

Reassessment of tritium content in CFC tiles exposed to the JET D-T campaign in 1997

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ABSTRACT

In 2019 two MkIIA divertor tiles (6IN3 and 4BN4) exposed during DTE1 were retrieved at CCFE for Thermal Desorption Spectroscopy (TDS) and pyrolysis analyses. A set of samples were prepared using a coring technique. The highest tritium (T) inventories were found in the shadowed corner of the inner divertor due to asymmetric deposition. TDS analyses indicated that T is desorbed at rather high temperatures with maximum release peaks at ~590 and 820 °C. A few samples were reannealed at 850 °C using the same heating procedure and it turned out that a further ~40–50 % of T was still released indicating that the annealing procedure used does not empty the sample completely. Pyrolysis results for thin disks cut from the surface of the tile were somewhat higher than the corresponding TDS results. T amounts were also investigated as a function of depth from the tile surface up to a depth of ~4.5 mm and T was detected at these depths. Comparison was also made with old results obtained with the pyrolysis technique and a PIN-diode method a few years after the DTE1 experiment, allowing for the natural decay and off-gassing of T. Our results agree within a factor of ~3 with these results.

Introduction

In 1996–1998 JET operated with a MkIIA divertor fitted with carbon fibre composite (CFC) tiles. A deuterium–tritium experiment (DTE1) was performed in 1997 introducing 35 g of tritium into the torus, mainly by gas puffing [1]. After the DTE1 experiment there was an extensive clean-up period to remove as much tritium from the vessel as possible, by running discharges in H or D and using glow discharge cleaning and baking. After this the machine was vented for tile exchange. All the divertor carriers were removed from the JET vessel, together with their CFC tiles still mounted. Two poloidal sets of carriers and tiles were retained for post-mortem analysis, whilst all the other carriers and tiles were put into storage in three ISOcontainers at ambient temperature vented to an extract system because of continuing chronic tritium release [2]. The MkIIA tiles are now considered redundant, and they are currently being de-tritiated by baking under oxygen in a furnace to

1000°C. During the intervening period there have been some measurements of the tritium off-gassing corresponding to the bulk tritium content of these tiles.

To satisfy Health and Safety concerns over movements of tritiated material from the JET tokamak following the DTE1 campaign, experiments at the Culham Laboratory determined a relationship between the off-gassing rate of carbonaceous dust and flakes collected from the divertor and their bulk content in 1998. This material had a very high surface-to-bulk ratio and as off-gassing is a surface phenomenon it may represent a safe limit for carbon samples removed from JET, since the released T is the associated hazard. The relationship between off-gassing rate (R, in MBq/day) and total T content is important for radioactive shipment regulations, which demand an estimate of the total T in a shipment. Based on these off-gassing measurements performed more than 20 years ago the following relation was established [3]:

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This relationship has subsequently been applied to all carbon-fibre composite (CFC) tiles taken from the JET machine (and indeed to other materials) for transport. However, off-gas values can only reflect the near-surface T content because diffusion of T from the bulk is very slow at room temperature, so off-gas cannot be a true measure of the total T content. Furthermore off-gassing must decrease with time as the surface inventory is reduced (otherwise in ~7000 days there would be none left). It was noted that measurements of off-gas to bulk content of tiles reported in 2014 gave somewhat lower ratios [4]. In this work we have highlighted a number of issues: the natural radioactive decay of T must be taken into account (half-life 12.3 years), off-gassing rate must also decrease with time and probably with a shorter half-life, thick surface deposits may spall off reducing both overall off-gassing and total T inventory, and T permeates into the bulk of CFC tiles depending on the extent of plasma bombardment.

The present work describes the last chance to make comparative measurements on these historic tiles. Two tiles (6IN3 and 4BN4) located at the inner corner of the MkIIA divertor were retrieved in 2019 for further analyses. The tile numbering in JET consists of the Octant number (e.g. 6), whether the tile is on an inner (I) or base (B) carrier, which may be a narrow (N) or wide (W) carrier, and the poloidal position of the tile (e.g. 3 or 4 – see Fig. 1). This work uses Thermal Desorption Spectroscopy (TDS) and pyrolysis (full combustion) to determine tritium contents in MkIIA divertor tiles, more than 20 years after the end of the DTE1 campaign, and to compare them with the T profiles measured shortly after DTE1 by the pyrolysis method on similar tiles.

The first pyrolysis measurements were conducted 20 years ago on very similar divertor tiles (including IN3_{SI} and 1BN4) retrieved from the JET machine in 1998. These first measurements showed that although the highest levels of T were present within the top 1 mm, T persisted throughout the tiles, and at that time cumulatively the bulk accounted for 16 to 72 % of the total T in the tile [5,6]. Other experiments have shown that even after a heat treatment (bake-out or RF heating) at about 500 °C a substantial amount of tritium remains in the tile [7,8]. Very high temperatures are required to almost “empty” tritium from a CFC tile; for instance, it was shown that an ex-situ heat treatment of a full MkIIA carbon tile at 1200 K with an open flame showed that 99 % of the tritium was released [9].

Experimental methods

In 1997 JET operated with deuterium and tritium fuelling for several months, the tritium amount in the plasma varying from 1 % to 100 %. Over this DTE1 campaign a total of 35 g tritium was introduced into the torus, mainly by gas puffing. At the start of the subsequent shutdown 6 g of tritium remained in the torus despite a thorough clean-up campaign lasting three months [1]. During the shutdown the torus was vented to air and continuously purged, and all the MkIIA divertor tiles were

removed due to the installation of the new MkII GB gas box divertor. Fig. 1 shows the poloidal cross section of the MkIIA divertor and highlights the tiles and samples analysed in this work. After the post-DTE1 shutdown with additional clean-up procedures it was estimated that ~3 g tritium remained in the torus [10].

In autumn 2019 two MkIIA divertor tiles, 6IN3 and 4BN4 (see Fig. 2) were retrieved from drums at CCFE for TDS and pyrolysis analyses. Both tiles are made of 2D carbon-fibre-reinforced graphite (CFC) with the weave planes normal to the plasma-facing surface. These tiles had all been handled (e.g. taken off their carriers) and were no longer bagged but just stacked in the drums. An inspection of the MkIIA divertors within their ISO-container in 2001 showed that the thick films on the shadowed ends of tiles 3 and 4 (see Fig. 2 (c)) and in the gaps between adjacent tiles 4 were spalling so were no longer present when the tiles were transferred to the drums.

For the present study samples were cut from each tile using a coring drill; the drill had an outside diameter of 20 mm and produced a core sample of 17 mm in diameter [11]. Fig. 2 (a-d) show the samples cored from both tiles. Two samples were also cut from the bottom of tile 6IN3 (see Fig. 2 (b)). For pyrolysis and TDS analyses three topmost disks with thicknesses of ~1-2 mm were cut from the cored samples from their plasma-facing sides (see Fig. 2(d)). For example, sample 6IN3/1-1 corresponds to the semi-cylindrical surface disk cut from core sample 6IN3/1 and sample 6IN3/1-2 is the next half disk cut from beneath the surface disk 6IN3/1-1, whilst 6IN3/1-3 is the one cut below that. The hatched area in Fig. 2 (a) and (c) indicates the samples cut for pyrolysis and TDS. Each half disc was cut into two pieces.

In this work the TDS system at UKAEA Materials Research Facility manufactured by Hiden Analytical Ltd (TPD Workstation type 640100) was used to anneal the cored samples from MkIIA tiles. The details of the TDS instrument with a base pressure typically of ~10⁻⁹ mbar have been reported extensively in a recent article [12]. Maximum annealing temperature of the system is 1000 °C but a protective layer of aluminium nitride (AlN) between the sample and heater plate limits the maximum sample temperature to ~850 °C, depending on the thermal contact with the AlN. The samples were annealed in vacuum with a linear ramp rate (10 °C/min) from room temperature (RT) to ~850 °C. The temperature was measured by a thermocouple embedded into the Mo sample stage which has direct contact with the sample. The released gases were measured with a line-of-sight quadrupole mass spectrometer as a function of time and annealing temperature. The D signal was calibrated using calibrated leaks for H₂ and D₂. The calibration factors for molecules DT and T₂ were extrapolated from corresponding factors for H₂ and D₂. The mass spectrometer can separate ⁴He from D₂ but not HT from D₂ because of insufficient mass resolution. The vacuum chamber was also annealed without a sample using the same annealing procedure to determine the background levels for masses 3 (HD), 4 (D₂), 5 (DT) and 6 (T₂).

Pyrolysis has been shown to be highly efficient for quantification of tritium in laboratory trials [13]. The samples were heated in air to achieve combustion. Tritium desorbed during the combustion of the sample (in the form ³H₂O) was collected in water bubblers and analysed by the Liquid Scintillation Counting (LSC) method in the Health Physics laboratory at CCFE.

Nuclear Resonance Analysis (NRA) measurements were made at Instituto Superior Técnico (IST, Lisbon) using ³He ions at an energy of 2.3 MeV for determining the amounts of deuterium using the D(³He, p)⁴He reaction [14]. The beam diameter was 1 mm. The 2.3 MeV ³He beam allows the deuterium content to be measured up to a depth of ~10 μm in carbon.

Secondary Ion Mass Spectrometry (SIMS) measurements were made using a double focusing magnetic sector instrument VG Ionex IX-70S at VTT. A 5 keV O₂⁺ primary beam with a current of 500nA was used [11]. The thickness of the co-deposited layer was determined from the SIMS depth profiles after the analyses by measuring the depths of the craters with a profilometer.

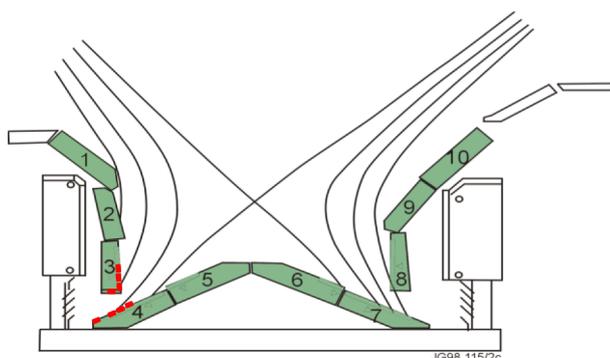


Fig. 1. Cross-section of JET MkIIA divertor. Analysed samples were cut from tiles 3 and 4.

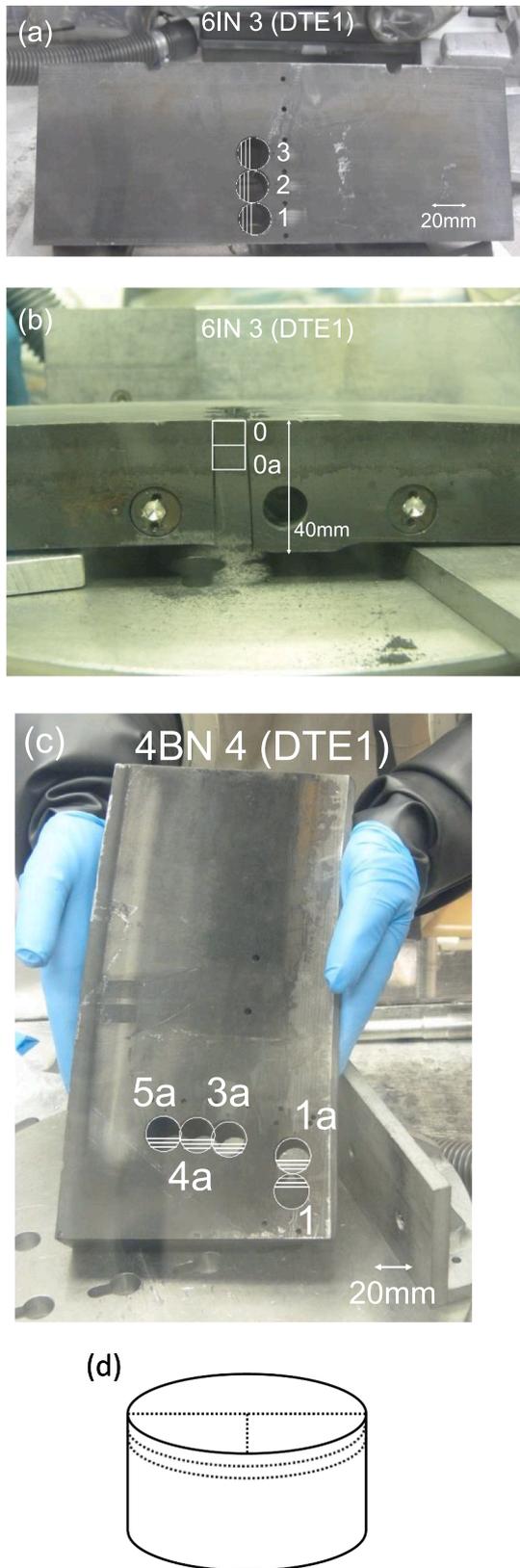


Fig. 2. Tile photos. Plasma facing surface of tile 6IN3 (a), bottom of tile 6IN3 (b) and plasma facing surface of tile 4BN4 (c). Samples for TDS and pyrolysis are marked with hatched area. Dashed lines in (d) show how disks were cut from a core sample for TDS and pyrolysis.

Results and discussions

TDS and pyrolysis results

Deposition and H-isotope retention in JET has always been asymmetric with heavy deposition found in the scrape-off layer (SOL) at the inner divertor and very little at the outer divertor. In this work we concentrated mainly on the inner divertor corner where the highest T amounts have been found [2], in agreement with the pyrolysis technique and the PIN-diode data [5].

Fig. 3 shows TDS spectra for sample 6IN3/0 (Fig. 3(a)) cut from the bottom of tile 3 and for sample 4BN4/1 (Fig. 3(b)) cut from the shadowed area of floor tile 4. For 6IN3/0 the desorption spectra for HD and D₂ show a broad release feature at ~590–600 °C and then a peak at ~820 °C (very close to the maximum temperature of 850 °C) whilst HD also has shoulders in the shape of the release spectra at ~195 and at ~350 – 425 °C; D₂ shows a clear peak at ~190 °C. Sample 4BN4/1 just shows a maximum release for HD and D₂ at ~820 °C with a small shoulder at ~590–600 °C. The DT release spectra for both samples mirror their respective HD and D₂ signals but without any evidence of lower temperature features, possibly due to the lower signal levels. The T₂ feature for 6IN3/0 is quite different, however, being dominated by a peak at ~590 °C with a slight shoulder at ~820 °C. The T₂ signal level for sample 4BN4/1 is rather low but seems to be a broad feature encompassing both the ~590 and ~820 °C regions. H₂ is always present as a background level in vacuum systems, but additionally it follows the shape of the respective HD and D₂ features. Although there is more D in 6IN3/0, the T and DT release appear to be comparable, whereas for sample 4BN4/1 the T is mainly released as a DT molecule, suggesting a different bonding mechanism for the T within the two sample surfaces.

D and T amounts for all the analysed samples measured with TDS and pyrolysis are given in Table 1. A comparison is made with the results published in Ref. [5]. The results in Table 1 allow for the natural decay

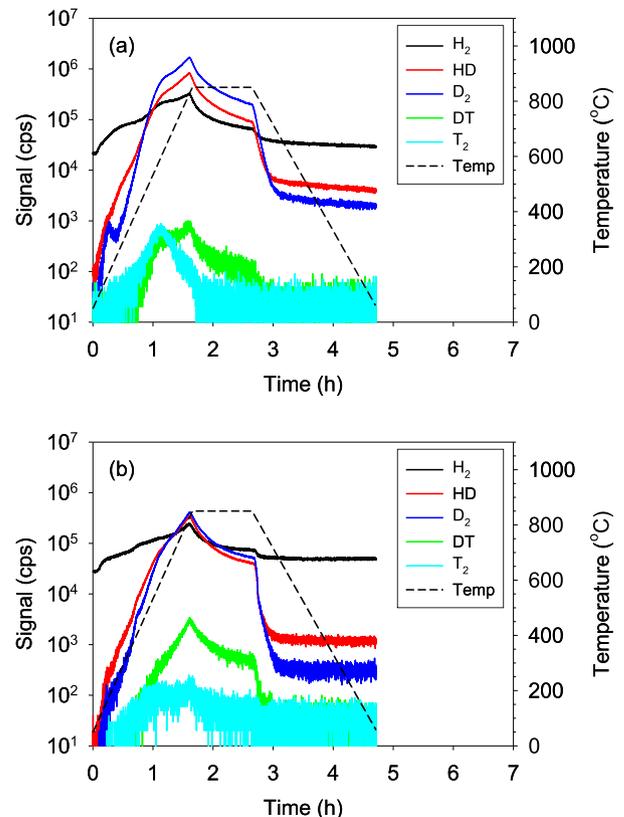


Fig. 3. TDS spectra and annealing temperature as a function of time for sample 6IN3/0 (a) and 4BN4/1 (b).

Table 1

D and T amounts for all the analysed samples measured with TDS and pyrolysis columns B-D). In columns E-G are shown the results for the samples in Ref. [5]. In column F the natural decay of T during ~20 years has been taken into account. In column G both natural decay and off-gassing of T have been included.

A Sample	B D amount (10^{15} cm^{-2}) TDS	C T amount (MBq cm^{-2}) TDS	D T amount (MBq cm^{-2}) Pyrolysis	E T amount (MBq cm^{-2}) Ref. [5] Pyrolysis	F T amount (MBq cm^{-2}) Ref. [5] Natural decay included	G T amount (MBq cm^{-2}) Ref. [5] Natural decay and off-gassing
6IN3/0	9480	9.1		192	62	52
6IN3/0a	22,600	25				
6IN3/1-1	581	3.4	13	83	27	16
6IN3/1-2	58	0.85	0.56	4.7	1.7	
6IN3/1-3	41	0.78		3.8	1.4	
6IN3/2-1	604	1.5				
6IN3/2-2	1.9	2	0.64	3.5	1.1	
6IN3/2-3	3.8	1.9	0.52	2.8	0.9	
6IN3/3-1	905	2.3				
4BN4/1-1	3900	28	165	215*	70	56
4BN4/1-2	11	0.75				
4BN4/1-3	25	2.0	0.29			
4BN4/1a- 1	1430	8				
4BN4/3a- 1	740	7.2				
4BN4/4a- 1	250	5.2		73	24	9
4BN4/4a- 2	13	0.3				
4BN4/4a- 3	5.0	1				

* We assume that original activity (859 MBq/cm^2) has decreased by a factor of 4 due to spalling. This factor is based on the ratio of the TDS results for the shadowed and the non-shadowed areas.

and off-gassing of T during the period 2000–2020. Off-gassing is estimated in the following way. For tile 3 samples were cut from the plasma-facing surface and from the plasma-occulted bottom face of the tile and gave values of 26.01 and 3272.96 MBq/cm^2 [5], respectively, there was a ratio of about 125 between these two measurements. However, in this work Table 1 shows T values varying by only a factor of ~10 between these same regions. As mentioned above, inspection of the MkIIA tiles after a few years in storage showed that most of the thick deposits in the plasma-occulted regions of both tiles 3 and 4 had spalled off, so if we assume that only 10 % of the co-deposited material is left on the shadowed surface of the tile this would make the data consistent. For tile 6IN3 the total T activity in 2000 of all tile 3 in JET was 13 TBq including the co-deposited material, with the shadowed bottom edge of the tile contributing 10.8 TBq to that total T activity (Table 4 in Ref. [5]). In addition, the estimated tritium surface concentration on all tiles 3 was 11.9 TBq. Out-gassing takes place mostly from the tile surface, so we use the estimated surface concentration (11.9 TBq) in our calculations. If we discount the 90 % that had subsequently spalled off, the effective T surface inventory would have been reduced by 9.7 TBq to 2.2 TBq and the total T surface activity per tile would have been 46 GBq since there are 48 such tiles toroidally in JET. Now if we use formula (1) given above (rounding the factor to 7000) the off-gas rate for tile 6IN3 would have been 6.5 MBq/day in 2000. The half-life of radioactive decay for T is 12.3 years so that alone would have reduced the T inventory for tile 3 from 46 GBq to 15 GBq, but there has also been T lost by off-gassing and we assume here an exponential decay in off-gassing. The measured off-gas rate in 2020 was 0.0582 MBq/day compared to the assumed off-gas in 2000 of 6.5 MBq/day; this implies a much shorter half-life for the off-gas decay of ~2.9 years. The approach explained above was used for calculating the T amounts in samples in Ref. [5] after a 20 year period.

Two samples were also cut from the bottom of tile 3 which is a deposition zone (see Fig. 2 (b)). These samples were cut from same toroidal location but sample 6IN3/0 is closer to the plasma facing surface and sample 6IN3/0a closer to the back side of the tile. There is more D and T on the sample 6IN3/0a probably because sample 6IN3/0 is closer to the plasma facing surface which gets hot during plasma pulses

(as was discussed in Ref. [5]). Sample 6IN3/0 from the bottom of tile 3 corresponds to sample 5 in Ref. [5] for which the T amount measured by pyrolysis was 192 MBq/cm^2 . If we take into account the natural decay of T only then the T activity in the sample would be 62 MBq/cm^2 . When both the natural decay and the off-gassing (using off-gas rate of 6.5 MBq/day and half-life of 2.9 years) are included then the T amount is 52 MBq/cm^2 . The result in Ref. [5] is somewhat higher than our TDS result but on the other hand it was observed in Ref. [4] that the T amount on tiles 3 and 4 can vary up to a factor of ~3 for tiles in other toroidal locations. Similar methodology was used for all the samples from tile 3 in Table 1 (columns E-G). When ratio between the results in columns E and G are calculated it can be observed that the ratio varies, i.e. for sample 6IN3/1-1 it is higher than for sample 6IN/0. We are using an average out-gas rate for the whole tiles. However, in reality most of the out-gassing will come from the high-T deposits and relatively little from the areas of plasma-facing surfaces with lower T amount. This means that the off-gas rate used (6.5 MBq/day) is most likely too small in case of sample 6IN3/0 but too high for sample 6IN3/1-1 resulting in too high T amount for sample 6IN3/0 and too low value for sample 6IN3/1-1, respectively. The column E/G ratios would be more consistent if it was possible to use local off-gas rates instead an average value. There are no possibilities to determine the off-gas rate separately for the high- and low-T areas so only possibility is to use an average value for the whole tile.

Similarly for tile 4; we find a factor of 3.8 between plasma-exposed and shadowed regions, whereas in Ref. [5] the factor is 11.8 (72.75 to 859.2 MBq/cm^2). If we therefore assume three-quarters of the deposit spalled off and that the shadowed region was ~20 % of the whole tile then the effective T inventory in 2000 reduces from 19 to 14.8 TBq on the full toroidal set of 48 tiles. Similarly, the estimated T surface concentration decreases from 7.3 TBq (Table 4, Ref. [5]) to 3.2 TBq for all the tiles 4 giving 67 GBq for one tile. Now if we use the formula (1) (rounding the factor to 7000) the off-gas rate for tile 4 was 9.5 MBq/day in 2000. The measured off-gas rate in 2020 was 0.141 MBq/day giving 3.3 years for the half-life of off-gassing with exponential decay. This agrees well with the half-life for off-gassing from tile 6IN3. In the case of

sample 4BN4-1 (corresponding to sample 5 in Ref. [5]) we have assumed that three-quarters of the deposit spalled off so the T activity would have been 215 MBq/cm² in 2000 instead of 859 MBq/cm². Radioactive decay reduces the T activity to 70 MBq/cm². When off-gassing is also taken into account the T activity for equivalent sample 4BN4/1 in Ref. [5] is 56 MBq/cm².

Depth distributions were obtained using the thin disks cut from 6IN3/1, 6IN3/2 and from 4BN4/1, and are shown in Fig. 4. TDS gives a somewhat smaller T amount in the surface disk 6IN3/1-1 than pyrolysis. On the other hand, the pyrolysis result agrees well with the result in Ref. [5] (sample 1) when both radioactive decay and off-gassing are included. In the case of bulk samples from Ref. [5] the radioactive decay only was taken into account in the depth profiles because off-gassing occurs only from the surface. The T amount decreases as a function of depth but the decrease in the TDS results is not as marked as for the pyrolysis data and as for the results obtained in Ref. [5, Fig. 4], or for the D amounts measured by Ion Beam Analysis (IBA). In the case of samples 6IN3/2 the D amount decreases even faster than for samples 6IN3/1 so there is very little D in the bulk of core 6IN3/2 indicating that D is mainly retained in the co-deposited layer on the surface of the sample. For all samples, in the bulk the TDS T results are comparable with the background level. Although a background level for D and T was determined by annealing the TDS chamber without a sample using the same heating procedure, subtracting this from the T levels may not be totally successful. Sample 6IN3/3 located near the centre of tile 3 has a rather similar T content in the surface disk to sample 6IN3/2.

Cores 4BN4/1 and 4BN4/1a were cut from toroidally adjacent areas where the original co-deposited layer appears to be at least partially present (see Fig. 2(c)). When comparing T amounts for these samples (Table 1) it can be observed that there is a difference by a factor of ~4 between the TDS results. Sample 4BN4/1-1 is closer to the edge of tile 4BN4 and may be just within the region shadowed by the adjacent tile 4, which may explain why there is more T (and D) than on sample 4BN4/1a-1. Sample 4BN4/1-1 was also analysed with pyrolysis. For sample 4BN4/1-1 the pyrolysis result is much higher than the TDS result and also clearly higher than the T amount in Ref. [5] for a corresponding sample. There is a scatter in the data in Ref. [5] as the T content varies in the range of 73–859 MBq/cm² so an error of a factor of 2–3 in the T values is acceptable agreement. T amounts in core 4BN4/1 decrease sharply as a function of depth but the TDS results (both T and D) for disk 4BN4/1-3 (3rd disk from the surface) seem to be higher than the result for the 2nd disk from the surface. Samples 4BN4/3a and 4BN4/4a have

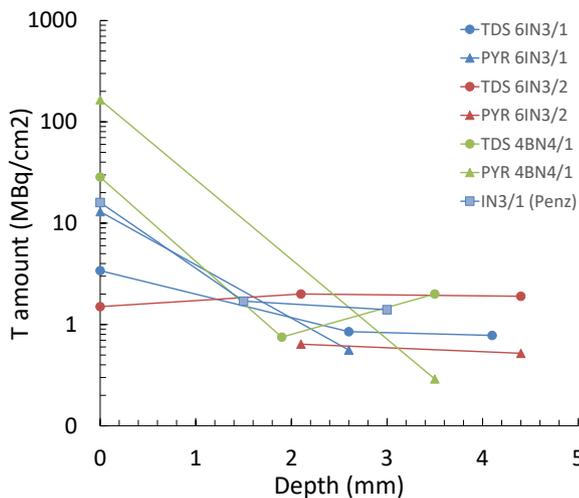


Fig. 4. T amount as a function of depth from tile surface measured with TDS (indicated as “TDS”) and pyrolysis (“PYR”). Results IN3/1 (Penz) are from Ref. [5]. Results for samples 6IN3/1-1 and 4BN4/1-1 include all the annealing steps.

comparable T amounts on the surface but there is more variation in the D amounts.

Two samples, namely 6IN3/1-1 and 4BN4/1-1, were annealed two and three times in the TDS system, respectively, with the result that during the second annealing a further ~40–50 % of T was still released so annealing to 850 °C does not release all the T. The third annealing of sample 4BN4/1-1 released a further ~10 % of T. Thus a single anneal leads to an underestimation of actual retention by at least 40 %. In the case of D the second annealing released a further ~20–30 % and the third annealing a further ~7 % which indicates that D is released somewhat more efficiently than T. The results for D and T for these two samples in Table 1 include the release from all the annealings. It can be observed in Fig. 3 that the high temperature release peaks are very close to the maximum annealing temperature of 850 °C. The amount of unreleased T is correlated with how close the maximum of the high temperature peak is to the onset of the holding period at 850 °C. Similar remaining fractions were observed in Ref. [15] in which bulk Be samples from JET limiters were annealed using the TDS system. The maximum annealing temperature of the Be samples was limited to 780 °C because at higher temperatures Be evaporation occurs leading to undesirable contamination of the vacuum system. The results obtained also indicate that when using the TDS system at UKAEA the samples need to be annealed at least twice for more accurate results.

In the case of samples from below the surface disk the DT and T₂ signals are very low and close to the background levels obtained when heating the TDS vacuum chamber without a sample. In all cases these background levels were subtracted from the signals of the annealed samples. This background subtraction is somewhat questionable for samples below the surface disk. The T content of these samples do not decrease as a function of depth (see Fig. 4) which suggests that the background subtraction fails, as was implied above. The results obtained in this work indicate that the quadrupole-based TDS used does not allow reliable quantification of T when the T amounts are below ~10¹⁵ at/cm². Pyrolysis and dissolution measurements combined with liquid scintillation are more reliable methods for measuring low T amounts [15].

As more general comments, off-gassing rates can provide an indication of the biohazard of shipping T-containing material but is not an accurate indicator of bulk T content. Off-gas measurement does require carefully controlled conditions such as control of the moisture content of the feed gas, detection of both tritiated hydrogen and water emanations, and reproducible containment; errors of a factor of two are possible. TDS is not an accurate method for measurement of bulk T content of carbon unless the sample is repeatedly cycled to at least 1000 °C, and the equipment has a higher sensitivity to T than that used in this work. Further experiments on samples from the two retained divertor tiles exposed during DTE1 combined with pyrolysis measurements would be very informative. Since TDS does at least disrupt the hydrogenic content of the sample, pyrolysis represents the best method for total T determination. Calorimetry is a possible non-destructive method for total T content, but as the heat of radioactive decay is small it requires extremely sensitive equipment [16,17]. The calorimeter used in Ref. [17] had a detection limit of 40 MBq for T which is much lower than for the instrument used in Ref. [16].

In the future the plasma-facing components (PFC) for ITER will be beryllium (Be) and tungsten (W). There will be requirements for transport of these materials both on and off the ITER site, and knowledge of T inventories will be required for Health and Safety. Off-gas measurements will be possible, but comparative measurements of bulk T content would also be expected, for samples from a variety of PFC locations. For W, heating to high temperatures can remove all T and give the bulk content. For Be it would be necessary to heat samples to the melting point and coupled with the high vapour pressure and toxicity of the material this becomes a less attractive option. Total T content of Be has been determined by chemical dissolution [18] but more work is required. JET is currently running further DT experiments with Be and

W as PFC materials before shutting down, so this could provide ideal materials to test methodology for ITER; unfortunately the removal of suitable materials is still uncertain.

NRA and SIMS results

NRA and SIMS were used mainly for analysis of D because T amounts are too low to be detected. In the case of SIMS the experimental setup does not allow analysis of T due to interference with the molecular HD ion which has a similar mass to T. Results for D amounts are given in Table 2. NRA and SIMS analyses were made from the remainder of the core samples after cutting the TDS and pyrolysis samples. The NRA results seem to be somewhat higher than the SIMS results but are of the same order of magnitude. SIMS analyses were made typically to depths of 20–60 μm so the fuel content in the whole co-deposited layer was analysed.

Conclusions

Analysis of MkIIA divertor tiles over 20 years after their exposure during the JET DTE1 experiment was performed using TDS and pyrolysis techniques. The highest T amounts were still detected in the shadowed inner corner of the divertor. TDS analyses indicated that T is desorbed at rather high temperatures with maximum release peaks at ~ 590 and 820 $^{\circ}\text{C}$. A few samples were reannealed using the same heating procedure and it turned out that a further ~ 40 – 50 % of T was then released indicating that the annealing procedure used does not empty the sample completely. In the case of D the second annealing released a further ~ 20 – 30 % and the third annealing a further ~ 7 % which indicates that D is released somewhat more efficiently than T. Pyrolysis results for thin disks cut from the surface of the tile were somewhat higher than the corresponding TDS results. T amounts were also investigated as a function of depth from the tile surface up to a depth of ~ 4.5 mm and T was detected in the bulk. Comparison was also made with results obtained with a pyrolysis technique and a PIN-diode method obtained a few years after the DTE1 experiment [5], compensating for the natural decay and off-gassing of T. Our results agree within a factor of ~ 3 with these results. In Ref. [4] it was pointed out that T amounts can vary by a factor of ~ 3 depending on the toroidal location of the tile. We also observed that T amounts can vary on the same poloidal location between neighbouring samples that are only a few centimetres away from each other.

Off-gas measurements of T from CFC tiles if performed under controlled conditions may be a good indication of the biohazard associated with the tiles but is not an accurate indicator of the bulk T content of the tiles. Furthermore the off-gassing becomes less indicative over time since the off-gassing rate decreases more rapidly than the natural decay rate for the T contained in the bulk of the tile.

CRedit authorship contribution statement

J. Likonen: Writing – review & editing. **N. Bekris:** Writing – review & editing. **J.P. Coad:** Writing – review & editing. **C. Ayres:** Resources. **N. Gotts:** Investigation. **I. Jepu:** Investigation. **Y. Zayachuk:** Investigation. **A. Widdowson:** Writing – review & editing. **I. Wilson:** Resources. **N. Catarino:** Investigation. **E. de Alves:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Table 2
D amounts measured with NRA and SIMS.

Sample	NRA ($\times 10^{15} \text{ cm}^{-2}$)	SIMS ($\times 10^{15} \text{ cm}^{-2}$)
6IN3/0	43,700	
6IN3/1-1	800	
6IN3/2-1	870	203
6IN3/3-1	1060	873
4BN4/1-1	8260	2700
4BN4/1a-1	8820	
4BN4/3a-1	2520	2030
4BN4/4a-1	943	640

Data availability

No data was used for the research described in the article.

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