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Tritium depth profiles in 2D and 4D CFC tiles from JET and TFTR

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Abstract

Carbon fibre composite (CFC) is currently the candidate material for the vertical target tiles in the International Thermonuclear Experimental Reactor (ITER) divertor because of its superior thermomechanical properties. However, its affinity for hydrogen isotopes and their co-deposition with eroded carbon may severely limit ITER plasma operations. Recently tritium depth profiles in divertor tiles retrieved from the Joint European Torus have been obtained by the coring/full combustion technique. The results revealed that a surprisingly large fraction (up to 61%) of the retained tritium had diffused deep into the bulk of the tile, most probably between the woven sheets of the CFC. Additionally, the coring/full combustion technique has shown that only the surface tritium (few ten μm) is efficiently released by air baking while the bulk tritium is almost not affected. Baking the tile under air even at 500 °C does not detritiate the bulk.

Keywords: 2D/4D; CFC; Detritiation; JET; TFTR; Tritium profiles

1. Introduction

To reduce the detrimental effects of impurities in the plasma, low Z materials, such as beryllium and carbon, were chosen to constitute the first wall materials (FWM) of International Thermonuclear Experimental Reactor (ITER). Because of its ability to withstand high heat flux and its favourable thermomechanical properties (carbon sublimes rather than melts during disruptions or edge localised modes) carbon tiles have been widely used for FWM at Joint European Torus (JET), UK, the Tokamak Fusion Test Reactor (TFTR), Princeton Plasma Physics Laboratory (PPPL), NJ, USA, and currently are the candidate material for the vertical targets of the divertor plates of ITER.

Carbon fibre composite (CFC) tiles used at JET were manufactured from fibre reinforced graphite by chemical vapour deposition (graphitisation) with methane in a furnace at high temperatures, e.g. 2000 °C [1], and had a 2D woven fibre sheets settlement. As the thermal conductivity is high in the plane of the woven fibre sheets and low perpendicular to the woven sheets, CFC is placed in JET with the weave planes normal to the plasma facing surface to maximise thermal conductivity (Fig. 1).

On the other hand, at TFTR carbon tiles with a multidirectional fibres weave were used. The tiles manufactured by FMI (Fiber Materials Inc.) had a 4D woven structure.

During plasma discharges hydrogen isotopes are implanted into the graphite tiles as ions or as energetic charge exchange neutral atoms which interact not only

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Fig. 1. 4D pyramidal configuration of a CFC tile (10× magn.).

with the outer CFC surfaces but also deeper in the tile on the surfaces of interconnected pores. As a result, the carbon atoms are chemically or physically sputtered and redeposit together with hydrogen isotopes in the plasma as a polymer-like hydrogenated carbon film (C:H film), formed mainly in the cooler and shadowed areas of the FW not accessible by the plasma. On the other hand, on plasma exposed surfaces hydrogen isotopes impact the tile surface generating a hydrogen saturated carbon layer. Hydrogen isotopes also penetrate deeper in the bulk through the interconnected pores or following the woven fibre sheets. Retention via co-deposition and implantation of energetic ions in the near surface layers as well as bulk effects, including migration through the network of interconnected pores and diffusion across the grains, are well known mechanisms for plasma material interaction during tokamak operations [2]. As this paper shows these mechanisms (especially diffusion) dependent not only on the plasma operation conditions but also on the type or the settlement of the woven fibre sheets constituting the CFC.

Tritium depth profiles in divertor tiles retrieved from JET have been obtained recently by the coring/full combustion technique [3]. The results revealed that a surprisingly large fraction (up to 61.6%) of the retained tritium had diffused deep into the bulk of the tile, most probably between the CFC weaves [4]. As the tritium inventory in FWM of fusion machines constitutes an important safety issue, accurate determination of tritium in these materials is therefore of high importance. Recent estimations for ITER gave an average co-deposition rate of the order of 2–5 g tritium per shot, reaching the 350 g safety limit after 70–170 shots [5].

Moreover, during deuterium–tritium experiment (DTE1) at JET 40% of the tritium introduced into machine was retained by the torus [6]. For that reason not only does the tritium retention but also the tritium recovery from FWM requires an extensive investigation. The tritium retention is expected to be depend to the machine's plasma conditions but also to the material structure. For this purpose, several tritium depth profiles of a 4D CFC tile retrieved from TFTR were compared to 2D CFC tiles obtained from JET. In this paper tritium profiles of TFTR and JET tiles will be presented and the relation between the tritium distribution within the tiles and the woven fibre structure will be discussed.

Additionally, the coring/full combustion technique was used to measure tritium remaining in TFTR tiles after detritiation of complete tiles by oxidation in air at 500 °C.

2. Experimental

The available tiles for the present tritium analysis were:

(I) 2D CFC tiles (IN3 and BN4) manufactured by Dunlop and exposed to D–D and D–T plasmas in the MKIIa divertor at JET and
(II) Two 4D CFC tiles (KC11 and KC18) manufactured by FMI and exposed to D–D and D–T plasmas in the bay K of the inner limiter at TFTR. Tile KC11 had been baked to assay the tritium content [7].

A comparison of JET and TFTR operation parameters was reported by Skinner et al. [6]. Both types of CFC's have a density of about 1.8 g/cm^3. The 2D CFC tiles have the weave plans parallel to each other, whereas the 4D CFC tiles were woven with yarns oriented in four directions giving to the whole structure a tetrahedral shape (Fig. 1).

Several cylindrical specimens were retrieved from each tile using the coring technique described elsewhere [8]. The obtained cylinders had a diameter of approx. 0.78 cm and a height corresponding to the thickness of the tile. Every cylinder was cut in disks with a thickness of 0.1 cm. The 4D configuration appeared to be harder than the 2D one as more than 3 h were needed to core a cylinder from a 4D tile in comparison to less than 1 h needed for the same operation in a 2D CFC.

Quantitative determinations of the tritium depth were obtained using the well established and very accurate full combustion technique in a Vance type apparatus followed by liquid scintillation detection of tritium [9].

3. Results and discussions

3.1. Tritium profile of JET CFC tile IN3s1

Measurements from the JET fusion machine revealed a non-uniform surface distribution of tritium over the vessel [10] and this was confirmed by the analysis of

deuterium [11]. The tritium analyses have also revealed that substantial amounts of tritium is retained in the divertor region in form of flakes.

A significant part of the retained tritium was also found trapped deep in the bulk of the jet divertor tiles, particularly for tiles 3, 4 and 7.

Fig. 2 illustrates typical profiles owing to diffusion, observed for tiles 3 and 4 and confirmed by PIN diode measurements [3]. The x-axis on these figures is in millimetres scale and consequently the last point in each figure represents the rear side of the tile. The diffusion profile is so evident that it was possible to fit it with an equation of the type:

$$A_D = A_B + A_0 e^{(-kD)},$$

where A_D is the tritium activity measured (or calculated for the part lost during cuts), A_B the activity in the bulk of the tile, D the depth in mm and k is a constant having a dimension of inverse length (mm^{-1}) and it is dependent on the temperature and the nature of the tile (graphite or CFC) as well as its structure (2D, 3D, 4D etc.). For the JET divertor tile $1IN3_{s1}$ its value is fairly constant and about 0.2 mm^{-1}. A_0 represents the tritium activity on the surface of the tile (this does not include the tritium content of any deposited film on top of the surface).

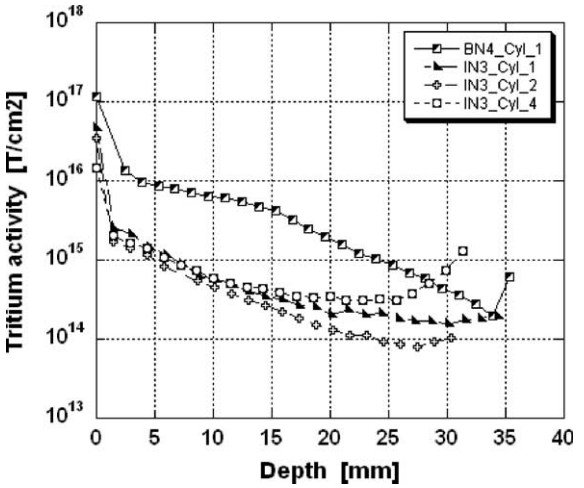

Fig. 2. Tritium depth profiles for IN3 and BN4, JET divertor tiles.

Having such equation allows also to estimate the activity lost during the cuts and consequently to better assess and correct the tritium content of the whole cylinder. Typical values for A_B, A_0 and k are given in Table 1.

Sample position was given in Ref. [3]. In the same reference the bulk fraction was estimated using an average value between two successive combustion measurements. Table 1 gives the corrected tritium activity present in the bulk of the tiles calculated by the above mentioned equation implying diffusion. It also compares the activity found in the first mm of the plasma exposed surface of tile (first disk) with the tritium activity in the bulk of the tile calculated by the equation. The high percentage (>67%) of tritium found in the bulk of cylinder 4 (divertor tile IN3) arises more from the low tritium concentration present in the surface of the sample, located in a shadowed area of the tile, rather than the amount of tritium trapped in the bulk of the tile.

Table 1
Tritium fraction in the bulk of various cylinders retrieved from JET 2D CFC divertor tiles after the DTE1 campaign and from TFTR 4D CFC tiles

Tile/ cylinder[a]	Volume of the cylinder (cm^3)	Plasma exposed disc (A_1) (T/disc)	Total activity (A_t) (T/cyl)	A_B (T)	A_0 (T)	k (mm^{-1})	Bulk fraction activity (%)[b]	Error (%)
IN3 cyl-1	1.85	2.24×10^{16}	3.59×10^{16}	8.46×10^{13}	1.65×10^{15}	0.21	37.6	5
IN3 cyl-2	1.60	1.65×10^{16}	2.57×10^{16}	2.74×10^{13}	1.03×10^{15}	0.16	35.8	6
IN3 cyl-4	1.55	6.98×10^{15}	2.12×10^{16}	1.57×10^{14}	1.16×10^{15}	0.21	67.1	4
BN4 cyl-1	1.70	5.58×10^{16}	1.43×10^{17}	–	–	–	**61.0**	12[c]
TFTR KC18 cyl_1	1.79	2.62×10^{16}	2.65×10^{16}	2.70×10^{12}	3.70×10^{14}	1.27	**1.1**	–
TFTR KC18 cyl_2	0.68	4.10×10^{16}	4.13×10^{16}	–	–	–	**0.7**	–
TFTR KC18 cyl_3	1.73	4.28×10^{16}	4.31×10^{16}	3.94×10^{12}	9.21×10^{13}	0.95	**0.7**	–

[a] The cylinders had a diameter of 0.78 cm.
[b] The bulk activity is calculated as $\{1 - (A_1/A_t)\} \times 100$ where (A_1/A_t) represents the fraction of the surface activity.
[c] The best fitting equation involves a double exponential expression taking in the account the front and the rear side diffusion.

It is also remarkable that for cylinder 4 strong diffusion seems to take place from the rear side of the tile. Such a phenomenon was already been observed for other tiles [3] but is not yet elucidated.

3.2. Tritium profile of TFTR 4D CFC tile KC18

The location of the cylinders drilled through the KC18 tile are indicated in Fig. 3. As to be seen cylinder 1 is located in a clear erosion zone whereas cylinders 2 and 3 belong to a more likely mixed erosion/deposition zone. Two additional samples (cylinders 4 and 5) were also retrieved from the side of the tile, as by visual inspection it appeared that one side of the tile was very clearly divided into two zones. An upper zone ('rainbow type') exhibiting almost a complete set of interference colours, and can be clearly distinguished from the darker bottom part of the tile.

In Fig. 4 tritium depth profiles for the TFTR tile 4D CFC KC18 are compared. For a better comparison with results obtained for the JET tiles, the activities were expressed in tritons per unit area of the plasma exposed surface. Naturally, bulk activities are better expressed in tritons per unit volume and are exactly one order of magnitude higher than the values illustrated in the figures as the thickness of each sample is 0.1 cm.

All cylinders have almost the same surface activity i.e. 1×10^{17} T/cm^2 with a notable maximum for the cylinder 4 at the edge of tile, while the neighbouring sample (cylinder 5) is much less active i.e. 3×10^{15} T/cm^2 (Fig. 4).

Another remarkable point is that the bulk activity of all cylinders sharply decreases after the first sample (1 mm below the plasma exposed surface) and seems to reach an almost constant value at about 2×10^{14} T/cm^3. In comparison to the bulk values observed for the 2D CFC tiles of JET, the TFTR value is more than one order of magnitude lower. Comparing tritium profiles for both type of CFC tiles (2D and 4D) it appears that for a similar tritium surface activity the tritium inventory on a 4D CFC is much lower. On the other hand, a diffusion profile (as observed for the JET divertor tiles) is not present for the TFTR ones (compare Figs. 2 and 4).

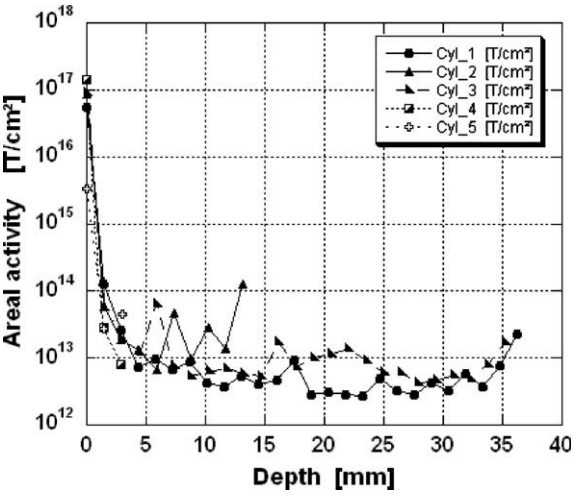

Fig. 4. Tritium depth profile for TFTR KC18 (4D CFC) tile.

If this observation is related to a lower diffusion (in a 4D) of small molecular species (Q_2, Q = H, D or T) between the weaves of the CFC fibres, weaving CFCs at higher levels (7D or even 11D) will considerably reduce the tritium retention in the bulk of the tiles, while at the same time the thermomechanical properties of the tiles will remain intact. Indeed, transient heating by laser scans of 2D and 4D CFC's have produced similar temperature excursions when a similar type of the tile surface was treated (co-deposited, or eroded zone) [12,13].

For both type of tiles bulk concentrations of tritium appear to be fairly homogeneous, irrespective of large variations in tritium concentrations on the plasma-exposed surface (see the data in Table 1 and Figs. 2 and 4).

3.3. Detritiation by oxidation in air

A variety of techniques are available for the in situ removal of tritium from the plasma facing components, i.e. glow discharge and air ventilation in TFTR [14,15] and JET [16], or by thermal release under different vacuum or oxidant atmosphere for the removing of H-isotopes in TEXTOR [17].

When dealing with the tiles outside of the reactor, more severe treatment techniques are acceptable, not only because single tiles or packages can be treated, but also because no precautions concerning the structural parts of the machine need to be taken.

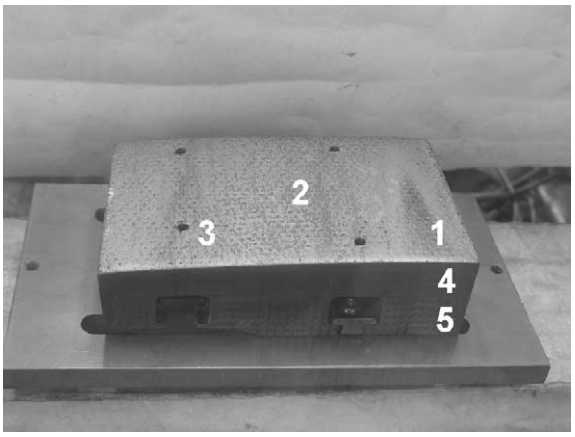

Fig. 3. KC18 4D TFTR, tile including sample location.

Among them, tested at laboratory scale [18], tritium release from co-deposited layers of graphite samples is known to be very rapid when temperatures rise above 350 °C [19]. Nevertheless, a substantial amount of tritium is still present in the sample after the thermal treatment. In a new series of experiments, complete CFC and graphite tiles retrieved from TFTR were heated under air at temperatures up to 500 °C, not only to improve the detritiation factor but also to assess the technique in full scale.

The tiles were heated up to 500 °C in an air atmosphere for 1 h. Then they were sent to Tritium Laboratory where several cylinders were drilled out and their tritium content measured using the coring/full combustion technique. The results for the 4D CFC tile KC11 are illustrated in Fig. 5.

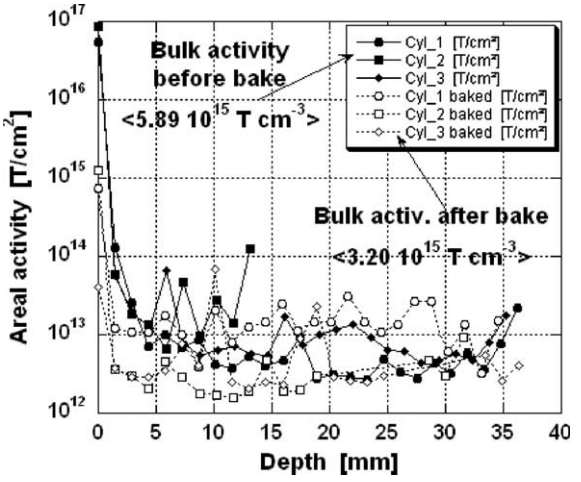

Fig. 5. Comparison of depth tritium profiles for the baked 4D KC11 and the unbaked 4D KC18 TFTR tiles.

It is remarkable to see that after the bake-out the surface activity of the tiles strongly decreases by up to three orders of magnitude. However, the total tritium contained in the bulk of the tiles is reduced by a factor two (see also Table 2).

Baking the tile under air even at 500 °C does not detritiate the bulk. Therefore, other techniques, such as laser heating [20], are more efficient for surface detritiation and more advantageous as they do not require tile removal and manipulation.

4. Conclusions

Tritium depth profiles in divertor tiles retrieved from TFTR and JET have been obtained by the coring/full combustion technique. For approximately identical surface activity, even if the tiles are retrieved from different location from both machines, the tritium distribution into the bulk is quite variable in level and profile. The results revealed that a surprisingly large fraction (up to 61%) of the retained tritium had diffused deep into the bulk of the 2D CFC tiles from JET in comparison to only few percent found in the 4D CFC tile from TFTR. The most probable mechanism involves tritium diffusion between the woven sheets of the CFC. The co-deposited layer and the saturated layer are independent of the woven substrate. Therefore, tritium inventory in FWM must be attributed to the hydrogen diffusion into the bulk of the tiles and is highly dependent to the woven fibre sheets. The comparison was made between several JET divertor tiles and only one TFTR tile. In order to increase the accuracy and verify the influence of the

Table 2
Tritium fractions of KC11 after 1 h bake-out at 500 °C under air compared with the unbaked tile KC18

Tile/cylinder[a]	Volume of the cylinder (cm^3)[b]	Plasma exposed disc (A_1) (T/disc)	Total activity (A_t) (T/cyl)	Bulk activity $(A_t - A_1)$/vol. (T/cm^3)	Bulk fraction activity (%)[c]
Baked KC11 cyl_1	1.73	3.56×10^{14}	5.87×10^{14}	2.31×10^{14}	39.3
Baked KC11 cyl_2	1.38	6.01×10^{14}	6.89×10^{14}	8.80×10^{13}	12.8
Baked KC11 cyl_3	1.61	1.93×10^{13}	1.51×10^{14}	1.32×10^{14}	87.2
Average	–	$\langle \mathbf{3.25 \times 10^{14}} \rangle$	$\langle 4.76 \times 10^{14} \rangle$	$\langle \mathbf{1.50 \times 10^{14}} \rangle$	–
Unbaked KC18 cyl_1	1.79	2.62×10^{16}	2.65×10^{16}	3.00×10^{14}	**1.1**
Unbaked KC18 cyl_2	0.68	4.10×10^{16}	4.13×10^{16}	3.00×10^{14}	**0.7**
Unbaked KC18 cyl_3	1.73	4.28×10^{16}	4.31×10^{16}	3.00×10^{14}	**0.7**
Average	–	$\langle \mathbf{3.67 \times 10^{16}} \rangle$	$\langle 3.69 \times 10^{16} \rangle$	$\langle \mathbf{3.00 \times 10^{14}} \rangle$	–

The apparent increase in the bulk fraction in KC11 is attributed to the detritiation of the surface rather to tritium diffusion deeper in the tile.

[a] The cylinders had a diameter of 0.78 cm.

[b] The volume of the cylinder takes in the account the different size of some bulk samples as some of them could be not complete because of the presence of structural material inside the tiles.

[c] The bulk fraction activity is calculated as $= \{1 - (A_1/A_t)\} \times 100$ where (A_1/A_t) represents the surface activity.

weave of the CFC fibre structure, a more systematic investigation must be undertaken involving also the analysis of higher level of multidirectional woven sheets.

Additionally, the coring/full combustion technique has shown that only the surface tritium (few ten μm) is efficiently released by air baking while the bulk tritium is almost not affected.

In conclusion, if the multidirectional CFC allows a very low tritium level in the bulk of the tiles the development of a detritiation technique to treat the tile's surface in situ may be sufficient to significantly reduce the tritium inventory in fusion machines.

In the above reported comparison between 2D and 4D tiles we have also to take in the account that the JET divertor tiles are continually at 150–250 °C [21] whereas the bulk of the TFTR tiles stayed cold. In order to have a better assessment of the woven fibre structure same type of tiles from both machines have to be compared in the future.

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