Mapping of Fuel Retention in Carbon Fibre Composites from Tokamaks

P. Petersson 1), M. Rubel 1), G. Possnert 2), A. Kreter 3), T. Dittmar 4), B. Pégourié 4), E. Tsitrone 4)

1) Royal Institute of Technology, Association Euratom – VR, Stockholm, Sweden
2) Ångström Laboratory, Uppsala University, Association Euratom – VR, Uppsala, Sweden
3) Institut für Energieforschung - Plasmaphysik, Forschungszentrum Jülich, Germany
4) CEA, IRFM, F-13108 Saint-Paul-lez-Durance, France

E-mail contact of main author: Per.Petersson@angstrom.uu.se

Abstract. Carbon fibre composite (CFC) exposed to high flux deuterium plasmas in the Tore Supra and TEXTOR tokamaks and in the plasma-wall interaction simulator PISCES-A have been examined by means of micro-nuclear reaction analysis (μ-NRA) with a $^3He^+$ ion beam. The aim was to determine fuel inventory on plasma-facing surfaces and in the bulk of the material. The study revealed significant non-uniformity in the distribution of accumulated deuterium. For the CFC exposed in PISECS-A a clear deposition pattern can be linked to the fibre structure. In the erosion zone of a test limiter exposed in TEXTOR the presence of fuel was detected in small pits acting as local shadowed areas. The study of bulk distribution of deuterium in tiles from Tore Supra showed the presence of fuel to depths exceeding 1 mm beneath the surface with deuterium accumulation in belts located parallel to the surface.

1. Introduction

Plasma-wall interactions (PWI) involve a set of complex phenomena [1-3]. Particles (ions, atoms, electrons) that escape the fusion plasma eventually hit the surrounding surfaces causing modification to the wall by erosion and subsequent re-deposition of the eroded material. Various eroded species and fuel atoms are deposited together, known as co-deposition, forming mixed material layers [3] with serious consequences for the in-vessel fuel inventory. This is especially dangerous in the case of deuterium-tritium operation as it may lead to an unacceptable inventory of radioactive tritium [1,4,5]. Co-deposition is particularly pronounced for carbon which undergoes both physical and chemical erosion thus leading to the formation of carbonaceous layers rich in hydrogen isotopes: C_xI_y, where I denote hydrogen, deuterium and tritium. Carbon, either graphite or carbon fibre composites (CFC), is a wall material in many present-day devices because of excellent power-handling capabilities. The use of CFC is also planned for a strike point zone in the ITER divertor. Therefore, detailed knowledge of fuel retention in CFC is crucial for: (a) the assessment of material erosion and overall fuel inventory in plasma-facing components (PFC) and (b) development of methods for fuel control. These are key factors concerning economy and safety in operation of future controlled fusion devices. The aim of this work was to determine fuel retention and its micro-distribution in CFC used in present-day fusion experiments [6].

2. Experimental

Measurements of deuterium content and distributions were performed for materials facing deuterium plasmas in the TEXTOR (Forschungszentrum Jülich, Germany) and Tore Supra (Cadarache, France) tokamaks and in a PWI simulator: PISCES-A operated at the University of California, San Diego, USA.

Probes made of the NB41, an EU ITER reference composite, were exposed in TEXTOR and PISCES-A. For the exposure in TEXTOR the sample was inserted from the bottom of the torus through a limiter lock system [7]. The sample was mounted on a roof-shaped holder [6] and positioned in the scrape-off layer (SOL) 10-30 mm from plasma edge at a 20° angle. The ions from the plasma were hitting the target for a total exposure time of about 300 s of plasma operation, corresponding to a fluence of 1.1×10^{21} D/cm^2 15 mm deep in the SOL. No active cooling of the holder was applied during the exposure. In PISCES-A the target surface was bombarded at normal angle with the total ion fluence of 3×10^{21} D/cm^2, the surface temperature was kept at 470 K.

In the case of Tore Supra the study has been carried out for several tiles (N11 composite, SNECMA) retrieved from the water-cooled toroidal pump limiter (TPL) after a long experimental campaign when a special DITS (deuterium in Tore Supra) program was realised. Details of the machine operational conditions during DITS can be found in [8]. A section of TPL is shown in Fig. 1 on which the location of studied tiles is marked by circles. The tiles position on the TPL is identified by two numbers denoting the finger (F, toroidal direction) and the individual tile (T, poloidal direction) with different plasma conditions for different positions on the limiter.

Fig. 1. Part of the toriodal pump limiter of Tore Supra. The numbers denoting the tile position and different deposition regions are marked.

Deuterium measurements were done by means of nuclear reaction analysis (NRA) which is based on specific nuclear reactions between the incoming ion and target nuclei. This means that different nuclear reactions have to be used depending on the elements investigated. Most nuclear reactions used for NRA are in fact fusion reactions where two light elements are combined to form new particles together with the release of energy. The analyses were done using a 2.5 MeV $^3He^+$ beam and tracing protons generated in reactions with deuterium and carbon: $d(^3He,p)^4He$, $^{12}C(^3He,p)^{14}N$, respectively. The energy spectrum for the protons of those reactions is shown in Fig. 2. To ensure good resolution and proper counting rate the background at low energies was reduced by a foil that stopped backscattered ^3He ions. The existence of very small samples and the need to have a good spatial resolution across the sample surface makes small beam spots necessary. By focusing the beam and by the use of well defined collimators small beam spots are achieved and used for μ-NRA studies. The beam spot in most measurements was 20x50 μm and the beam current typically 0.25 nA; in some cases the beam was focused to 10x10 μm.

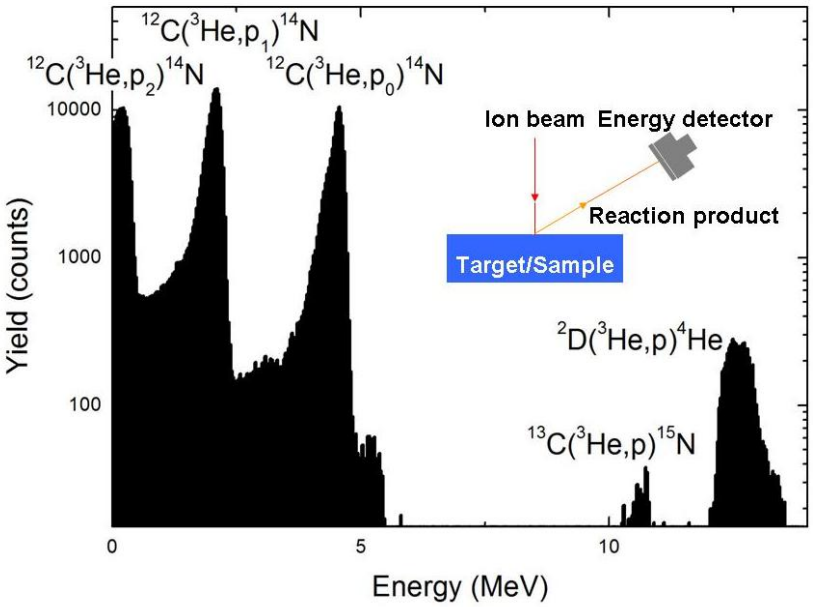

Fig. 2. Energy spectrum of protons used for $^3He^+$ NRA; different distinct peaks are identified.

3. Results

After the exposure in PISCES-A, the fibre structure was still visible by the naked eye but a thin layer formed by the bombardment of deuterium ions was visible as a darkening on top of the material. Fig. 3 shows a map of deuterium content as measured with the microbeam. Two regions with distinctly different D content can be distinguished. They correspond to the two different types of fibres, ex-PAN and ex-pitch. This indicates that surface roughness and porosity play a role in distribution and amount of fuel retention. Broken fibres in the ex-pitch region (perpendicular to the surface) are rougher when compared to the smoother surface in the ex-PAN region (fibres run along the sample surface). The total D content determined with thermal desorption spectrometry (TDS) for a very similar sample was 2.7×10^{17} D/cm^2 [6], i.e. significantly greater than measured with NRA. This suggests that a significant portion of deuterium is stored at a depth greater depth than the 8 µm which is the information depth for deuterium analysis in carbon matrix with a 2.5 MeV ^{3}He beam.

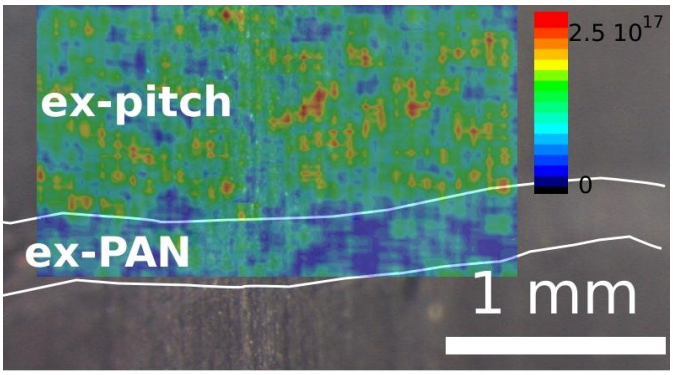

Fig. 3. Deuterium distribution (in at./cm2) recorded with µ-NRA in the 8 µm thick surface layer of the NB41 exposed in PISCES-A. Different retention is observed in ex-pitch and ex-PAN fibres.

Micro-distribution of deuterium and carbon for the same area in the erosion zone of the target exposed in the scrape of layer of TEXTOR is shown in Fig. 4. It is noted that the distributions of

D and C are not uniform. In the complex pattern there are only some spots, up to 100-200 µm wide, with high deuterium content ($6x10^{17}$ cm^{-2}).

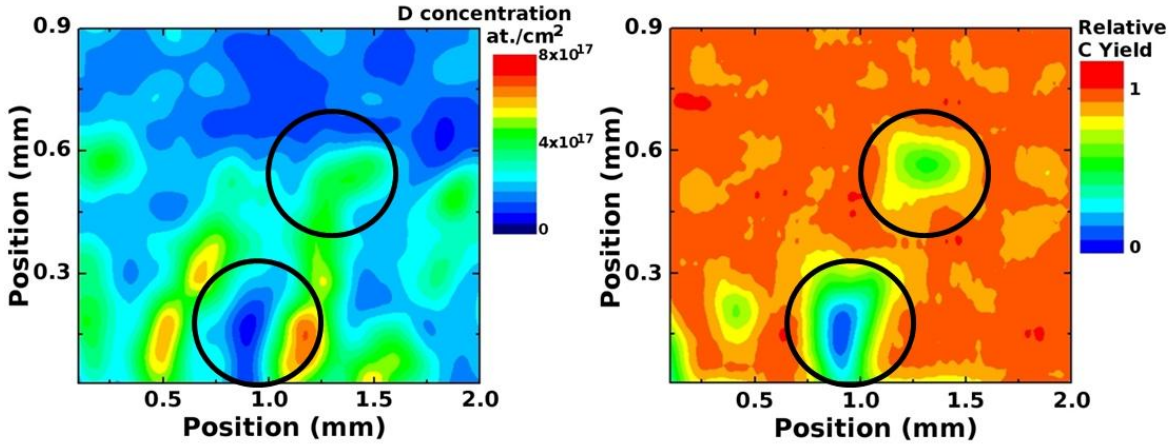

Fig. 4. Deuterium and carbon distribution on the NB41 surface exposed in TEXTOR for ~300 s. The regions with low carbon yields are also marked in the deuterium map.

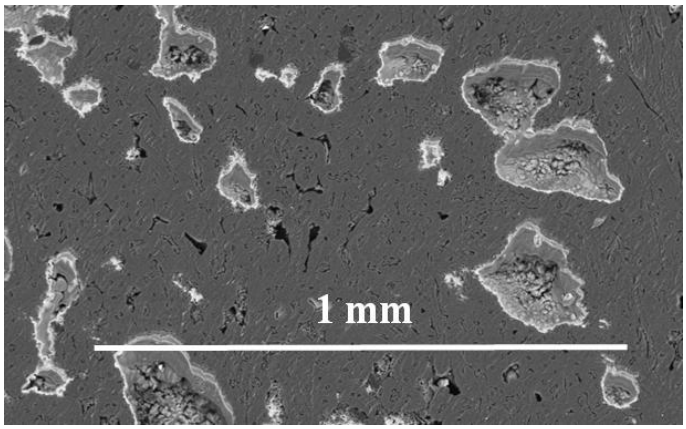

Fig. 5 SEM image NB41 exposed in erosion zone with small pits containing high Z elements

There are also places with small relative carbon content, thus indicating the presence of other species accumulated in those areas. As reported previously, the surface of erosion zone is smoothened by the plasma and heavy impurities are accumulated only in some remaining pits (see Fig. 5) which can be considered as micro-regions shadowed from the direct plasma impact. In such regions metallic plasma impurities can be accumulated, as discussed in [9].

Tiles from the TPL of Tore Supra were analysed a part of the DITS project [8]. Distribution maps recorded with µ-NRA of the D content on the plasma-facing surfaces of tiles of the TPL are shown in Fig. 6. To give a better comparison of different tiles, the deuterium signal has been normalised with the intensity of protons from the p_0 and p_1 reaction channels of carbon.

The differences in the D content over the micro-region under examination reach over 10 for tiles with net deposition (see Fig. 6a-c). On the contrary, in the erosion zone, the content is lower and fairly uniform. However, in some spots higher concentration is found (Fig. 6d). This may be attributed to the retention in small cavities or pits which act as tiny shadowed areas protecting

implanted or co-deposited species from the direct plasma impact [9]. The image of micro-areas with different properties on tiles with net deposition can also be seen in Fig. 7a and is in contrast to the situation in Fig. 7b with a more homogenous surface and small pits from the erosion zone.

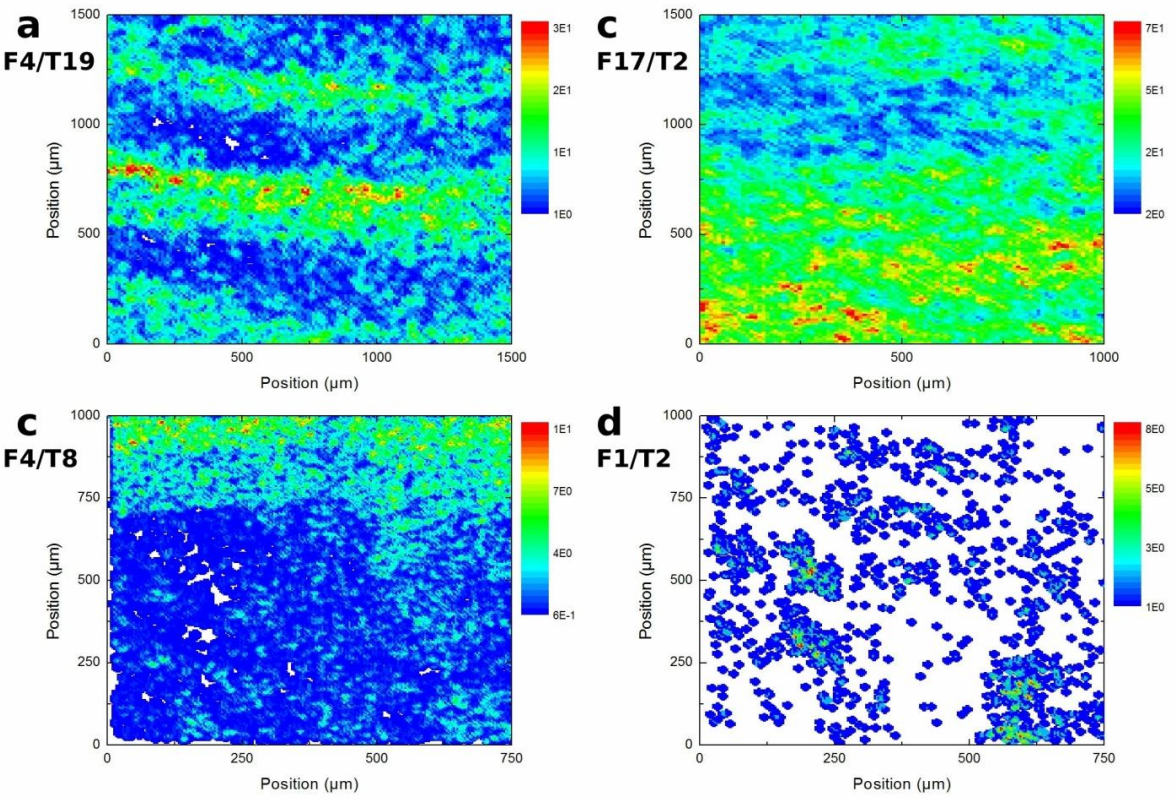

Fig. 6. Map of the relative surface deuterium to carbon yield for a µ-NRA measurement for tiles from the TPL of Tore Supra.

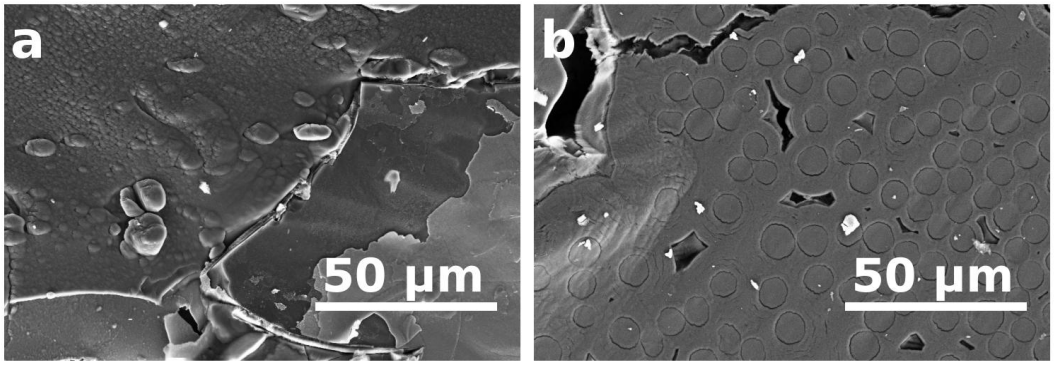

Fig. 7. SEM images of (a) tile F4/T8 with net deposition and (b) tile F1/T2 with net erosion

The deuterium content further into the sample was investigated by micro-beam analyses of the cross section and can be seen in Fig. 8. The plasma-facing surface was coated with a hard paint (as explained in [10,11]) to block the surface deuterium and prevent contamination and then

cracked with the help of a sharp edge. In the graph the position of the plasma-facing surface is located at 0 µm, which is outside of the plot except for d where it is marked with a dashed line.

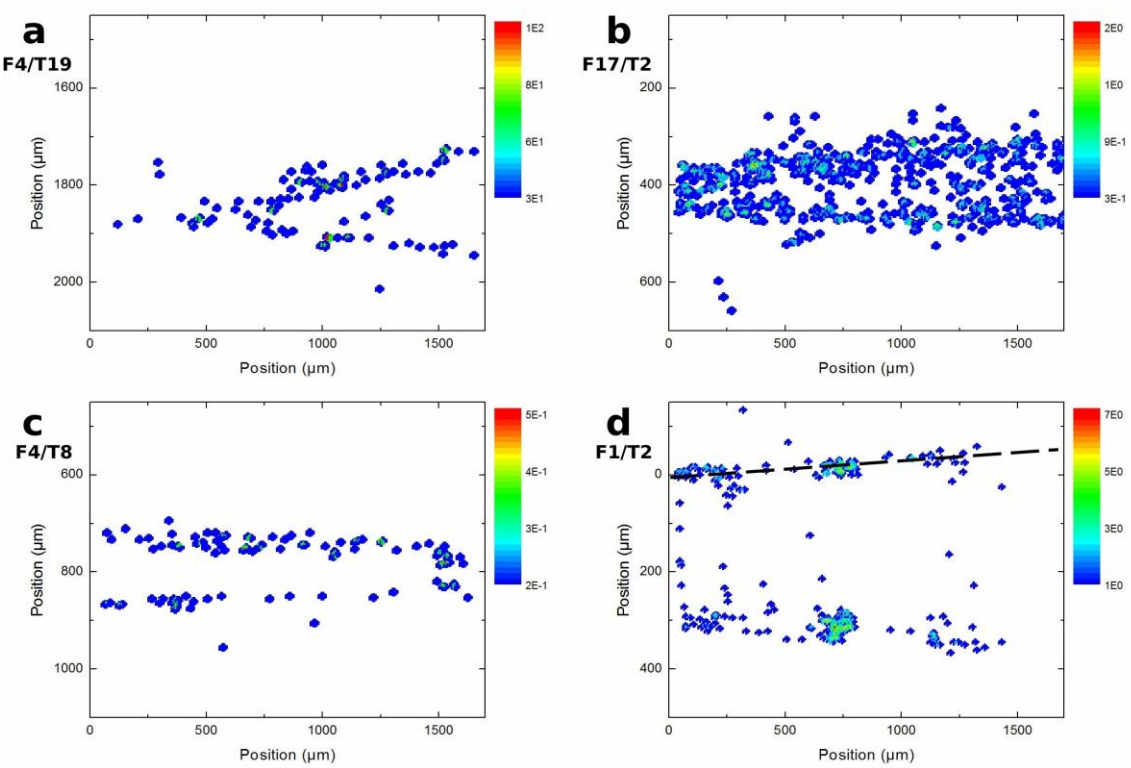

Fig. 8. Deuterium distribution in the bulk, measured on cross-sections on the TPL tiles from Tore Supra.

There is one characteristic feature seen in for all of the cross sections: deuterium is detected in bands approximately 100 µm wide located roughly parallel to the plasma-facing surface. The belts were found at depths of more than 1 mm. These "well structured" distributions seem to be independent on the tile location of the TPL and the systematic observations indicate a relation between the internal material structure and fuel storage in the bulk.

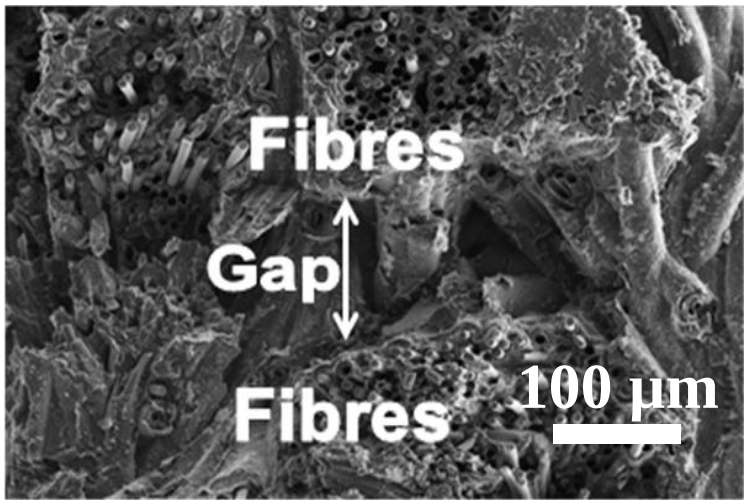

Fig. 9. SEM of the cross-section obtained by cleaving Tile F4/T19 from the region with thin deposits on the TPL.

N11 CFC has very high porosity and low magnification SEM microscopy image as seen in Fig. 9 proves the existence of gaps separating the bundles of fibres. The width of the gap and the bundles is similar, around 100 - 150 µm. It is difficult to say with a high degree of confidence whether the deuterium resides in

the bundles (perpendicular to the image plane) or on side surfaces of the fibres which are parallel to the image plane.

4. Concluding remarks

Comprehensive studies of deuterium retention has been performed with μ-NRA for probes and PFC exposed to deuterium plasmas in the TEXTOR and Tore Supra tokamaks and in a PWI simulator. The major contribution of this work to fuel inventory studies is the detection of significant inhomogeneity on a micro-scale of deuterium distribution both in the surface layers and in the bulk of PFC made of carbon fibre composites. For the first time a focused ion beam has been used for studies of bulk concentration on cross-sections obtained by cleaving. In the bulk of limiter components from Tore Supra distribution of fuel in narrow bands (100-150 μm) located parallel to the tile surface was determined. On CFC surfaces from the erosion zone in Tore Supra and in TEXTOR micro-regions rich in deuterium were detected. This allows for a conclusion that in the erosion zones fuel is stored in small pits and cavities, i.e. in regions shadowed from the direct plasma impact. In summary, the use of focused beams has contributed to a better understanding of fuel deposition and migration into the bulk of PFC.

Acknowledgements

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