



# Dust and flakes in the JET MkIIa divertor, analysis and results

A.T. Peacock <sup>a,\*</sup>, P. Andrew <sup>a</sup>, P. Cetier <sup>b</sup>, J.P. Coad <sup>a</sup>, G. Federici <sup>c</sup>, F.H. Hurd <sup>a</sup>,  
M.A. Pick <sup>a</sup>, C.H. Wu <sup>d</sup>

<sup>a</sup> JET Joint Undertaking, Abingdon, Oxfordshire OX14 3EA, UK

<sup>b</sup> CEA, Saclay, F-91191 Gif-sur-Yvette, France

<sup>c</sup> ITER JCT Garching, Boltzmannstrasse 2, 85748 Garching, Germany

<sup>d</sup> NET, Boltzmannstrasse 2, 85748 Garching, Germany

## Abstract

After operation of the JET machine with the MkIIa divertor, samples of dust and flakes/deposits have been removed from the divertor region for analysis. Flakes/deposits up to 40  $\mu\text{m}$  thick have been found in the inner leg of the divertor. The flakes/deposits formed on cold surfaces shadowed from the plasma. The flakes were found to be mostly carbon saturated with deuterium to a D/C ratio of 0.4. Analysis of the material removed has shown that it contains significant fractions ( $\sim 4\%$ ) of the tritium (produced by D–D reactions) and deuterium introduced in the JET machine during this period of operation. Dust material has also been collected. Vacuum cleaning collected very little material but a significant quantity of material was collected by smears. The analysis of this smeared material has shown it to be of a very similar chemical composition to that of flakes but to contain a significantly lower inventory of tritium. The dust has an average particle size of 27  $\mu\text{m}$  and a specific surface area of 4  $\text{m}^2/\text{g}$ . The vast majority of this material is associated with co-deposited layers in the divertor region. The dust is on average present at a level of 120  $\mu\text{g}/\text{cm}^2$ . © 1999 JET Joint Undertaking, published by Elsevier Science B.V. All rights reserved.

**Keywords:** Beryllium; Carbon; Carbon deposition; Carbon erosion; Co-deposition; Deuterium inventory; Dust; Hydrogenic chemical erosion; JET; Inventory; Material erosion; Nuclear reaction analysis; Surface analysis; Tritium inventory; Tritium retention

## 1. Introduction

A number of studies have been performed in the past to look at the level of dust present in the JET tokamak [1]. These studies collected and analysed both airborne and surface based material. It was considered to be worthwhile to revisit this issue as a result of the changes to JET machine.

There are a number of differences between the present study and the original study. Firstly, the JET machine now has a divertor structure, not present during the original investigations. Secondly, the appearance of flake-like material in the divertor region has a great

impact upon the divertor hydrogenic inventory, and thirdly, the original study did not include particle size analysis, specific surface area measurements or SEM investigations of the dust.

There are three different ways of defining dust: the material that becomes airborne when the vessel is vented, the material that can be collected with a vacuum cleaner, or the material that can be collected by taking a smear sample from the tile surfaces. Resuspended dust [1] has been collected at JET previously and is not studied here. This paper, therefore, focuses upon dust collected by the other two techniques.

A new type of material, not seen previously at JET, has been found in the inner leg of the JET divertor. This material takes the form of a carbonaceous film deposited in cold areas of the divertor shadowed from the plasma. In some areas the films spall off resulting in flakes several

\* Corresponding author. Tel.: +44-1235 464 911; fax: +44-1235 465 373; e-mail: atp@jet.uk

millimetres long on the divertor floor. Flakes of material have previously been seen at JET in the scrape-off regions of plasma facing components [2] but not to this level in shadowed regions.

The flakes/deposited material are reported here as they could potentially be a major source of tritium in tokamaks fuelled with tritium [3,4]. Analysis of the flakes allows estimates of tritium uptake to be made. The paper details the conditions under which the flakes form, in order that efforts may be made to remove tritium from these films or to stop their formation in the first place.

## 2. Experimental

The MkIIa divertor started operation in the JET machine in April 1996. The divertor, shown in Fig. 1, consists of an inconel support structure containing an arrangement of large carbon–carbon fibre composite tiles attached to inconel tile carriers [5]. The JET machine then ran approximately for 2000 pulses between April 1996 and October 1996 without manned entry. During this period the gas input into the machine was  $9 \times 10^{25}$  deuterium atoms by both gas fuelling and neutral beam injection (NBI). The machine was then vented and manned access established. Samples of dust were taken from the divertor and surrounding regions during the first manned entry into the machine, three days after the end of operations.

Two methods of sample collection were used during this first entry. Firstly, a cyclone vacuum cleaner was used to collect debris or loosely adhered material down to  $2 \mu\text{m}$  in size. An area of approximately  $1 \text{ m}^2$  was vacuumed.

Secondly, a moist cloth was placed against the tile surfaces and rotated to collect finely divided particles present upon the tile surfaces. The cloth samples were mounted on a tool specially designed to apply a constant

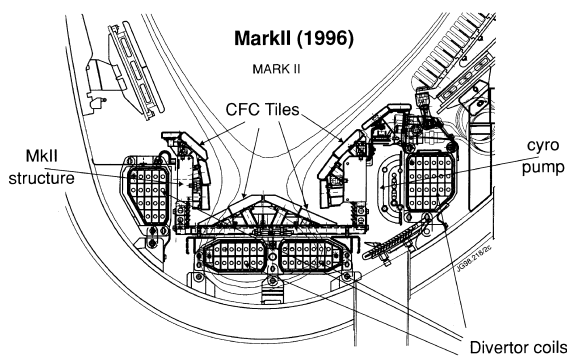


Fig. 1. Schematic view of the lower half of the JET machine with the MkII divertor.

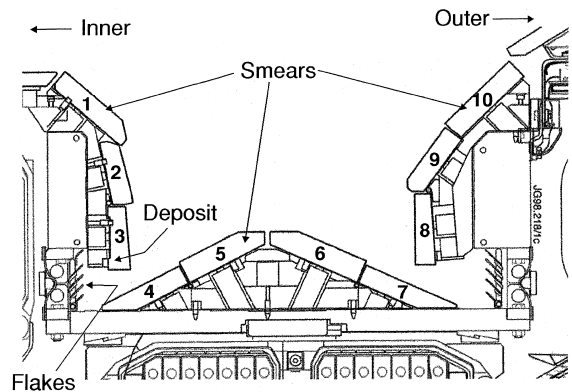


Fig. 2. Schematic view of the MkII divertor showing where samples were taken.

force whilst giving a rotating motion to the cloth. This method had previously been tested using a clean JET tile coated with different levels of dust. The polyester cloth has a very low metallic background level. Twelve positions were sampled and at each position five samples (60 samples in total) were taken from the adjacent positions. Each of the five different samples was used for a different type of analysis.

The flake material was not visible during the first entry into the vessel when the dust was collected and only became visible when an inner tile carrier was removed, several weeks into the shutdown. The flake material was easily seen and samples of the flake material were collected manually from the bottom of the divertor structure. Samples of the deposited material were also removed from the underside of the bottom tile on the inner carrier. Fig. 2 shows a poloidal section of the JET divertor; the positions where the different samples were taken are indicated.

The dust samples were analysed for their tritium activity (burning sample and measuring water by liquid scintillation), gamma activity, particle size (filtering samples through Isopore membranes), specific surface area (B.E.T.), morphology (SEM), carbon content, beryllium content and for the main elements of inconel 600 (the material of the JET vessel structure). The flakes/deposits were analysed for tritium activity, chemical content and thickness by SEM.

## 3. Results

### 3.1. Cloth sample results

Fig. 3 shows the total quantity of material present in the different areas sampled. In the divertor, samples were taken from the target plate tiles both from regions that were exposed to plasma field lines, and from areas that,

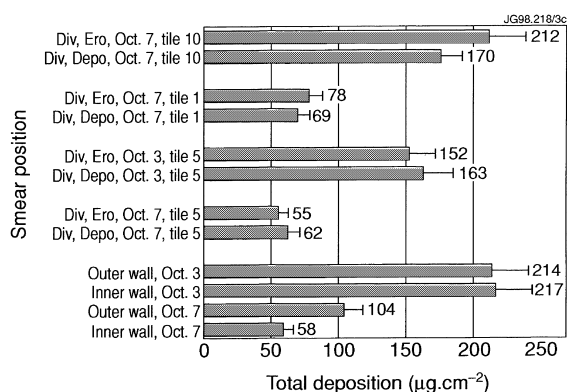


Fig. 3. Total quantity of dust as function of location.

although exposed to the plasma, were shadowed from the field lines. The majority of the material present in the samples was carbon. The two locations on the vessel outer wall which showed much higher than average beryllium levels were near to the Beryllium evaporators. Excluding these two areas the rest of the Be content in the samples are, in tokamak terms, remarkably uniform at  $1 \pm 0.5 \mu\text{g}/\text{cm}^2$ . The value of Ni in the samples varied over a much wider range from essentially zero to, at one location, 40% by weight of the sample. The high nickel level may have been the result of a localised splat of inconel. Excluding the high level Ni and Be samples, the average concentration on the smears is  $120 \mu\text{g}/\text{cm}^2$  of which 97% is carbon, 1% Be and 2% metals by weight. This represents a carbon layer over half a micron thick on the surface of the components.

The radioactivity present in the samples consists mainly of tritium and  $^7\text{Be}$ . The tritium levels range from 8 to  $460 \text{ Bq}/\text{cm}^2$ .  $^7\text{Be}$ , produced by photo-activation, was present at the level of  $1 \text{ Bq}/\text{cm}^2$ .

There appears to be no significant difference between the different locations measured based upon the data collected, either from the carbon levels, tritium levels, Be levels or metal levels. Many more samples would need to be collected to establish any possible correlation with location in the tokamak.

The material present on the cloth was recovered and filtered through membranes with successively smaller pore size. The results are presented in Fig. 4 as the cumulative mass distribution against the logarithm of the particle diameter. The median diameter of the particles collected was  $D=27 \mu\text{m}$ . This figure is obtained from the measurements taken for three different size ranges.

The specific surface area, measured for the largest size fraction is  $4 \pm 2 \text{ m}^2/\text{g}$ .

SEM micrographs are shown in Fig. 5 for the particles in the largest size range. It was noticed that particles prepared dry for analysis tended to form agglomerates consisting of several dozen to several hundred particles.

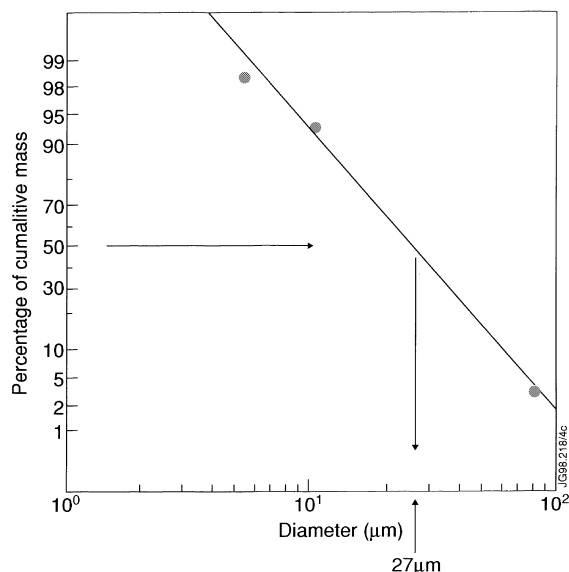


Fig. 4. Dust particle size distribution.

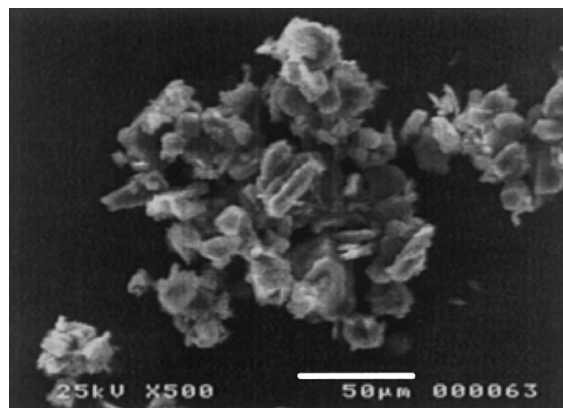


Fig. 5. Scanning electron image of dust.

Nominally the particles are spherical but some oblong and polygonal particles were also seen.

### 3.2. Debris from the divertor region

The attempt to collect debris from the divertor region with a cyclone vacuum cleaner collected less than 1 mg of material. This was judged as too little to analyse.

### 3.3. Flakes/deposits

Flakes of material with lateral dimensions of several millimetres were found upon the base of the divertor structure. These self-supporting flakes had apparently fallen off the water cooled louvres on the inner leg of the

Table 1  
Comparison of the chemical composition of flakes and dust

Elements present	Flakes (wt%)	Dust (wt%)
Carbon	99	97
Beryllium	0.6	1
Metals (Fe, Ni, Cr)	0.5	2<

divertor. Deposits of similar materials were also found on the bottom of tile 3 (Fig. 2) which had remained attached to the tile.

The flakes/deposits are found only in the inner leg of the divertor and come from material which is deposited on those surfaces which are shadowed from both conducted and radiated power from the main plasma volume. The temperature of these surfaces are range from 25°C (the water cooled support structure) to 150°C (tiles not directly facing the plasma).

Ion beam analysis showed that the deuterium content of the flake material was high with a deuterium/carbon ratio of 0.4. The chemical composition of the flake material is shown in Table 1 compared with the average chemical composition of the dust. This data excludes any deuterium present.

The thickness of the flakes, measured at a number of positions both by SEM and optical microscopy, was found to be of the order of 40 µm. Some thinner flakes were found but an SEM micrograph of the flakes, Fig. 6, shows a possible explanation for this observation. The flakes have a laminated structure with variations in density as a function of depth through the film. Fig. 6 shows the deposit delaminating resulting in thinner films. Optical microscopy and SEM observation have shown 40 µm films in samples taken in several different places.

The poloidal and toroidal extent of the areas where the flakes/deposits were found are well known (additional sources of flakes have subsequently been found, see Section 4). Combining the thickness of the film and the known ratio of deuterium to carbon gives a quantity of deuterium present in the flakes of the order of 4% of the total deuterium fuelling for this period.

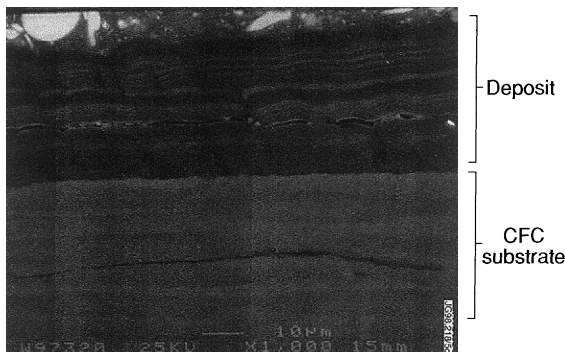


Fig. 6. Scanning electron image of deposit.

The tritium activity of the flakes was measured to be of the order of  $9 \pm 75\%$  MBq/g. Using this figure and the quantity of material mentioned above this corresponds to  $4 \pm 3\%$  of the total quantity of tritium produced by D–D reactions during the campaign prior to the removal of the flakes.

Thus, a significant fraction of the deuterium and tritium used in the machine is present in the flakes/deposits. This was also confirmed by independent results of the tritium content of the flake material [6]. The results presented, however, are post-mortem analyses of the flakes several months after they were removed from the torus. SIMS analysis shows the flakes/deposits contain significant amounts of protium in addition to deuterium, despite the fact that little protium is used in the operation of JET. This can only be explained by postulating that the films have a hydrogen isotope to carbon ratio in excess of 0.4. Films exist in which this ratio is of the order of 0.8–1.0. Thus, in the JET vessel, during deuterium campaigns, films may be formed with deuterium amounts of the order 0.8–1.0 D/C and that upon exposure to air the deuterium isotope exchanges with water to reduce the deuterium amount to the level observed. Further evidence for this is that after the DTE1 at JET components covered with these films had very high tritium off-gassing rates [4].

### 3.4. Comparison of the flakes and dust

Table 2 compares the divertor inventories present in the dust and flakes. The divertor surface area is nominally 25 m<sup>2</sup>. Table 2 shows the quantity of tritium in the flakes/deposits is much higher than in the dust, despite the chemical composition being very similar (Table 2). On a concentration basis the flakes contain 10 times more tritium than the collected dust. The deuterium inventory present in the divertor (as measured by ion beam analysis) is normally thought to reside in co-deposited layers. Assuming that the ratio of tritium to deuterium is the same in the dust, co-deposited layers and the flakes it is possible to extrapolate the above results to give a figure for the amount of deuterium in the dust. This represents approximately a third of the inventory of deuterium measured in the divertor by ion beam analysis.

The deuterium inventory in the JET divertor was calculated from detailed ion beam analysis of the divertor tiles.

## 4. Discussion

Vacuum cleaning reveals that there is a little loose dust/debris on the surfaces of the divertor tiles (<1 mg/m<sup>2</sup>). This is consistent with the visual appearance of the tiles and the observation of very little first wall damage.

Table 2  
Comparison of the inventories present in the flakes/deposits and dust

Material	Carbon (g)	Deuterium (atoms)	Metals (g)	Tritium (Bq)	Be (g)
Dust	30	—	0.6	$4 \times 10^7$	0.3
Flakes/deposits	180	$4 \times 10^{24}$	1	$1.6 \times 10^9$	1

However, significant quantities of material can be removed by smearing. Regions may exist where dust has settled that are inaccessible to the vacuum cleaner.

The MkIIa divertor at JET has areas of deposition and erosion similar to other divertors [7]. From optical examination there is an inner and outer strike zone from the most frequently used plasma configurations which has a shiny appearance. Other areas, especially the inner regions of the divertor, show a matt black finish indicative of heavy deposition.

The quantities of dust present seen here can be adequately explained by two mechanisms. Firstly, mechanical damage of in-vessel components as a result of interactions with the plasma, broken tiles, etc. For the period studied there was a little of this type of damage. The other source of dust appears to be the break-up of the co-deposited layers which are formed on the divertor tiles.

Taking smear samples removed material in all the areas tested. The quantity of material collected upon smears varies greatly and does not appear to be linked to any visible features on the tiles. For example, the strike points appear clean but yield average amounts of material, though this might indicate that these strike points were not the strike points for the last series of plasmas.

Results from the neutral beam (NB) test bed at JET suggest a possible explanation for the smear results. During testing of the tiles for MkIIa it was discovered that if a tile was incorrectly cleaned then an anomalously high surface temperature was measured. This was linked to the instantaneous vapourisation of dust particles on the surface of the tiles [8]. Clean tiles did not initially show this effect but after a series of pulses the dust became apparent again, probably as a result of damage to the tile surface by the neutral beam [8]. Continued bombardment by ions and charge exchange neutrals could explain the presence of dust in all areas exposed to the plasma.

The flakes/deposits represent a significant inventory of hydrogenic material. Given the fact that the films are saturated the main determining factor in the inventory is the presence of carbon. Co-deposition of carbon has been observed before at JET and layers of similar thicknesses have been seen [2]. The difference here is that these flakes do not appear to have been formed by a process of sputtering followed by reionisation and then prompt redeposition as is seen in co-deposited films. The process of formation of the flakes/deposits appears to be

by deposition of neutral particles. Observations show that there are shadows of the deposited material. In addition flaked material has also been seen on the back of the louvres during the recent RTE shutdown when improved visual access was available. These observations indicate a line of sight process but the only explanation for the material behind the louvres consistent with this is that the carbon atoms striking the louvres have a finite chance of reflection. It has not been possible to quantify the level of material on the front of the louvres compared with the back by direct measurement.

The design of the new divertor at JET has regions where these deposits can form, which are cold and not exposed to the plasma. Similar processes may have previously occurred in JET divertors but the deposits will have been exposed to the plasma and in warmer areas and hence did not contain significant hydrogenic inventories.

The apparent source of the carbon material, consistent with the shadows, is the innermost tile on the base of the divertor. This is normally in the outer regions of the scrape-off layer for most JET plasmas and an area of net deposition. The mechanism for the transport of carbon to the louvres is not known. The deuterium ion flux to the inner target for this campaign measured by Langmuir probes is  $2.5 \times 10^{26}$  [9]. This would require a sputtering coefficient of the order of 0.04 to produce the required quantity of carbon assuming 100% transmission of the carbon from the target to the louvres. In addition the ion flux is mainly to tile 5 or the top most half of tile 4, the main plasma strike zone. The solid angle subtended from tile 5 to the louvres is small. Thus, it appears that ion sputtering cannot be the sole mechanism.

One possible mechanism is vapourisation of the dusty deposited material by the high conducted power in the divertor especially during ELMs. This would be analogous to the observations seen in the NB test bed. In fact, IR cameras viewing this area show higher temperature readings than would be expected from the input power from a homogeneous target suggesting that the surface material is not well connected to the bulk [10]. Another explanation could be neutral particle sputtering especially during detached plasma periods: the number of neutrals impinging the inner strike zone is considerably greater than the number of ions, so that the net sputtering coefficient from the combined fluxes would be correspondingly lower. Further investigation is needed into these mechanisms. Any mechanism would also need

to explain the asymmetry between the inner and outer legs of the divertor.

The deposits/flakes contain an order of magnitude more deuterium than the divertor tiles as measured by ion beam analysis. The main reason for this high inventory appears to be that these deposits are not subject to high temperatures and are hence saturated with deuterium.

The inventory previously calculated does not include the amount of deposit/flake upon the back of the louvres. An estimate of the quantity is of the order of 25% of the inventory already reported above and comes from the detailed remote camera inspections carried out during the current remote tile exchange (RTE) to modify the divertor configuration in JET.

Since these films contain significant amounts of tritium next step machines will need to incorporate technologies to stop the deposits from forming or techniques will be required to facilitate their removal. One possible solution would be a change in geometry to ensure the deposits were exposed to the plasma.

## 5. Conclusions

The MkIIa divertor at JET has been shown to be reasonably clean of very loose dust. However, a layer of loosely adherent material exists on all areas of the divertor when smears are taken. For the divertor this constitutes a significant quantity of carbon material. However, the dust samples are only a fraction of the loose material on the tile.

The flake/deposited material represents a significant new source of hydrogenic material which could constitute a significant problem for next step machines. The two factors which lead to the high inventories are the fact that the deposits are mostly carbon and, because they are formed at low temperature, saturated with hydrogenic species. Taking into account the deposits on the back of the louvres and the fact that the deuterium measured is only a fraction of the inventory at the time the machine is vented, then the flakes/deposits could contain up to 10% of the deuterium and tritium that had

been introduced/produced in the machine for this campaign.

The material deposited on louvres and shadowed tiles has a dense structure. The material deposited on carbon surfaces is adherent but the material deposited on the cool louvres tends to spall off forming flakes. The spallation probably occurred for films (of the order of  $\sim 40$   $\mu\text{m}$  thick) when the torus was vented to air; if films continue to grow thicker the internal stress in the deposit may cause spallation during normal operation.

The source of the flakes/deposits is probably the co-deposited material on tile 4. The region of the JET vessel from which this material originates is not known, nor is the mechanism of transfer from tile 4. The process is line of sight but does not appear to be ion sputtering. Vapourisation of poorly adhered material by ELMs or neutral particle sputtering appear to be possible explanations. The fact that the chemical composition of the flakes is similar to the dust favours the former explanation.

## References

- [1] J. Charuau, Y. Belot, Ph. Cetier, L. Drezet, L. Grivaud, A.T. Peacock, C.H. Wu, Characterisation of erosion dust and tritiated products inside the JET vessel after the first tritium experiment, Proceedings of the 17th SOFT, vol. 2, Rome, 14–18 September 1992, pp. 1700–1703.
- [2] G.M. McCracken et al., J. Nucl. Mater. 162–164 (1989) 356.
- [3] G. Federici et al., these Proceedings.
- [4] P. Andrew et al., these Proceedings.
- [5] H. Altmann et al., Design of the MkII divertor with large carbon-fibre composite tiles, Proceedings of the 18th SOFT, vol. 1, Karlsruhe, 1994, pp. 275–278.
- [6] E. Kaiser et al., JET carbon flakes: preliminary results from first studies, Forschungszentrum Karlsruhe (unpublished report).
- [7] J.P. Coad, M. Rubel, C.H. Wu, J. Nucl. Mater. 241–243 (1997) 408.
- [8] D. Ciric et al., Proceedings of the 18th SOFT, vol. 1, Karlsruhe, 1994, pp. 391–394.
- [9] R. Monk (private communication).
- [10] S. Clement et al., these Proceedings.