0.06, respectively. Only at the interface there is relative increase of the oxygen content attributed to: (a) unavoidable presence of the oxide ( $\text{MoO}_2$ ,  $\text{MoO}_3$ ) layer on the original mirror surface and (b) larger amount of oxygen impurities gettered by Be at the very beginning of the JET-ILW operation. Another increase of oxygen content at the very surface corresponds to the uptake of atmospheric oxygen. Carbon also has two regions of the increased content. They correspond to the initial phase of the ILW operation and exposure to air after the mirror retrieval from JET. In general, the C content is very low, on average 20 times less than Be, thus being in a very good agreement with spectroscopy measurements [4]. There is also a clear feature of Inconel and steel components eroded from components such as the main chamber wall. The thickness of the layer where features of nitrogen and tungsten appear is smaller than for the elements described above. It reflects that fact of divertor operation started a few weeks after the initial ILW phase with limiter plasmas. The deposition of W is accompanied by the accumulation of nitrogen in the deposit. It is a strong indication that the nitrogen presence is directly related to the tokamak operation. Similar profiles have been also determined on the mirrors from the inner divertor [10] where discrete structure of tungsten and Inconel deposition profiles could be traced. Depth profiles in Figure 5 (b) have been recorded for a cover of QMB located in the outer divertor. The features of beryllium, carbon and oxygen are as those on the mirror. The amount of Be on the cover is even more pronounced: O/Be, N/Be and C/Be ratios equal to 0.1; 0.03 and 0.01, respectively. The profile of steel components is different than that of Inconel on the mirrors because on the QMB there is deposition of Ni+Cr+Fe from plasma and also mixing of those elements at the cover-deposit interface. Tungsten is accompanied by nitrogen isotopes:  $^{14}\text{N}$  used during the entire divertor operation for a long period and, a rare isotope  $^{15}\text{N}$  (natural abundance 0.37%) used as a marker puffed locally from a single gas inlet module (GIM 14) in the divertor floor between Tiles 6. This was done only during four pulses which were still followed by two-weeks operation period (151 pulses) before the shut-down. The detection of  $^{15}\text{N}$  in the deposits leaves no doubts regarding the origin of nitrogen in all studied deposits. It also shows a great value of HIERDA in studies of plasma-facing materials.

In summary, both on the mirror and the QMB cover the amount of impurities is much lower compared to PFC surfaces of the divertor. It shows only limited transport of species to the shadowed areas. Even in the case of carbon only small quantities are measured on the probes. However, there are some differences between the probes. For instance, the cover has 30 times less deposition when compared to the louvre clip [12] exposed for the same time in the outer divertor:  $5 \times 10^{16} \text{ cm}^{-2}$  on the cover versus  $170 \times 10^{16} \text{ cm}^{-2}$  on the clip, but this amount is nevertheless two orders of magnitude smaller found in flaking layers after the JET-C operation.

## CONCLUDING REMARKS

The quantitative depth profiling of light and heavy elements in various parts of the divertor have



proven low carbon contents on plasma-facing surfaces and in remote areas. The work has also brought two important results to the better understanding and description of material migration processes in JET-ILW: (a) significant residence of nitrogen used for edge cooling and (b) transport of metals to the remote areas. On all surfaces some nitrogen was found although generally in lower concentrations than those of carbon and oxygen. There is no doubt that a fraction of the injected nitrogen is retained. The sticking mechanisms and the identification of a chemical form of nitrogen-containing compounds deserves further studies taking into account that the injected small amount of  $^{15}\text{N}$  was not desorbed or isotopically exchanged despite over 100 plasma pulses before the end of campaign. All types metals (Be, W, Inconel components) eroded from PFC are transported to locations with no plasma line-of-sight. This poses questions regarding their transfer mechanism whether this simple transport of neutrals sputtered from the target plates or chemical transport. While  $\text{BeD}_x$  compounds can be responsible for the beryllium transport [29,30], the chemical transfer of tungsten would require oxides. The presence of W oxides on PFC surfaces has been detected on several occasions [17,25], and a conclusive answer regarding the transport could probably be determined a dedicated marker experiment with the  $^{18}\text{O}_2$  rare isotope.

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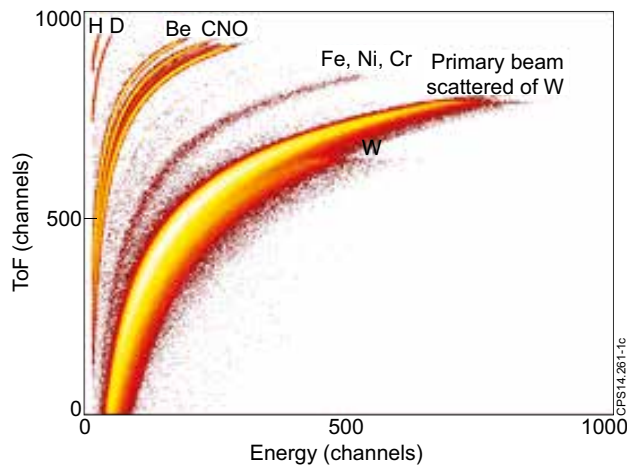


Figure 1: Example of ToF-HIERDA spectrum from tile 4.

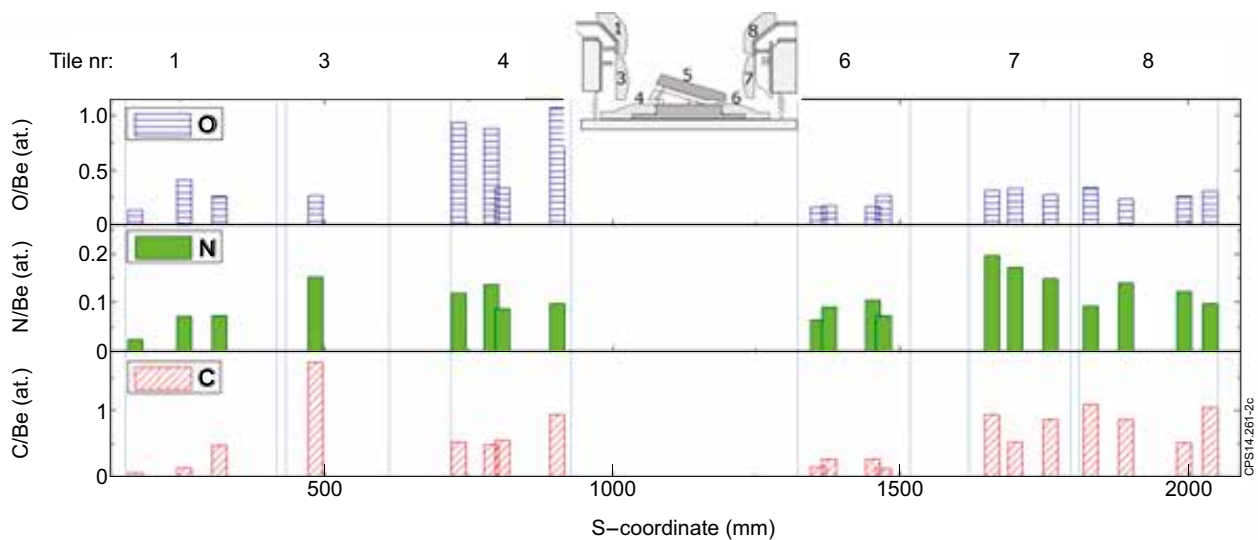


Figure 2: Amount of light impurities related to the concentration of Be are given for different positions in the divertor . On top of the plot the corresponding tile numbers are given. The position of the tiles are given in the insert.

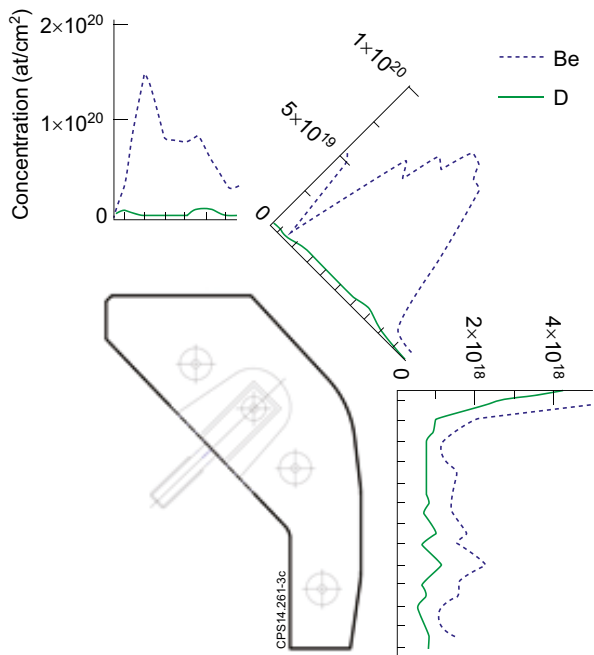


Figure 3: Be and D concentration on Tile 1, taken from [11] as measured by backscattered protons.

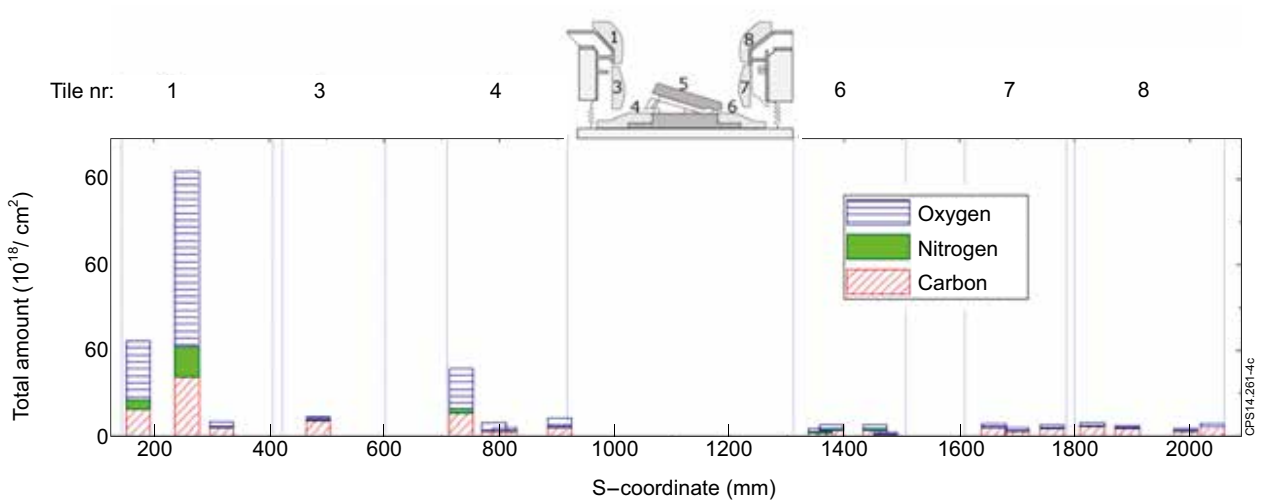


Figure 4: Calculated total amount of impurities, see details in the text for assumptions in the calculation.

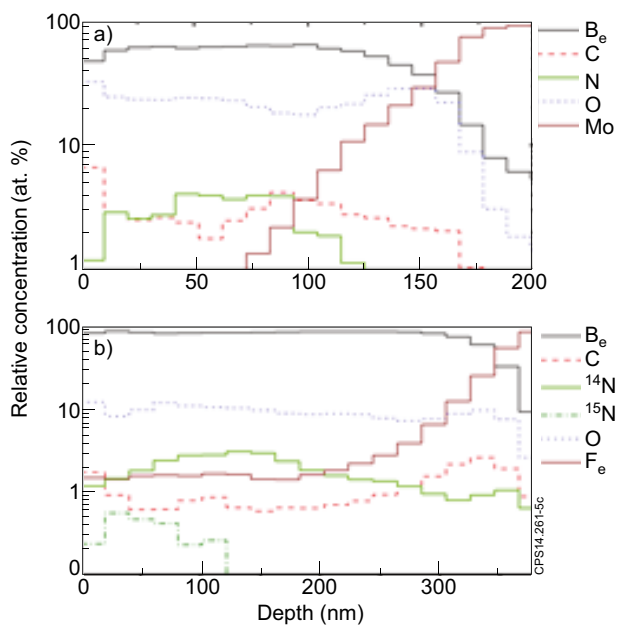


Figure 5: Depth profiles from depositions probes in the outer divertor.