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A T Peacock¹, P A Andrew, D Brennan, J P Coad,
H Hemmerich, S Knipe, R-D Penzhorn², M Pick.

JET Joint Undertaking, Abingdon, Oxfordshire, OX14 3EA,

¹Present address: EFDA Close Support Unit, Garching, D-85748, Garching, Germany.

²Tritium Laboratory, FZK, Postfach 3640, D-76021, Karlsruhe, Germany.

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ABSTRACT

At the start of the shutdown following the DTE1 experiment at JET there remained 6.2g of tritium in the JET vacuum vessel. An attempt has been made to establish the location of this tritium by measurement of components taken from the vessel. 2.0g of tritium was recovered by out-gassing and 1.5g was originally estimated for the flakes/deposits from measurement of collected flakes. Measurements on tiles removed from the vessel indicate that all the vessel tiles contain less tritium than expected at $\sim 0.1\text{g}$. Some uncertainty exists in the location of the remaining tritium. Further investigation has shown that a significant quantity of flakes (more than originally estimated) exists in the sub-divertor region. It is believed that the discrepancy between the tritium missing and that accounted for is present in the flakes in the sub-divertor region. However, until this material can be collected and quantified other possible sources cannot be excluded. The reasons for the formation of the flakes/deposits are discussed.

1. INTRODUCTION

During May to November 1997 JET was operated using mixed deuterium-tritium (D-T) plasmas, in a campaign known as "DTE1". The torus was fuelled with a total of 35g of T during this period, and the amount recovered from the vessel was re-processed and accurately measured by the Active Gas Handling System (AGHS). The AGHS measurements showed that the tritium inventory retained in the vessel accumulated at about 40% of the input [1]. Following the last tritium-fuelled pulse the experimental programme continued with deuterium- and hydrogen-fuelled pulses aimed at reducing the retained T inventory. The most effective means of removing T from the vessel was to run plasmas in deuterium (D) or hydrogen (H), and after a few days the T content in the plasma was reduced to $<1\%$, precisely similar to the experience gained in the JET Preliminary Tritium Experiment (PTE) in 1992. However, at the end of the clean-up period the amount of T left in the vessel remained at about 6g (17% of the fuelling). This was not in line with data from the PTE.

H isotopes retained in first-wall surfaces interacting with the plasma are rapidly exchanged (the dynamic inventory), and this accounts for the clean-up of the plasma. However, analyses of deuterium retained in the vessel prior to DTE1, but with the same divertor and first-wall geometry, showed very non-uniform patterns of retention over the vessel, with areas of erosion and deposited films distributed around the vessel. These deposited films were expected to retain tritium inventory after DTE1. One such area, a new source of retention specific to the MKIIa divertor, was the discovery of deposits/flakes in the inner divertor.

This paper describes the potential sources of retained tritium and their location. It also describes an analysis programme to map the T remaining in the vessel and to confirm the similarity (in percentage retained and distribution) to D retention. Similar deposition patterns are seen in other tokamaks, but it is not known when during a discharge they occur, or how they vary with plasma parameters and off-normal events such as ELMs. It has also not been possible to model

the tritium retention after DTE1. It has become clear that processes are occurring, which are not fully understood. It is vital that these processes are understood and that a model be developed that describes the H isotope retention in JET (and other tokamaks with a divertor geometry), and that this model be tested by varying parameters in JET. Such a model can then be used with confidence to predict the T retention in a next-step device.

2. SOURCES OF RETENTION

At the end of the operational campaign that contained DTE1 (a series of tritium-fuelled pulses and a clean up campaign) 6.2 g of tritium was still outside the AGHS, in the JET vacuum vessel. On opening up the vacuum vessel the out-gassing rate, expressed as a fraction of the retained tritium inventory, was 20 times higher than expected. As it takes a finite time to take samples and measurements, the location of the tritium cannot be measured at the start of the shutdown. The end of the shutdown will be considered as the date at which the location of the tritium will be evaluated.

The potential locations considered for tritium at the end of the shutdown are: Tritium released into the air during the shutdown, dust, deposits/flakes, divertor tiles, non-divertor tiles and tritium permeation through the vacuum vessel walls, particularly at the bellows which are 2 mm thick [2].

3. THE REMOTE TILE EXCHANGE (RTE)

After the DTE1 experiment a shutdown to exchange the divertor tiles was started [3]. This operation removed the MKIIa divertor and replaced it with the so-called “Gas box” divertor. The DTE1 experiment at JET produced $2.3 \cdot 10^{20}$ D-T neutrons and resulted in an in-vessel activation at the start of the RTE of 7 mSv/hr [4]. This precluded manned activity inside the Torus and all activities were performed remotely. The higher than expected out-gassing had only a limited effect on the shutdown.

As part of the RTE, special equipment was developed to enable samples of dust, flakes and tiles to be removed from the vessel for analysis. An example of this equipment was a special vacuum cleaner equipped with a cyclone collection device, shown in Fig.1. This vacuum cleaner was handled remotely and collected dust and flakes in four separate cyclone pots. Care was taken to avoid cross contamination of samples by exchanging the cyclone housing and pipes between collection exercises. All operations were planned and

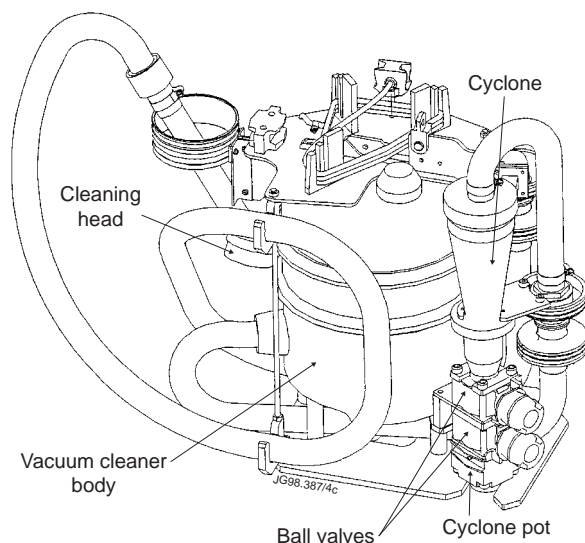


Fig.1: Specially adapted vacuum cleaner

practised prior to the start of the shutdown. It was originally foreseen that only samples of the flakes would be collected. However, it became apparent that the flakes contained a significant amount of tritium. This sample collection turned into a tritium inventory mitigation exercise, and an attempt was made to collect as much as possible of the flakes and the deposited films in those areas of the structure near the divertor inner leg. The location of the deposited material extends in all shadowed areas of the divertor inner leg. Some of this material was attached to tiles, as seen in Fig.2. Some of the flaked material had fallen through holes in the structure and was irrecoverable. Observation of the state of the inner divertor leg before and after cleaning and observation of the cleaning process produced an initial estimate of the tritium removed. This estimate states that 35% of the deposited material was removed with the tile carriers, 35% removed by the vacuum cleaning exercise, and 30% remained in the vessel. Access to the sub-divertor volume was not possible during RTE. Only samples of the dust and the tiles were taken.

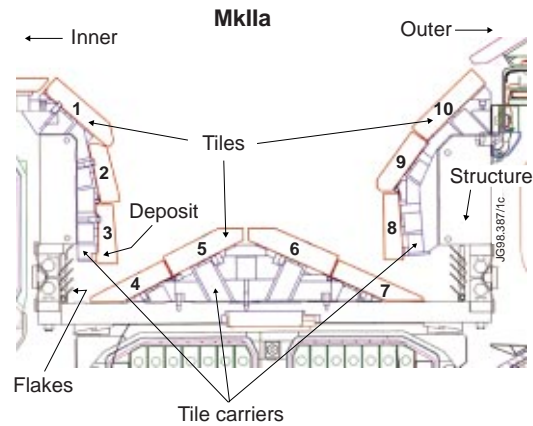


Fig.2: Section of the JET MKIIa divertor

The following were taken from the JET machine during the RTE for analysis.

- 10 divertor tiles
- 8 poloidal limiter tiles
- 6 inner wall guard limiter tiles
- 2 flake collections
- 2 dust samples

4. DEPOSITS/FLAKES

The co-deposition of deuterium with carbon in tokamaks has been known for many years. Carbon eroded from surfaces inside the machine is deposited onto other surfaces and during this process deuterium is incorporated. Normally these deposits are subject to continuous bombardment by the plasma. In JET it was also usual that the films would be deposited on surfaces which were hot, typically 300°C. The resulting films contained deuterium at a D/C level of up to 0.4 and in many instances much lower than this [5]. Such films still exist on the tiles in the main chamber of JET and on the divertor tiles.

On examination of the JET MKIIa divertor after operations thick films of material were found in the inner leg of the divertor in those areas shadowed from the plasma ions. These films had spalled from water cooled components (such as the structure louvres) forming flakes, typically 40 microns thick, as seen in Fig. 3. No such films or flakes were found in the outer leg. The films/flakes were found to be mostly carbon with some oxygen. However, the major difference

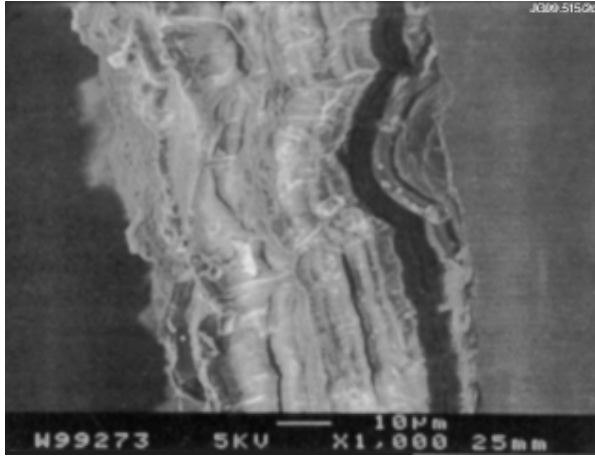


Fig.3: Flake photograph

between these flakes and material found previously in JET is the much higher content of hydrogen isotopes; the H/C ratio was measured by ion beam analysis (IBA) techniques to be at least 0.7 to 0.8. Furthermore, since these carbon films are not subject to plasma bombardment and are in relatively cool regions ($<200^{\circ}\text{C}$), the high levels of incorporated hydrogenic material are retained. This material is expected to contain a significant percentage of the T retained following DTE1.

4.1. Tritium measurements in flakes after the DTE1

The high levels of tritium and the high out-gassing rate from the flakes meant a special glove box had to be constructed to allow the flakes to be handled. This glove box was connected to the AGHS exhaust de-tritiation system to stop release of the tritium to the environment. For operational reasons the flakes were collected in two separate pots. The collected material was taken from each cyclone pot, weighed and transferred to a calorimeter to measure the tritium content. The operation was performed in air for the first sample and high levels of tritium off-gas were measured. The second sample was shrouded in inert gas and the levels of off-gas were greatly reduced.

Calorimetry is a standard technique that measures the heat evolved in a tritium sample as a result of radioactive decay. In performing the calorimetry measurement on the first sample it was noticed that the sample took a long time to settle down. This was attributed to an exothermic reaction of the flakes with water in the atmosphere. This effect also took place for the second sample, as air was not totally excluded, as can be seen from the curves in fig. 4. Results obtained with calorimetry for these quantities of tritium are usually accurate to better than 1%.

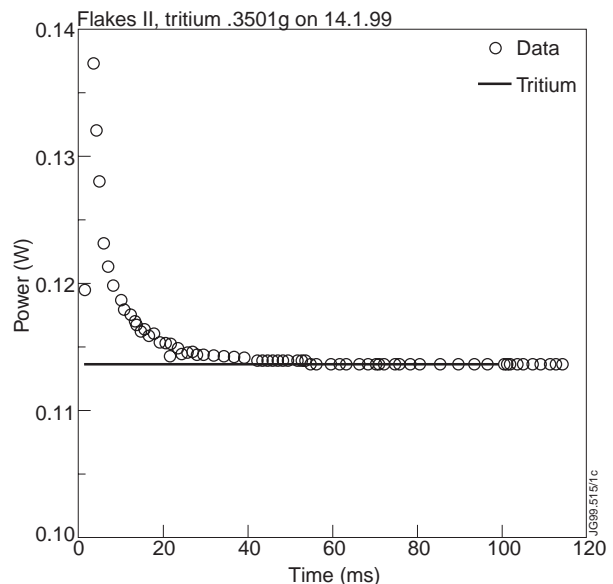


Fig.4: Calorimetry of the JET flakes

Table 1 shows the results of the measurements performed upon the flakes. The major uncertainty in the estimate of the tritium associated with the flakes/deposits in the inner divertor leg is the collection efficiency. Flake material can fall down behind the louvres and through holes in the divertor structure, and deposits can stay adhered and not be removed.

Table 1 Tritium content of flake samples.

Sample Number	Weight collected (g)	Tritium content (g)	T content (g)/(g)
1	56.8	0.17	3.10^{-3}
2	97.5	0.35	$3.6 \cdot 10^{-3}$

A programme is in place to measure the physical and chemical attributes of the tritium flakes. It should be noted that for the entire DTE1 experimental campaign the tritium used was 2.3% of the total gas fuelling. The tritium levels in the flakes would be much higher for a 50:50 D-T fuelling mixture. The weight of flakes and the tritium content gives a D:C ratio of 0.7, in agreement with IBA measurements.

5. DUST

Dust was collected from two places in the vessel. The first sample was taken from the divertor region. It was not possible to take this sample without contaminating it with pieces of flake material sucked in from adjacent regions. Hence the sample was sieved before analysis to remove particles >90 microns in size. The second sample was taken from components in the main chamber. The sample from the main chamber had a grey appearance and the sample from the divertor was black. Table 2 shows the results of chemical analysis of the two samples.

Table 2. Chemical composition (wt%) of the Dust material

Element	C	O	Al	Si	Fe	Cu	Mg	Be
Divertor (<90 μm)	87	11	0	0	0	0	0	2
Main chamber	15	51	2	17	2	1	9	2

It is clear that there is a significant difference between the two samples. The sample from the divertor is typical of deposited films but the sample in the main chamber is thought to result from material used for cable insulation. There appears to be little evidence for this material interacting with the plasma and being transported in to the divertor. The quantity of material collected is shown in Table 3. The dust from the divertor region has a quite high specific activity, which is thought to result from contamination with flake material which has become small enough to pass through the filter.

Table 3. Weight and Tritium content of dust material.

Sample	Weight collected	T content	Specific T content
Divertor (<90 μm)	0.1172g	43 GBq	368 GBq/g
Main Chamber	0.085g	3.33GBq	40GBq/g

Both samples were collected from areas of a few square metres. The quantity of dust material and the level of tritium in the dust material shows that dust represents only a very small fraction of the tritium inventory. This data is very much in line with data obtained during deuterium campaigns [6].

6. TILES

In each poloidal section of the MKIIa divertor there were 10 tiles. One divertor tile from each poloidal position was taken for analysis, together with 8 poloidal limiter tiles and 6 inner wall guard limiter tiles. All the tiles are relatively large components, the typical dimensions of the divertor tiles are 350mm x150mm x 40mm. Samples were cut from the tiles and then burnt to release the tritium. For some of the tiles, which potentially contain high tritium levels, calorimetry is also to be performed. Typically five samples were taken from each tile for T content analysis. Table 4 shows the different inventories associated with each tile type. Additional samples were also taken for analysis by other techniques. Based upon values obtained from parts of tiles not interacting directly with the plasma, an estimate was made of the tritium content of the whole first wall and is included in Table 4. The values obtained for tritium levels are a factor 2 lower than expected from pre-DTE1 deuterium levels.

Table 4. Tile locations against inventory

Tile types	T Inventory(g)
Outer divertor tiles	3.10^{-3}
Inner divertor tiles	7.10^{-3}
Base divertor tiles	4.10^{-2}
Inner wall tiles	8.10^{-3}
Poloidal limiter tiles	4.10^{-3}
Other first wall areas	5.10^{-2}
Total	$1.1.10^{-1}$

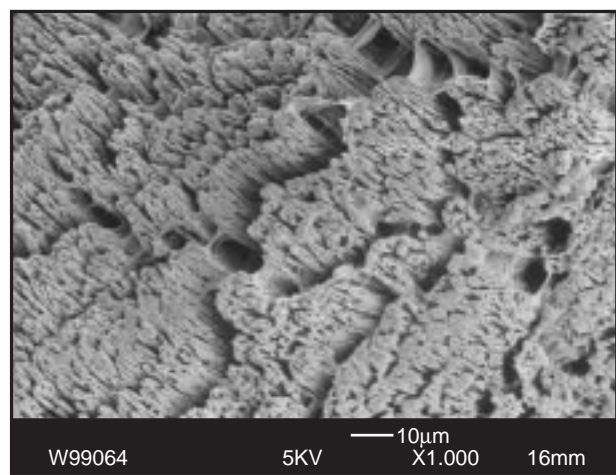
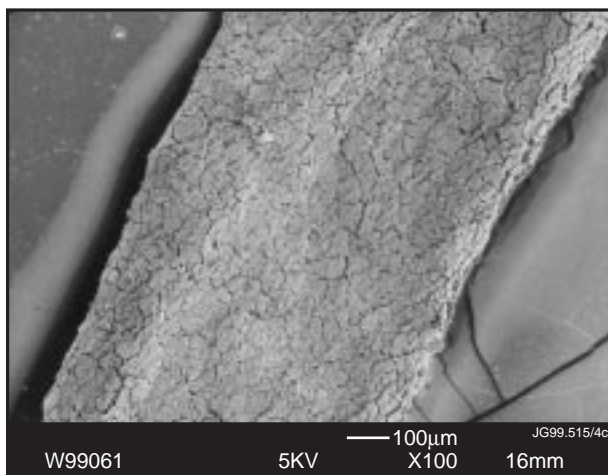


Fig.5: Deposits on tile 4

To assist in the understanding of erosion/deposition occurring on the plasma-facing surfaces, as opposed to the flake areas, water soluble adhesive tape was attached to the plasma-facing areas of the divertor tiles after DTE1. The tapes were then pulled off, the tape dissolved and the

deposits weighed and analysed. On tile 4 a deposit of 50 microns thickness was found, as shown in Fig 5. This result shows that tile 4 in the divertor inner leg (Fig. 2) is a region of heavy deposition. In comparison to the flakes (Fig.3), these deposits in areas that interact heavily with the plasma have a T:C ratio at least a factor 10 lower.

7. INVENTORY

The breakdown in tritium inventory at the end of the RTE shutdown is shown in Table 5. These figures show that there is a significant difference between what can be accounted for and the retained inventory. Possible explanations are that the collection efficiency for the flakes has been over-estimated and thus a higher level of tritium can be attributed to these flakes/deposits. The other explanation is that there is tritium elsewhere in the vessel, not found here. During a recent shutdown at JET a year after RTE, it was possible to perform an inspection of the sub-divertor volume with an endoscope. Large quantities of flakes have been found in this area. This material provides a possible explanation for the location of the missing inventory. However, the quantity of tritium cannot be known until these flakes are collected and analysed.

Table 5 Inventory results

Component	Quantity (g)
Out-gassing	2.0
Flakes/deposits	1.5
Dust	<0.01
Divertor tiles	~0.05
Vessel tiles	~0.06
permeation	0.1
Total	<3.8

8. DISCUSSION

The use of tritium in JET during DTE1 has allowed an accurate check to be performed upon the in-vessel inventory during operations as a function of time. The tritium was introduced directly into the vessel and promptly removed after cryopump regeneration, avoiding many of the complications that occurred during the PTE. As a result we have a clear picture of the tritium inventory in the vessel at the start of a RTE shutdown. The level, at 17%, was higher than expected from extrapolations from PTE.

Three types of material were measured to provide an indication of the location of tritium inside the vessel. It is clear from the results presented that the dust, collected by vacuuming, does not represent any significant tritium inventory.

Extrapolation of data from PTE post-mortem analysis predicts a tile inventory of 1g for the DTE1 experiment [7]. Since there was no divertor structure in JET during the PTE, this prediction was expected to represent the contribution from the main vessel chamber. However, the results shown in table 4 indicate that the levels in first wall components are an order of magnitude lower. This shows that the changed plasma configuration and vessel geometry have

produced a much reduced main vessel inventory. The remaining inventory must therefore come from the flakes/deposits or other sources. Tile inventory data obtained so far is lower (by a factor 2) than expected from previous deuterium post-mortem analyses using ion beam analysis. This may be a result of the hydrogenic material in these components reflecting the dynamic inventory, dominated by deuterium in the last months of operation.

The final physical component measured was the flakes. From the estimated collection efficiency and the amount of tritium measured in the collected flakes an estimate was made of the total T activity in the flakes.

The total tritium accounted for is thus 2.4g less than reported as missing from the AGHS. If the flake collection was not as effective as previously thought, there would need to be a similar quantity of flakes in inaccessible positions to account for the difference. Recent examination of the sub-divertor volume has shown significant quantities of flakes. By visual examination alone it is not possible to predict the exact quantity present. However, it is possible that there is sufficient material there to explain the missing tritium. Alternatively there needs to be an additional source of tritium inventory identified.

Tritium out-gassing was an important factor during the shutdown. Data from the PTE predicted a 20x lower out-gassing rate. Out-gassing measurements can be unreliable and the out-gassing rate does change rapidly at the beginning of the shutdown. However, over the period of the shutdown 2g of tritium was collected from out-gassing. Comparison with the PTE shows the only major difference to be the flake/deposit material. The D:C ratio measured in the flakes is 0.7-0.8. The flakes have been found to be chemically reactive and have high out-gassing rates. Flakes from the pre-DTE1 MKIIa campaign contain significant amounts of oxygen and hydrogen. Examples of carbon films containing high H:C ratios are also found in the plasma processing industry with H/C ratios of up to 1.2 [8]. These range from hydrocarbon films to amorphous H:C films with different types of bonding dependent upon deposition temperature and ion energy. If films are formed in the vessel with D:C ratios of > 1 which subsequently reduce on exposure to air to D:C levels seen in post-mortem analysis (0.7 to 0.8), this could be a major source of out-gassing. If the flakes are responsible for this missing tritium, then they could also be responsible for outgassing.

The mechanism for the formation of the deposits/flakes is still unclear, as is the asymmetry between the inner and outer divertor leg. The amount of material in the flakes/deposits and their location cannot be explained by classical sputtering mechanisms. A programme of work is now underway at JET to understand the phenomena of flake formation and their asymmetric deposition in the divertor. This work includes gas balance measurements, post-mortem analyses, in vessel diagnostics, and modelling work.

Gas balance measurements at JET for the Gas box campaign show gas retention at a level consistent with long-term gas retention (about 10%). These measurements were performed after

long periods of operation in one typical plasma configuration and have shown that it is unlikely that ELMs are responsible for enhanced retention, certainly for the Gas box. The Gas box divertor does, however, have significant differences to the MKIIa. The plasma strike point in the Gas box sits mainly on the vertical target rather than on the horizontal target as in the MKIIa. The tile temperatures are also different, as are the typical plasma parameters in front of the target. The Gas box divertor will be inspected in the forthcoming shutdown (June-July 1999) to discover whether or not similar flaking deposits exist.

Recent attempts to model the flakes using the DIVIMP code [9] were not successful without introducing extra parameters and altering other parameters beyond their normal range. A better match to the data was obtained by:

- (i) Introducing an additional flow in the SOL from outside to inside
- (ii) Enhancing the sputtering at the vessel walls.
- (iii) Introducing reflection for carbon atoms at the inner divertor target.

The physical basis for this modelling has been supported by recent work by von Keudell [10] that has shown that films similar to the JET films can be formed by the deposition of hydrocarbon radicals. These hydrocarbon radicals could be formed in the divertor and then transported to shadowed regions of the divertor. A variety of radicals exist, each with different sticking coefficients. The deposits/flakes could result from the accumulation of different species: physically sputtered material and different radicals. Some of the radicals have low sticking coefficients and could be responsible for the formation of films at remote parts of the vessel. These results could also offer an explanation for the fact that there is an additional unexplained source of inventory at JET. Such an area could be below the divertor structure. The existence of flakes of material below the divertor structure of ASDEX has recently been reported [11].

From the geometry of the divertor it appears that the source of carbon for the flakes must originate from tile 4. However, we have shown that significant deposition also occurs on tile 4. This indicates that carbon must come to tile 4 from other parts of the vessel, either from tile 5, nearer the strike point or from the JET vessel. This is also in line with the previous modelling assumptions.

The understanding of deposits/flake formation and the inventory situation can only be resolved by additional physical measurements. This requires extra diagnostics to be installed in JET and programme time for these measurements. One particular diagnostic of potential interest being developed is a system based upon a quartz crystal oscillator. Unfortunately the conditions in JET (high temperature bake-out, long cable paths, high operating temperature and temperature transients) do not allow such a device to be easily installed. If such a device can be developed (and recent progress is encouraging), it should be possible to resolve the deposition on a pulse by pulse basis to determine the conditions under which these deposits/flakes are formed, and the quantities involved.

9. CONCLUSIONS

Analysis of in-vessel components taken from the JET machine has been performed. In terms of the importance of the location of the retained tritium, the results have been in line with expectations.

Out-gassing from the vessel has been higher than expected.

Deposits and flakes in the shadowed area of the inner divertor leg contribute significantly to the retained inventory and out-gassing. Recent observation has shown that the total quantity of flakes is higher than originally expected and could be responsible for the tritium that is unaccounted for. It is also possible that the breakdown of the flakes could be responsible for the high out-gassing rates. However, the processes responsible for the flakes/deposits are not understood. Further understanding can only be gained by additional physical measurements.

Tritium retained in First Wall components has also been shown to be significantly lower than expected from extrapolations from the PTE.

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