Journal of Nuclear Materials 415 (2011) S765–S768

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

# Structural changes and distribution of accumulated tritium in the carbon based JET tiles

E. Pajuste [a,*], G. Kizane [a], J.P. Coad [b], A. Vitins [a], A. Kirillova [a], M. Halitovs [a], JET-EFDA Contributors [1]

[a] EURATOM/UL, Institute of Chemical Physics, University of Latvia, 4 blvd. Kronvalda, LV-1586, Latvia
[b] EURATOM/CCFE Fusion Association, Culham Science Centre, Abingdon OX14 3DB, UK

ARTICLE INFO

*Article history:*
Available online 30 October 2010

ABSTRACT

In this study the tritium distribution and the effect of structural changes thereon have been analyzed in the bulk of the tile selected from the JET Mark II SRP divertor. Tritium content has been analyzed by the full combustion technique [1]. The structure has been investigated by the method of Scanning Electron Microscopy.

Tritium depth profiles have been measured at different poloidal positions. A high specific activity of tritium (up to 156 MBq $g^{-1}$) was found at the plasma-facing surface. At some tile positions up to 98–99% of the T can be in the surface slice of 1 mm thickness, whereas in other poloidal positions there can be more T in the bulk than at the surface. The structural changes of the tile material both at the surfaces and in the bulk have been measured.



## 1. Introduction

Carbon fibre composites (CFC) are a candidate material for divertor areas near the plasma strike points in future fusion devices. This choice is based on the ability of carbon to handle off–normal heat loads without melting and its low Z. However, high physical and chemical erosion yields leads to a limit to the lifetime of the plasma-facing component and build up of tritium inventory is an important safety issue [2].

As a result of erosion large amounts of carbon are introduced into the plasma edge and then re-deposited at different locations of the chamber together with the hydrogen isotopes. This co-deposition is considered the dominant retention mechanism in fusion devices, nevertheless also the implantation of energetic ions in the near surface layers and diffusion into the bulk can make significant contributions to the fuel immobilization. These mechanisms depend not only on the plasma operation conditions, but also on the structure of the materials, in this case particularly of the woven fibre sheets constituting the CFC [3].

In the literature [4] tritium distribution on the surface of divertor tiles of JET has been described, but there is a lack of information about the tritium depth profiles. Therefore it is important to assess the tritium amount accumulated in the bulk of the tiles and also the conditions that have an effect on that. In this study both tritium depth profiles and structural changes of the fibres in selected CFC tile from the JET divertor have been investigated.

## 2. Experimental

The analysed tile was from the MkII Septum Replacement Plate (MkII SRP) divertor used in the 2001–2004 operation period of JET: a cross-sectional view is shown in Fig. 1.

The analysed tile is made of Carbon Fibre Composite, Concept I manufactured by Dunlop Ltd. (CFC) [6]. It is manufactured from fibre reinforced graphite by chemical vapour deposition (graphitization) with methane and it has a 2D woven fibre sheets settlement. Tile has been placed in plasma chamber with the weave planes normal to the plasma-facing surface [3]. The divertor tiles are bolted to carriers connected to a water cooled structure, so the backs of the tiles are indirectly cooled following plasma heating of the front surfaces: bulk temperature between pulses is ~100 °C.

Since the greatest tritium accumulation occurs on the inner divertor shadowed areas [4], tile 4 (14BWG4B) has been selected for this investigation (Fig. 1). During the operation period no full D–T mixture discharges were made, however in the year 2003 the Trace Tritium Experiment had been performed with an introduction of 380 mg tritium into the vacuum chamber. Tritium was introduced by the methods of gas puffing and neutral beam injection, therefore, both the thermalised T+ and fast T+ (~100 kV) were present in the plasma chamber. During the exploitation period tritium ions with energy up to 1 MeV had also been produced as a result of D + D reactions [5].

* Corresponding author. at: EURATOM/UL, Institute of Chemical Physics, University of Latvia, 4 blvd. Kronvalda, LV-1586, Latvia.
*E-mail address:* elina.pajuste@lu.lv (E. Pajuste).

[1] See Appendix of F Romanelli et al., Fusion Energy 2008 (Proc. 22nd Int. Conf. Geneva) IAEA, (2008).

0022-3115/$ - see front matter © 2010 EURATOM. Published by Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2010.09.045

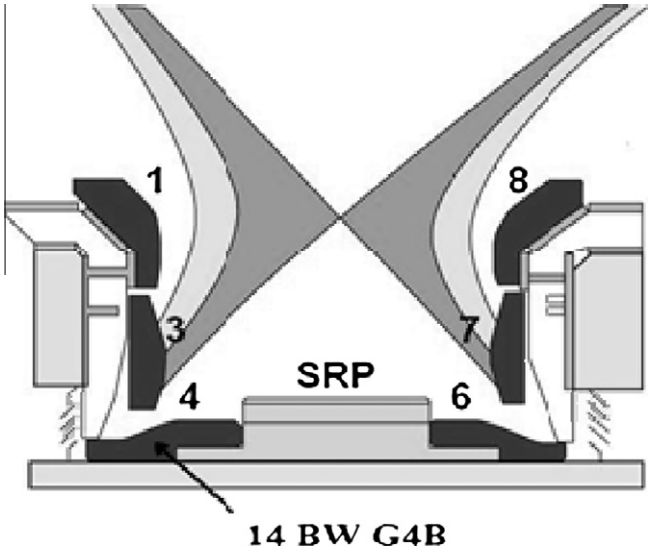


**Fig. 1.** Cross-section of the JET MkII Septum Replacement Plate divertor (SRP).

Plasma interaction was essentially limited to the sloping part of tile four, since the rest of the tile was shadowed. During the SRP campaign the inner strike point was on tile four for a total of $\sim 4 \times 10^4$ s, and, assuming a similar distribution of ion fluxes per second for pulses with strike points on tile three (which can be measured by Langmuir probes), the integrated ion flux for the campaign would be of the order $10^{27}$ ions $m^{-2}$ [7].

Samples for the analysis of tritium and structure were made by a core-drilling method. Cylinders (∅ 1 cm and 1.5 cm) were cut from the CFC tiles normal to the plasma exposed surface (and thus parallel to the tile planes, including a number of such planes) and sliced into separate slices of thickness 1 mm. From tile 14BWG4B 66 cylinders were core-drilled (11 rows in poloidal and six rows in toroidal directions) in order to give a comprehensive picture of the tile (see below) and each sliced into 11–23 slices depending on the thickness of the tile at the particular position. In Fig. 2 the labelling and positions of the first row cylinders drilled out of the tile is shown. Because of the differences in exposure conditions, the tile was divided virtually in three parts (Figs. 1 and 2):

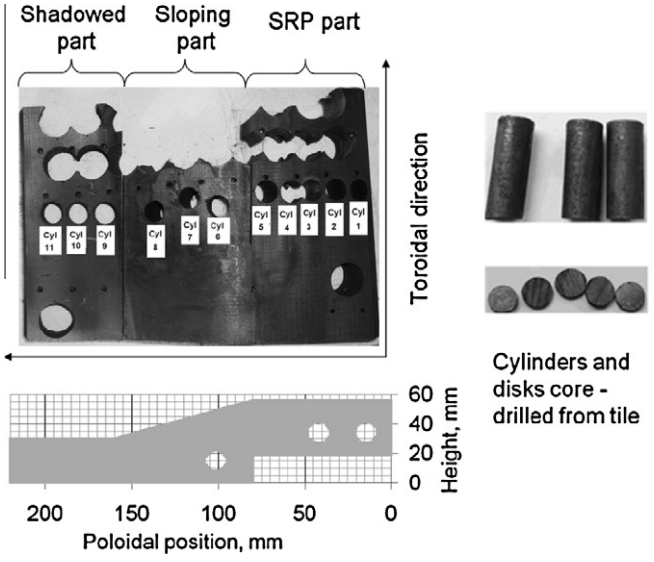


**Fig. 2.** Preparation and labelling of the samples.

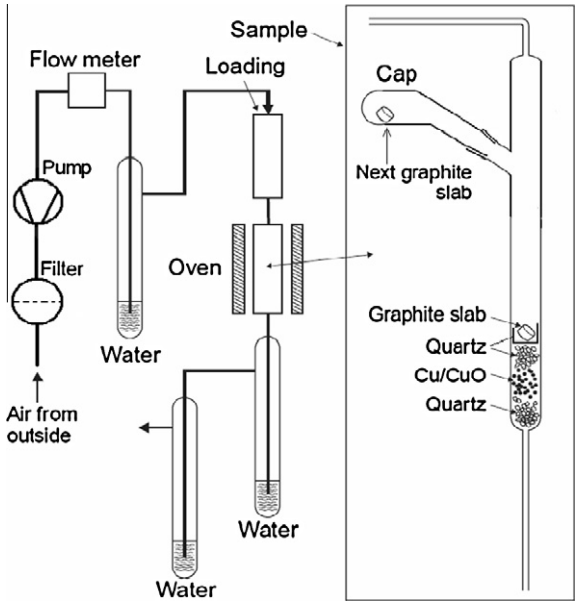


**Fig. 3.** Scheme of a modified Vance apparatus for the combustion of carbon samples.

(1) "SRP part" – horizontal part of tile nearest the SRP that has some exposure to plasma and some shadowing from SRP tile. Deposition layer about 10 μm or less [6].
(2) "Sloping part" – sloping part of the tile that has been exposed to plasma and is the furthest part into the corner of the divertor that can be reached by the plasma. Deposition layer can reach thickness up to 300 μm, dusty in nature and of low density[6].
(3) "Shadowed part" – horizontal part of tile shadowed from the plasma by tile 3, deposition layer up to 250 μm in form of a smooth, dense film with high H/C ratio [7].

In order to determine the tritium content in the separate carbon discs, the combustion technique proposed by Vance et al. [8] and employed in study JET-P(99)53 [1] was used. A scheme of the glass experimental apparatus used is shown in Fig. 3. The combustion was normally performed at a temperature of 850–870 °C in a flow rate of moistened air of 15–20 mL/min. The tritium was collected in two bubblers (volumes of distilled water 100–200 mL and 100 mL respectively); 98–99% of tritium was collected in the first bubbler. The time for complete combustion, 4–6 h, depends on the thickness of the sample being combusted. After each combustion 5 mL water aliquots were taken from each of the two bubblers. Each of these aliquots was mixed with 15 mL of *Ultima Gold* scintillation cocktail and analyzed for total tritium with a TRi-Carb 2910TR counter [PerkinElmer, Inc.].

Analysis of the structure has been done by the methods of Scanning Electron microscopy (Hitachi S-4800, 2 kV, 15μA), SEM images analyzed by the *ImageJ* program [Developed by W. Rasband, National Institute of Health]. The structure was analyzed by the assessment of the changes in separate fibre diameters.

## 3. Results

### 3.1. Tritium depth profile

The depth profiles of tritium trapped in the CFC tile were determined. Tritium mass activity of the plasma surface layers (within the depth of 1 mm), the bulk and the backside layers have been compared in the cylinders from different parts of the tile. Data

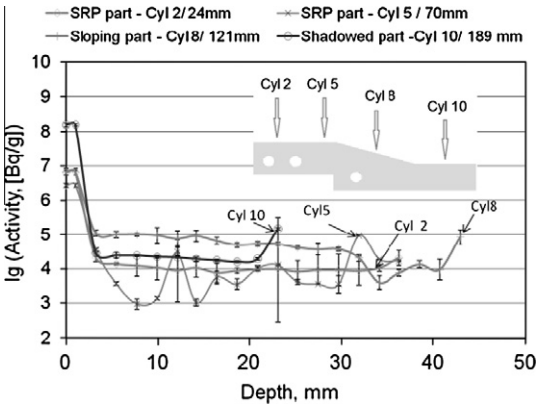


**Fig. 4.** Tritium depth profiles in selected cylinders representing *SRP*, *Sloping* and *Shadowed* parts of the tile (in logarithmic scale).

points of the tritium mass activity of a typical cylinders cut from each of the three areas of the tile 14BWG4B are shown in logarithmic scale in Fig. 4, the *x*-axis shows the depth position in millimetres and the last point of each figure represents the backside surface (cylinders from sloping part were also cut perpendicular to the plasma-facing surface).

Typical values of average tritium mass activities (and T/C mass ratios) of the plasma-facing surface layer, bulk and the backside layer are given in Table 1.

A common feature for all the cylinders investigated is that a large fraction of the tritium is localized in the plasma-facing surface slices of 1 mm. This is due to the co-deposition of tritium in the thick deposition layers that are present on the tile four and also the implantation of the high energy tritons coming from the plasma could have some contribution. There is then a sharp decrease by 2–4 orders of magnitude to a bulk tritium activity which is at a more or less uniform level until it reaches the backside layers where the activity increases again by about one order of magnitude. The phenomenon of increasing tritium content in the backside layer has been observed also by other authors [3] and no explanation has yet been given.

The highest surface activities of tritium were found in the shadowed part of the tile. For instance, the mass activity of the surface slice of Cyl10 was 0.156 GBq $g^{-1}$, while in Cyl2 representing the SRP part it was only 0.006 GBq $g^{-1}$. Tritium activity in the surface layer can be described as a variable proportional to the thickness of deposition layer and inversely proportional to the energy deposited on a tile since tritium accumulated in the co-deposition layer and the hydrocarbon species would be released at a high temperature [9]. The shadowed area has both a thick deposition layer and no contact with plasma, in contrast to other parts of the tile. The sloping part of the tile has the thickest deposition layer (up to 300 μm), but at the same time it has been subjected to high temperature due to the plasma striking the surface and ELMs. Therefore tritium surface activity has similar values to that in the SRP part where the deposition layer is only 10 μm or less.

Migration of tritium into the bulk of a tile may be expected at high temperatures [8]. The surface of the sloping part of the tile may reach 1000 °C during plasma exposure: this is clearly reflected in the results from the sloping part in Fig. 4 where the tritium mass activity of the bulk was found to be about an order of magnitude higher than in other parts of the tile.

**Table 1**
Tritium mass activities in the different poloidal positions on the tile.

| Part of the tile | Cylinder/ poloidal position (mm) | Average surface activity, kBq $g^{-1}$ (T/C mass ratio, ppm) | Average bulk activity, kBq $g^{-1}$ (T/C mass ratio, ppm) | Average backside layer activity, kBq $g^{-1}$ (T/C mass ratio, ppm) |
|---|---|---|---|---|
| SRP part | Cyl2/24 | 6344 ± 64 (0.01767) | 10 ± 2 (0.00003) | 20 ± 1 (0.00006) |
| | Cyl5/70 | 2700 ± 40 (0.00755) | 15 ± 13 (0.00004) | 16 ± 2 (0.00005) |
| Sloping part | Cyl8/121 | 6640 ± 51 (0.01789) | 65 ± 32 (0.00016) | 84 ± 4 (0.00024) |
| | Cyl9a/134 | 7610 ± 57 (0.02120) | 46 ± 22 (0.00013) | 9 ± 2 (0.00003) |
| Shadowed part | Cyl10/189 | 158000 ± 1000 (0.44265) | 34 ± 3 (0.00010) | 153 ± 1 (0.00043) |
| | Cyl11/210 | 48400 ± 84 (0.13531) | 20 ± 2 (0.00006) | – |

### 3.2. Structure

Several thousand SEM pictures have been taken in order to analyse structure of the carbon fibre material in different positions-both close to the surface and in the bulk of the tile. The fibres deep in the bulk of the tile were assumed not to have any modification and were used as a reference. The structure of the separate individual fibres corresponds to the core–sheath type of fibre [10] and has a diameter of (31.1 ± 0.1) μm (Fig. 5).

The deposition layer on the surface of the tiles has been described elsewhere widely [11]. On the erosion dominated areas of the plasma-facing surface (some areas in the SRP part) the bending or complete destruction of the fibres was observed.

To estimate the changes of the material structure the carbon fibre modifications were analyzed. An increase of the fibre diameter close to the backside surface was observed to be up to 30% (in the sloping part) (Fig. 6). There was also a small increase near the plasma-facing surface of the SRP part.

Increase of fibre diameter can be caused by neutron irradiation as a result of formation of extra graphite planes [12] or interplanar voids [13]. However, the neutron flux in the JET is not sufficient to cause such changes ($3.60 \times 10^{14}$ n $cm^{-2}$ for the 2001–2004 operation period [14]) and neutron damage would be uniform through the tile: the largest modification was observed at the backside of the tile. This might mean that fibre modifications have been caused by some other reason, e.g. mechanical forces due to the method of mounting the tile in the divertor. The tile is mounted by pulling down on a central bolt from the backside with the tile supported at the corners, so that the backside is in tension. Therefore, there are no extra graphite planes but existing planes have separated as a result of the deformation, and extra space for tritium to migrate has appeared. Another process that might have an effect on

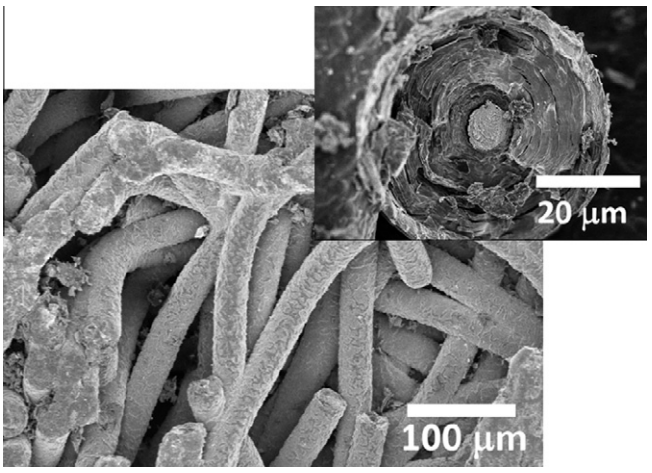


**Fig. 5.** SEM images of carbon fibres in the bulk of JET divertor tile. The inset shows an expansion of a fibre cross-section.

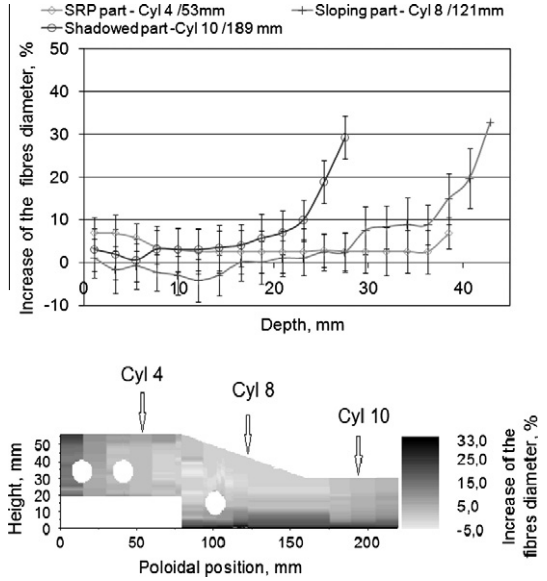


**Fig. 6.** Increase of the fibre diameter in the tile 14BWG4B: a plot showing the increase of the fibre diameter with depth through the tile and distribution of the fibre swelling within the tile.

the structure is CFC interactions with oxygen during the storage in air. Whereas the plasma-facing surface could be protected from an action of atmospheric oxygen by the deposition layer, it might explain small changes of the fibres in the SRP part where the deposition layer is relatively thin. Though, these interactions are at room temperature so would be expected to have a small effect. Increase of specific surface area and concentration of trapping sites resulting from destruction of fibres that follows from the 30% radial growth could lead to increased tritium retention [15].

## 4. Discussion

Tritium depth profiles show an increase of the tritium concentration in the backside layers of the tile. In contrast to the plasma surface the co-deposition of tritium and energetic triton implantation is not possible. One of the explanations could be migration of gaseous tritium and tritiated hydrocarbons around the divertor, reaching the backsides of the tiles and adsorption. However the gas pressure in the plasma chamber is extremely low for this mechanism to take place.

The structure analysis of the tiles showed that the largest deformation and contingent destruction of the carbon fibres has occurred in the backside layers of the tile: this may be a result of mechanical forces.

Since CFC cannot be described as a homogenous material, differences of tritium migration and trapping in the fibre matrix, fibre core and sheath must be considered [16]. Therefore, changes of the volume ratio between these components as a result of deformation or/and destruction may have an effect on the tritium penetration and retention process in the bulk. Increase of the space between the sheath graphite planes facilitates the transport of tritium between the planes, while full destruction of fibre sheath leads to the increase of specific surface area and concentration of high energy trapping sites. It has also been demonstrated by other authors that hydrogen isotope retention increases considerably in the damaged structures of carbon based materials [9,17].

It is possible that an increase of the trapping site concentration because of the fracture deformations might be the reason for the higher tritium retention in the backside layer of the shadowed part of the tile since there the largest structure changes were observed.

## 5. Conclusions

Tritium depth profile in a CFC tile from the JET MkII SRP divertor has been obtained by the full combustion technique and SEM analysis of the structural changes of the fibres has been made.

(1) Depth profile of the tritium shows high tritium concentration in the plasma-facing surface layer that is followed by a sharp decrease to a bulk tritium activity which then stays at a uniform level until it reaches the backside layer where it increases again.
(2) Increase of the fibre diameter followed by a full destruction of the fibres sheath structures were observed in the backside layer. It is possible that the changes occur as a result of long term exposure to air, though such processes are very slow at room temperature (however, T and D are steadily released due to interaction with water vapour at room temperature [18]). It is more likely that the changes in fibre structure can be explained by mechanical forces applied on the tile during exploitation in the plasma chamber.
(3) The increase of the specific surface area and concentration of the hydrogen traps in the CFC material as a result of deformations and destruction of the fibres might be one of the reasons for increased tritium retention in the backside layers of the tile.

## Acknowledgements

This work was performed within the EFDA task JW8-FT-1.12. The support of European Social Fund ESF and L'Oréal Latvian Fellowship for Women in Science 2010 with the support of the Latvian National Commission for UNESCO and the Latvian Academy of Sciences is also gratefully acknowledged.

## References

[1] R.-D. Penzhorn et al., J. Nucl. Mater. 279 (2–3) (2000) 139–152.
[2] Tanabe et al., Fus. Eng. Des. 81 (2006) 139–147.
[3] N. Bekris et al., J. Nucl. Mater. 313–316 (2003) 501–506.
[4] T. Tanabe et al., J. Nucl. Mater. 363–365 (2007) 960–965.
[5] T.T.C. Jones et al., Fusion Sci. Technol. 48 (1) (2005) 250–257.
[6] J.P. Coad et al., Nucl. Fusion 46 (2006) 350–366.
[7] A. Widdowson et al., J. Nucl. Mater. 363–365 (2007) 341–345.
[8] D.E. Vance et al., Report LA-7716 UC-4, University of California, Los Alamos Scientific Laboratory, Los Alamos, New Mexico, USA, 1979, p. 5.
[9] Atsumi et al., Phys. Scr. (2003).
[10] G.M. Pennock et al., Carbon 31 (4) (1993) 591–609.
[11] J.P. Coad et al., J. Nucl. Mater. 363–365 (2007) 287–293.
[12] Timothy D. Burchell, Phys. Scr. T64 (1996) 17–25.
[13] T. Munsat, Technical report, Princeton Plasma Phys. Lab., Princeton, USA, 1999, p. 30.
[14] Personal communication with Dr. Sergey Popovichev, EFDA JET Team.
[15] R.D. Kolasinski et al., Fusion Eng. Des. 84 (2009) 1068–1071.
[16] Luis A. Sedano et al., J. Nucl. Mater. 273 (1999) 285–293.
[17] V.N. Chernikov et al., J. Nucl. Mater. 264 (1999) 180–197.
[18] J.P. Coad et al., J. Nucl. Mater. 160 (1988) 95–97.