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Tritium depth profiles in graphite and carbon fibre composite material exposed to tokamak plasmas

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Abstract

Tritium inventories in the plasma-facing surface and the bulk of tiles were investigated with a highly sensitive, accurate full-combustion technique and a PIN-diode method. Examined were (i) a tokamak fusion test reactor (TFTR) graphite tile (D–D plasmas), (ii) a JET graphite tile (low-tritium D–T plasmas), and (iii) several JET carbon fibre composite (CFC) divertor tiles as well as a graphite limiter tile (all high-tritium D–T plasmas). Whilst the bulk tritium concentration in graphite tiles appears to remain at very low levels (about 0.3% of the total tritium) the tritium bulk concentrations in CFC divertor tiles can be as high as three times that in the surface layer. The latter is attributed to plasma-induced trapping of tritium between the fibre planes of CFC in the hot divertor zone. In addition to carbon/hydrogen co-deposition, this contribution constitutes another important source of tritium inventory in the torus that so far had not been recognised. © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

While much effort has been put into determining the tritium trapped in near-surface layers of tokamak plasma-facing components, the distribution of tritium in the bulk of these materials has been largely neglected. This is primarily because hydrogen isotopes other than tritium are difficult to quantify at very low concentrations. For instance, the determination of deuterium by surface analysis techniques requires at least 10^{15} D/cm^2 within the outermost 1 μm of the sample. As opposed to this, detection limits can be achieved for tritium which are higher by more than six orders of magnitude with techniques such as full-combustion, accelerator mass spectrometry (AMS) or a PIN-diode device.

Recent full-combustion and AMS studies with samples from JET tiles exposed to high levels of tritium, i.e., the first tritium experiment (FTE) and the first deuterium/tritium experimental (DTE1) campaign, gave peak deuterium/tritium concentrations at less than 50 μm from the surface [1–3]. In zones beyond the plasma-exposed surface of tiles only few measurements have been performed, all of which have provided evidence of low-hydrogen bulk levels. No deuterium was found by thermal desorption spectroscopy at distances greater than 1.5 mm from the surface of samples from ASDEX divertor tiles [4]. In a review [5], on the retention of hydrogen isotopes by graphite it was noted that the penetration of tritium into graphite increases with its porosity and can be influenced significantly by the presence of traces of metals such as occur in fusion machines (see also [6]). Peacock et al. [7] reported that after the FTE about 2% of the total tritium was trapped in the bulk of carbon fibre composite (CFC) tiles. These measurements were confirmed by Penzhorn et al. [1] in a study with graphite tiles from the tokamak fusion test reactor (TFTR) and from FTE JET.

The DTE1 campaign of JET using very high-tritium levels provided the opportunity for a re-determination of bulk concentrations at much higher anticipated tritium concentrations than those of previous campaigns.

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The results of these tritium profiling measurements are reported in this paper.

2. Experimental

2.1. Tile material

Graphite and CFC tiles were used during the DTE1 campaign. A description of the JET Mark IIA divertor arrangement existing at the time was published in [8]. Target CFC tiles were made from 2-D carbon-fibre-reinforced graphite with the weave planes normal to the plasma-facing surface.

CFC is produced by the so-called chemical vapour deposition process, which involves cracking of a hydrocarbon gas into woven carbon fibre sheets heated to temperatures of about 1000°C. After the carbon fibre substrate has been subjected to several heating cycles in the presence of the hydrocarbon, the composite is heat-treated at 2800 K to increase its thermal conductivity. This treatment gives rise to a product of comparatively low bulk density and high thermal stability up to temperatures above 2300 K. The thermal conductivity is high in the plane of the laminations and low perpendicular to the laminations. CFC is mainly used as a first-wall material because of its good mechanical properties, i.e., it is highly resistant to cracking by induced currents. Some physical properties of graphite and CFC are compared in Table 1.

2.2. Determination of tritium

Tritium in the tiles was determined either by the ‘coring’/PIN-diode method or the ‘coring’/combustion technique. With respect to ‘coring’, cylindrical specimens were cut with a hollow drill from selected graphite and CFC tiles used during the DTE1 campaign. For tritium analysis numerous disks were cut from each cylinder following procedures described in [1]. Most disks had a diameter of 7.8 mm and a surface area of 0.44 cm^2. Those obtained from tile 10N8 had a diameter of 11.5 mm. A brief outline of both techniques is given below. The disks typically weighed (0.090 ± 0.0033 g).

2.2.1. ‘Coring’/PIN-diode method

For a tritium depth determination by the ‘coring’/PIN-diode method, 22 disks were cut from cylinder 2 of the DTE1/$IN3_{S1}$ inner wall divertor tile, starting at the plasma-exposed side. The reverse face of each disk was marked to allow the unequivocal identification of the side nearest (obverse), and farthest (reverse) from the plasma. Disk faces were examined with a portable, windowless PIN-photodiode detector [9] in a darkened room to minimise background light contributions. The distance between the specimen and the detector was kept at about 1 mm. All measurements were performed while flushing the chamber with ≈ 50 cm^3/min of helium. The depth resolution of the technique is less than 1 μm and the detection limit under helium is of the order of 10^9 T/cm^2. To improve the statistics, each side of each disk was measured at least three times and average readings were recorded as a function of depth, beginning at 1 mm below the plasma-exposed surface of the tile.

2.2.2. Depth profiles in graphite and CFC tiles determined by the ‘coring’/full-combustion method

To obtain accurate tritium depth profiles in graphite and CFC tiles, cylinders were drilled at pre-determined positions of selected tiles and disks prepared as above. The tritium content in each disk was determined as described in detail in [1]. Basically, the disks were fully oxidised with moist air, and the produced tritiated water trapped in the water of a bubbler, which was then analysed by liquid scintillation counting. Data on the tiles examined are summarised in Table 2.

3. Results and discussion

3.1. Depth profiles determined with the ‘coring’/PIN-diode method

PIN-diode measurements performed on disks from cylinder No. 2 of the divertor tile $IN3_{S1}$ (see Fig. 1) are shown in Fig. 2 and compared with data from the full-combustion method. As evident, the concentration of tritium decreases with increasing distance from the plasma-exposed surface. Notably, the tritium concentration is in most cases higher on the obverse than on the

Table 1
Some physical properties of the graphite and CFC used at JET

Physical property	Ringsdorff graphite (EK98)	CFC (Dunlop DMS 704)
Density [g/cm^3]	1.85	>1.75
Porosity (open) [%]	10	<15
Specific electrical resistivity [μΩ m]	14	25–30
Thermal conductivity [W/mK]	90	x,y direction: >150; z direction: >40
Flexural strength [N/mm^2]	65	x,y direction: >120; z direction: >85

Table 2
Tiles examined for their tritium surface and depth profile (one from TFTR and all others from JET)

Tile designation	Type	Cylinder no.	Number of disks cut	Type of plasma exposure	Tritium concentration on cylinder surface [MBq/cm^2][a]
TFTR 121-1 SNO11 HOC/95-2	Graphite	1–9, 16, 17	23	D/D	0.0060 ± 0.0032
FTE/004/2-20	Graphite	2	12	D/D[b]	0.014
Poloidal tile PL4B MOD 7B-TOP	Graphite	2	13	D/T	592.1
Inner wall divertor tile DTE1/IN3$_{\rm S1}$	CFC	1	25	D/T	83.30
Inner wall divertor tile DTE1/IN3$_{\rm S1}$	CFC	2	22	D/T	61.50
Inner wall divertor tile DTE1/IN3$_{\rm S1}$	CFC	4	23	D/T	26.01
Inner wall divertor tile DTE1/IN3$_{\rm S1}$	CFC	5	9[c]	D/T	192.17[d]
Inner wall divertor tile DTE1/IN3$_{\rm S1}$	CFC	6	8[c]	D/T	3272.96[d]
Divertor base tile DTE1/1BN4	CFC	1	25	D/T	207.73
Divertor base tile DTE1/1BN7	CFC	3	22	D/T	59.86
Divertor base tile DTE1/1BN7	CFC	4	17[c]	D/T	213.27
Outer wall divertor tile DTE1/10N8	CFC	2	21	D/T	6.76

[a] In top 1 mm of the plasma-exposed side.
[b] Plus small D/T contribution from FTE.
[c] Limited by other holes drilled through the tile or cut-outs.
[d] Occulted side of the tile.

reverse side of the disks underlining the reliability of the PIN-diode measurement. Because of the steady and regular decrease in tritium concentration with distance from the tile surface these disks are ideally suited for these measurements.

A semi-logarithmic plot of the tritium surface concentration against depth yields

$$y_{\rm o} = 686.8\exp(-0.0816x)\ \text{(only obverse side of disks)},$$
$$y_{\rm r} = 650.9\exp(-0.0859x)\ \text{(only reverse side of disks)},$$

where x is in mm and y in Bq/cm^2. The ratio $y_{\rm c}/y_{\rm f} = 1.06$ is in line with plasma-induced tritium diffusion from the plasma-exposed side into the CFC bulk. In addition, some low activation energy diffusion combined with trapping, probably along grain boundaries of the tile, is apparent from the rear surface of the tile.

3.2. Surface and depth profiles of tritium in selected graphite tiles as determined with the 'coring'/full-combustion technique

Tritium surface and depth profiles were determined in three different graphite tiles, i.e., TFTR tile 121-1 SNO11, JET tile FTE/004/2-20 and JET poloidal tile DTE1/MOD7B TOP. Those in the first two were already discussed in detail in an earlier publication [1] and will not be treated further here. The estimated bulk fraction of tritium in these tiles is given in Table 3.

The sampling points (1–5) for the poloidal belt limiter tile MOD7 TOP are shown in Fig. 3 and the tritium levels in the first mm are listed in the caption. As anticipated [10], the tritium concentration increases progressively towards the deposition zones within the scrape-off layer (SOL) at both ends of the tile. Physical and chemical sputtering contribute to the erosion observed in the plasma-exposed zone of these tiles which manifests itself by relatively low-tritium concentrations (disks from cylinders 4 and 5). In the bulk, the tritium concentrations were found to be very low but still easily detectable and much higher than the normal background (which is ≈10 Bq/cm^2). The tritium concentrations in each of the 13 disks from cylinder No. 2 of the poloidal tile are plotted in Fig. 4. With progressing depth first a very abrupt decrease in tritium concentration is observed followed by a practically constant concentration. Based on the data, it is calculated that only ≈0.3% from the total tritium in the cylinder is present in the bulk. This result is consistent with previous measurements performed with other graphite tiles (see Table 3 and [1]).

3.3. Surface and depth profiles of tritium in selected CFC divertor tiles as determined with the 'coring'/full-combustion technique

In this section, the tritium surface and depth profiles of four representative CFC tiles, whose positions in the JET MkIIA divertor are illustrated in Fig. 5, are treated individually. The examined tiles were arranged with the weave planes normal to the plasma-exposed surface. The surface concentrations of tritium on these tiles showed normally variations within a factor of 4–25 (Fig. 1). Occasionally, however, much larger variations up to a factor of 126 (tile IN3$_{\rm S1}$) were observed, due to a very high contribution from co-deposited layers at the plasma-occulted side. In general, measurements performed on the tile surface with the PIN-diode yielded much higher variations than those determined by full com-

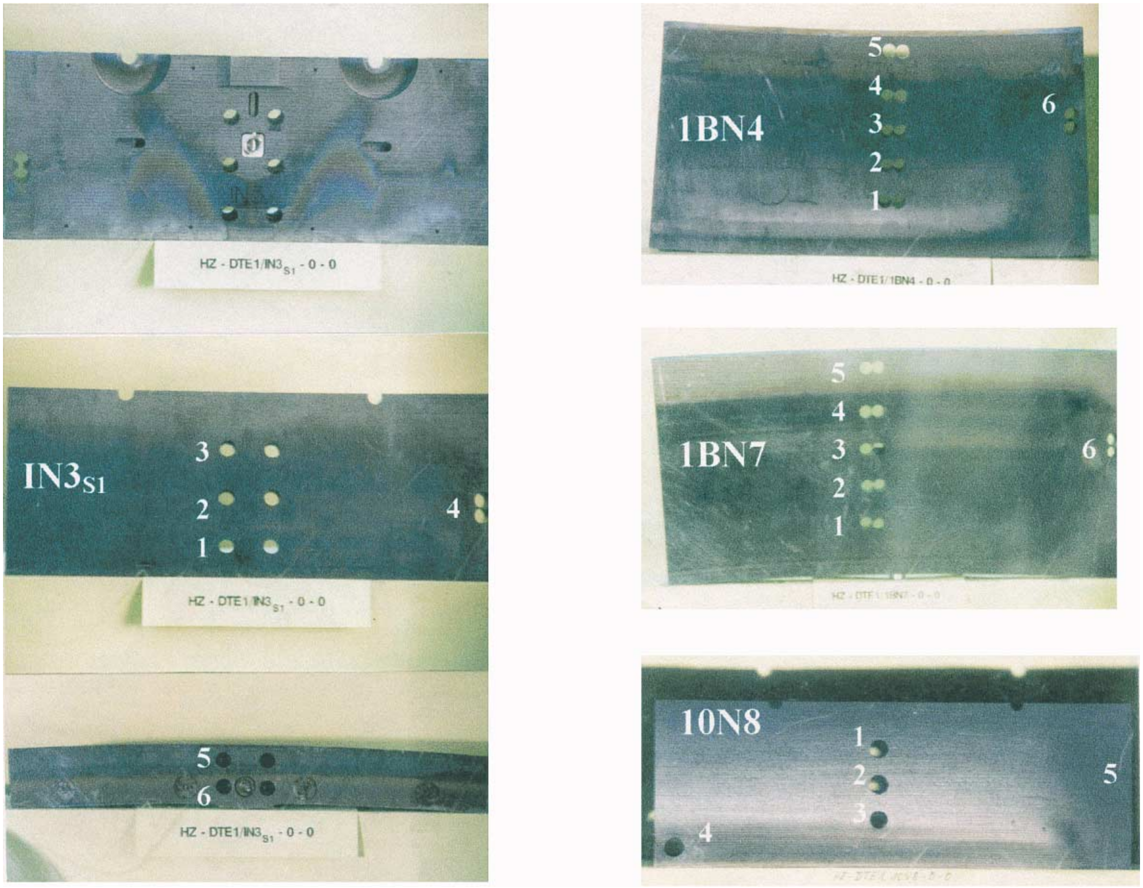

Fig. 1. Sampling points and measured tritium activities on JET, CFC divertor tiles. Shown on the left from top to bottom are the rear, plasma-exposed and lower edge sides of tile IN3$_{S1}$, respectively, and on the right the plasma-exposed sides of tiles 1BN4, 1BN7 and 10N8. The concentrations in the first mm depth (i) on tile IN3$_{S1}$ are 83.30 (1), 61.50 (2), 68.50 (3), 26.01 (4), 192.17 (5) and 3272.96 MBq/cm^2 (6), (ii) on tile 1BN4 are 207.73 (1), 306.10 (2), 72.75 (3), 163.75 (4), 859.20 (5) and 90.41 MBq/cm^2 (6); (iii) on tile 1BN7 are 263.96 (1), 222.39 (2), 59.86 (3) 213.27 (4), 12.86 (5) and 10.49 MBq/cm^2 (6); and on tile 10N8 are 3.49 (1), 6.76 (2), 5.86 (3), 2.73 (4) and 1.81 MBq/cm^2 (5), respectively.

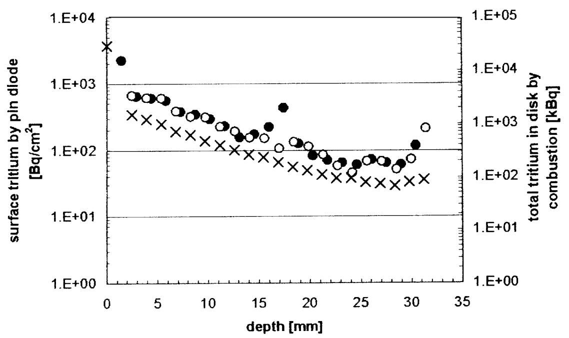

Fig. 2. Comparison between the tritium profile measurement performed with the PIN-diode (● obverse and ○ reverse side) and by full combustion (X) using the disks from cylinder No. 2 of the CFC inner divertor tile IN3$_{S1}$.

bustion, which is rationalised by the high-specific tritium activity of co-deposited/implanted surface layers.

3.3.1. *Tile IN3$_{S1}$*

The sampling points for tile IN3$_{S1}$ and the pattern of tritium are given in Fig. 1. The highest concentration was found at the most remote side of the bottom edge of the tile within the shadowed zone. The tritium concentrations on the plasma-exposed zone of the tile surface were all similar, varying by not much more than a factor of three. The total tritium activity on the surface of all the tiles in that poloidal position (i.e., integrated around the torus) was estimated to be 1.1 TBq at the plasma-exposed zone and 10.8 TBq at the plasma-occulted zone (see Table 4).

It was of interest to compare the depth profile from the shadowed region of tile DTE1/IN3$_{S1}$, where the

Table 3
Comparison of the tritium fraction in the bulk of a TFTR and various JET tiles

Tile designation	Cylinder No.	Depth examined [mm][a]	Total tritium in examined bulk depth [MBq][b]	Total tritium in first mm depth [MBq][c]	Fraction of total activity in the bulk [%]
TFTR 121-1 SNO11 HOC/95-2	Many	12	–	0.0022	<2
JET FTE/004/2-20	Many	12	–	0.0066	<1.1
Poloidal tile DTE1/PL4B MOD 7B TOP	2	18.2	0.7	275.9	0.3
Inner wall divertor DTE1/IN3$_{\mathrm{S1}}$	1	37.2	27.4	39.8	40.8
Inner wall divertor DTE1/IN3$_{\mathrm{S1}}$	2	31.2	16.6	29.4	36.1
Inner wall divertor DTE1/IN3$_{\mathrm{S1}}$	4	32.4	31.9	12.4	72.0
Inner wall divertor DTE1/IN3$_{\mathrm{S1}}$ shadowed zone low activity	5	12.5	17.9	91.9	16.3
Inner wall divertor DTE1/IN3$_{\mathrm{S1}}$ shadowed zone high activity	6	11.3	9.4	1564.5	0.6
Divertor base tile DTE1/1BN4	1	35.3	159.2	99.3	61.6
Divertor base tile DTE1/BN7	3	31.70	6.3	28.6	18.1
Outer wall divertor DTE1/10N8	2	29.4	4.2	7.0	37.5

[a] The examined depth was limited by holes or the back of the tile itself.

[b] The total tritium in the bulk was obtained from the sum of the tritium concentration in all the disks plus that estimated to be in the cuttings plus that obtained by extrapolation of all measured tritium concentrations to a depth of 0.2 mm.

[c] It was assumed that most of the activity in the first mm depth was in a layer <50 μm thick.

Fig. 3. Sampling points and measured tritium activities on JET graphite limiter tile PL4BMOD 7B TOP. Shown from top to bottom are the lateral, plasma-exposed and front sides, respectively. The concentrations in the first mm depth are 3.8 (1), 592.05 (2), 290.51 (3), 42.77 (4) and 12.61 MBq/cm^2 (5).

fibre planes are parallel to the surface (cylinder 5), with that nearby on the plasma-facing side of the tile (cylinder 1), where the fibre planes are normal to the surface (and approximately parallel to the incident plasma). Fig. 6 shows the tritium profiles from cylinders 1 and 5 measured normal to the respective surfaces. As shown for cylinder 5, in spite of the very high-tritium concentration in the surface layer, the tritium concentration first drops rapidly and then changes only slowly in the direction across the fibre planes. Actually, the concentration of tritium parallel to the plasma-exposed side remains effectively constant and that normal (cylinder 1) continues to decrease exponentially. The two lines cross at the depth/concentration point at which both tritium profiles have the same values, i.e., about 7 mm, an indication that diffusion between the carbon fibre planes is much more efficient than perpendicular to the planes.

Tritium depth profiles were obtained with disks cut from cylinders 1, 2 and 4 removed from the plasma-facing side of the inner wall divertor tile DTE1/IN3$_{\mathrm{S1}}$ and are compared in Fig. 7 (see also Fig. 1). As within the first 20 mm of depth all three profiles are very similar it is clear that the tritium surface concentration, which in this case varies by a factor of more than three, has no significant impact on the tritium concentration profile in the bulk. Furthermore, in all three cylinders one can observe a small, progressively increasing tritium concentration towards the back of the tile. This indicates that to some extent tritium also penetrates the CFC tiles from the reverse side. The reason for the particularly high increase observed in cylinder 4 is at present not

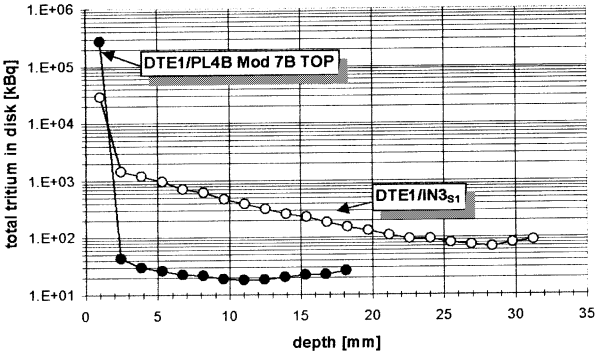

Fig. 4. Depth profiles of tritium in the bulk of the graphite limiter tile PL4B MOD 7B TOP in cylinder No. 2 (●) and in the bulk of the CFC divertor base tile DTE1/IN3$_{\mathrm{SI}}$ in cylinder No. 1 (○).

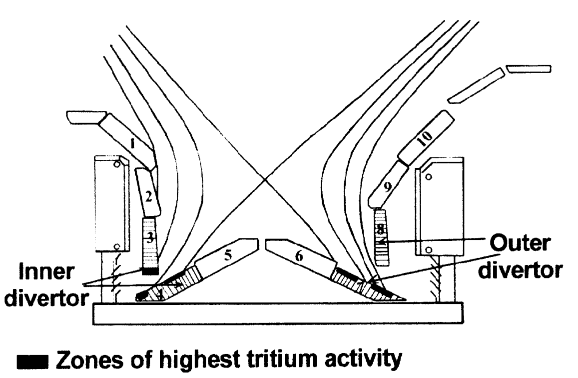

Fig. 5. Cross-section of the JET Mark IIA divertor giving the arrangement of the investigated tiles. The typical orientation of the weave planes of four divertor tiles are indicated schematically.

understood. It may have to do with the position of the cylinder at the edge of the tile.

A good test whether tritium in the co-deposited layer on top of a fibre layer influences the bulk profile was provided by cylinders 5 and 6, whose surface concentration varied by a factor of 17 (see Fig. 8). The profiles for these two cylinders are measured normal to the tile edge and are therefore at a fixed distance from the plasma-facing surface of the tile ($\approx$10 and 20 mm for cylinders 5 and 6, respectively). As it is seen, the cylinder with the lower tritium surface concentration (cylinder 5) has actually the higher bulk values in agreement with its position closer to the plasma-exposed side of the tile. This supports the postulation that plasma-induced bulk trapping takes place mainly between the carbon planes and to a much lesser extent through them.

The tritium depth profile in one cylinder determined by full combustion/liquid scintillation counting is compared with that obtained with the PIN-diode method in Fig. 2. With the exception of a few PIN-diode values at depths around 15 mm, the correlation between both measurements is remarkably good. Even the increase in tritium concentration registered at the rear of all tiles is detected by both techniques. The tritium concentration at the surface of each of these disks appears to be proportional to that in the bulk. (As a rule the usefulness of surface tritium detection techniques is limited by spurious surface concentrations originating from such plasma/wall interactions as co-deposition, implantation, erosion, etc.)

Assuming a depth resolution of the PIN-diode of 1 μm and considering a disk thickness of 1 mm, it is possible to estimate the tritium content in a bulk disk. The obtained values agree within a factor of about two with those gained from full combustion (Fig. 9). The PIN-diode method could thus be used advantageously to measure bulk tritium and contribute to the solution of waste certification issues.

Fig. 4 illustrates the significant difference between a graphite and a CFC tile with respect to the tritium

Table 4
Tritium concentrations in selected divertor tiles integrated around the torus

Tile type	Range of measured tritium surface concentrations [MBq/cm^2]	Estimated tritium surface concentration on tiles [TBq]	Estimated total tritium concentration of tiles [TBq]
IN3$_{\mathrm{SI}}$	26.01–3272.96	11.9[a]	13
1BN4	72.75–859.20	7.3	19.0
1BN7	10.49–263.96	5.1	6.2
10N8	1.81–6.76	0.2	0.3

[a] Incl. 10.8 TBq from co-deposited material on the shadowed bottom edge of tile.

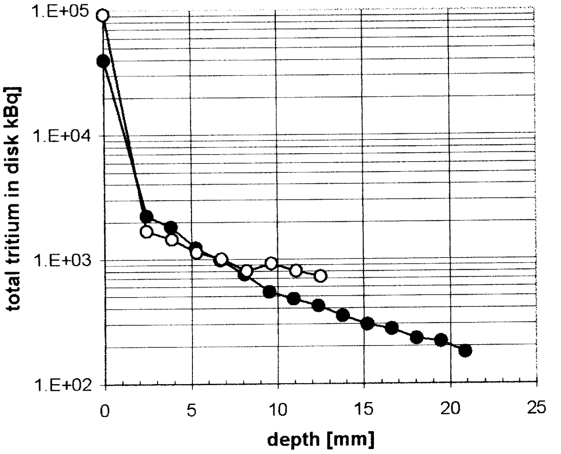

Fig. 6. Tritium depth profile parallel (○, cylinder No. 5) and normal (●, cylinder No. 1) to the plasma-exposed side of the JET divertor tile DTE1/IN3$_{\mathrm{SI}}$.

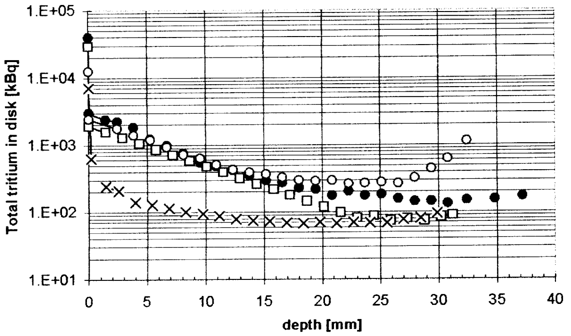

Fig. 7. Tritium depth profiles measured from the plasma-exposed side of the inner wall divertor tile IN3$_{\mathrm{SI}}$ and the outer wall divertor tile 10N8. The symbols ●, □ and ○ denote the tritium in the disks 1, 2 and 4 (0.44 cm^2 surface area), respectively, of tile IN3$_{\mathrm{SI}}$. Shown for comparison is the depth profile of cylinder No. 2 (0.44 cm^2 surface area) of divertor tile 10N8 (X). The values at 0.2 mm depth were obtained by extrapolation.

fraction in the bulk. While almost four orders of magnitude difference are registered in graphite between the surface and the bulk, this difference amounts to about one order of magnitude in the case of CFC. At this stage, it is still too early to decide on whether this difference is only due to the different carbon structures or the specific plasma wall interactions. More investigations on the tritium distribution in tiles are presently under way to improve the understanding of these issues.

3.3.2. Divertor base tile DTE1/1BN4

Whilst high surface concentrations of tritium are observed in the plasma-shadowed region of divertor base tile 1BN4 (cylinder 5, Fig. 1), the rest of the surface has only intermediate concentrations because of the erosion. Cylinder 1 was taken from a tile region that experiences the highest incident power from plasma ions (strike zone) and has probably reached high bulk temperatures. The depth profile of this cylinder indicates that tile 1BN4 has rather large tritium bulk concentrations (see Fig. 10). For a full evaluation of the tritium distribution in this tile further data are presently being obtained at the TLK. As with all CFC and graphite tiles, there is also for this tile strong evidence for some tritium penetration from the back.

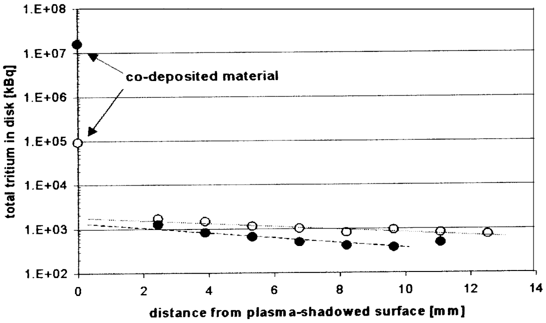

Fig. 8. Tritium depth profiles in cylinders No. 5 and 6 from the bottom plasma-occulted side of the inner wall divertor tile IN3S1. The symbols ○ and ● denote cylinders 5 and 6 having tritium concentrations of 91.86 and 1564.47 MBq/disk, respectively, at 50 μm depth.

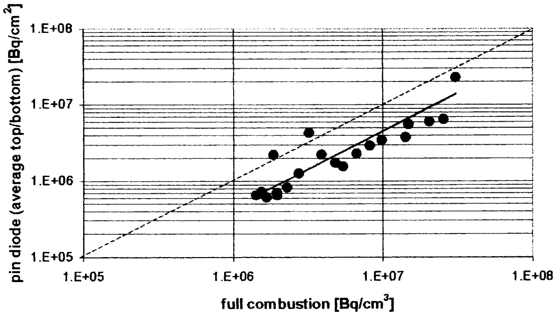

Fig. 9. Correlation between the tritium determination by full combustion with that by the PIN-diode method when it is assumed that the activity measured in 1 μm depth is the same throughout the whole 1 mm disk.

3.3.3. Divertor base tile DTE1/1BN7

Depth profiles of the almost symmetrically arranged divertor base tiles 1BN7 and 1BN4 are compared in Fig. 10 (see also Fig. 5). Cylinder 1 from tile BN4 and cylinder 3 of tile BN7 are similarly placed with respect to the usual strike zones on the tiles. The significant difference between the depth profiles of these inner and outer divertor tiles is not readily understood.

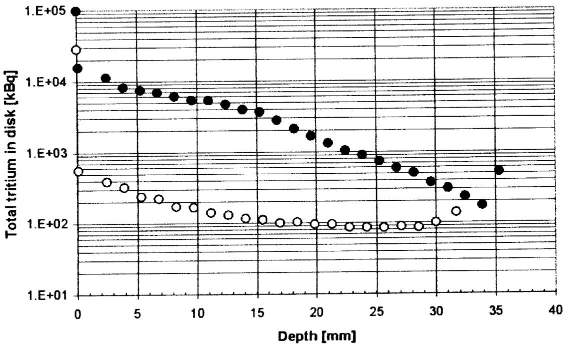

Fig. 10. Comparison between the tritium depth profiles of the symmetrically arranged JET divertor base tiles 1BN4 (●, cylinder 1) and 1BN7 (○, cylinder 3). The activities at 0.2 mm depth are extrapolated values.

3.3.4. Outer wall divertor tile DTE1/10N8

The depth profile of divertor tile 10N8 is plotted in Fig. 7 together with that of tile $IN3_{SI}$ on the symmetrically arranged opposite divertor side. In spite of the fact that both tiles are in a zone of high-flux and temperature, tile 10N8 shows very low-tritium surface concentrations and a relatively flat tritium depth profile at low levels. The latter is conceivably due to tritium diffusion into the large open porosity followed by thermal diffusion into the graphite and/or adsorption on energetic defects and/or isotopic exchange with pre-existing hydrogen isotopes. More relevant experiments are necessary to explain this difference.

3.4. Implications of the results

Tritium was found to be present in the bulk of all examined tiles (Table 3). The lowest tritium bulk-to-surface concentration ratios were found in graphite tiles, i.e., TFTR 121-1, JET FTE/004/2-20 and DTE1/PL4B MOD 7B TOP. In the first two tiles, the concentrations were so low that it was not possible to obtain good quantitative data. In the PL4B poloidal tile, on the other hand, the lowest activities were of the order of 20 kBq, easily distinguishable from the background. Since the activity is distributed fairly uniformly within the bulk without showing a clear depth profile, it is probably located in the graphite macroporosity trapped by an adsorption or an isotope exchange mechanism.

The tritium concentrations in CFC tiles were higher by at least an order of magnitude than those in graphite. No correlation was distinguishable between the concentration on the CFC tile surface layer and that in the bulk either in the direction parallel or normal to the fibre planes. It should be noted that the measured tritium concentrations were obtained after a limited DT campaign and that the tiles have subsequently been subjected to numerous deuterium plasmas, glow discharge cleaning and other wall conditioning treatments. It is therefore probable that the measured values are well below the steady-state values achievable after prolonged tritium operation of the machine. The tritium bulk concentrations in JET tiles other than the divertor remain to be investigated.

The 'low-limit' tritium activity in tiles $IN3_{SI}$, 1BN7 and 10N8 is of the order of 1.10 MBq/g (weight of disk 0.09 g) and can be ascribed to a low activation energy diffusion into the macropore system followed by trapping [4]. In a tile of 3.5 kg weight this gives rise to a 'low-limit' activity of about 4 GBq/tile.

Table 4 summarises the impact of the tritium profiling results on the tritium inventory in the divertor poloidal position integrated around the torus. The large contribution of co-deposit layers on the lower edge of tile $IN3_{SI}$, previously identified as highest source, is now less than that in the bulk of tile 1BN4. Clearly, the inner divertor constitutes the most tritium-contaminated zone. That the outer wall tiles are much cleaner than the divertor floor or the inner wall tiles had already been noted by Coad et al. [11] in deuterium surface analysis measurements. Inventories lower by a factor of two in the outer divertor zone were also reported for ASDEX upgrade [4].

4. Conclusions

The PIN-diode technique is a highly reliable and sensitive method for the detection of tritium on surfaces, provided the distribution of tritium is homogeneous. If these conditions prevail, a good correlation between the absolute amount in the bulk and that on the surface can be achieved. The technique has potential for application in bulk characterisation and possibly waste certification.

CFC tiles were found to contain more tritium in the bulk than graphite tiles. Although more data are still needed, it would appear that this enhanced tritium concentration is caused by plasma-induced trapping in the fibre planes of CFC.

Certain divertor tiles of CFC were found to contain a particularly large fraction of tritium in the bulk. The importance of this type of tritium retention, which is in addition to implantation, co-deposition and diffusion into the macropore system, had not been recognised previously. The bulk fraction is likely to increase with temperature and may be influenced by the ion flux on the divertor target tiles. No simple correlation was found between tritium co-deposited/implanted on CFC tile surfaces and that penetrated into the bulk.

The existence of a significant, accumulating, tenaciously held tritium fraction in the bulk of first wall materials represents an inventory with a long time constant that may be of significance for the next step machine. The saturation levels in a routinely operated machine are expected to be higher than those found in this work and need to be determined.

The tritium bulk fraction in the divertor tiles is not recoverable with methods that only treat the surface, e.g., a gas/solid interaction treatment, exposure to an open flame or laser irradiation. Simple isotopic exchange with protium or deuterium is also not expected to be effective.

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