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Dust and Flakes in the JET MKIIa Divertor, Analysis and Results

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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 40µm 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 (~ 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 the flakes but to contain a significantly lower inventory of tritium. The dust has an average particle size of 27µm and a specific surface area of 4 m^2/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 120µg/cm^2.

1.0 INTRODUCTION

A number of studies have been performed in the past to look at the level of dust present in the JET tokamak [1,2,3]. These studies collected and analysed both airborne and surface based material. The original studies were under the framework of the safety considerations for NET. The latest studies have been performed to supply data for safety considerations at ITER. It was considered to be worthwhile to revisit this issue as a result of changes to the 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, which was 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 [3] has been collected at JET previously and there appears to be no difference between the machine then and now that would have a significant effect upon the amount of airborne material generated upon venting. 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 millimetres long on the divertor floor. Flakes of material have previously been seen at JET in the scrape-off regions of plasma facing components [4] 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 [5,6]. Analyses of the flakes allows estimates of tritium uptake to be made. The paper also 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.

2.0 EXPERIMENTAL

The MkIIa divertor was installed in the JET machine during the shutdown period that ended 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 [7]. Following the installation of the divertor, the JET machine then ran for approximately 2000 pulses between April and October 1996 without manned entry. During this period the gas input into the machine was 9.10^{25} deuterium atoms by both gas fuelling and neutral beam injection (NBI). At the end of this operational period the machine was 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.

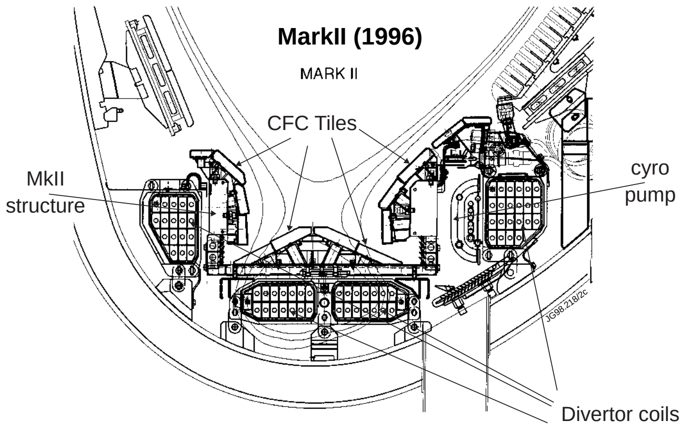

Fig 1 Picture of machine with divertor

Two methods of sample collection were used during this first entry. Firstly, to collect debris and other dust material unattached or only lightly attached to the tiles, a cyclone vacuum cleaner was used capable of collecting material down to 2μm in size. An area of approx $1m^2$ was vacuumed.

The second type of sample collection used a moist cloth placed against the tile surface 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 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 cloth, made of polyester, had a very low metallic background level. Twelve positions were chosen inside the vessel for this type of sampling and at each position five samples (60 samples in total) were taken from adjacent positions. Each of the five different samples was used for a different type of analysis.

The flake material was found in the inner leg of the divertor after the dust samples had been collected. This material was not visible during the first entry into the vessel when the dust was collected. The flake material only became visible when an inner tile carrier was removed.

This occurred several weeks into the shutdown. The flake material was easily seen and no special equipment was necessary for its removal. 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. Figure 2 shows a poloidal section of the JET divertor; the positions where the different samples were taken are indicated.

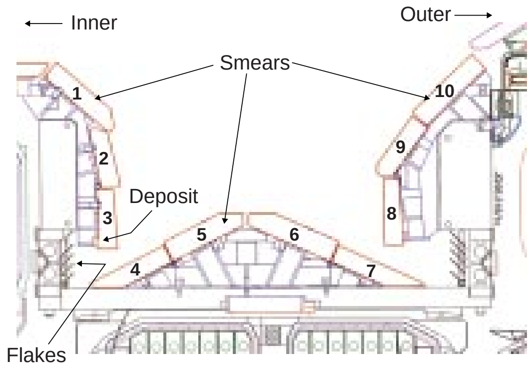

Fig 2 Picture of where samples were taken

The dust samples were analysed for their tritium activity, gamma activity, particle size, specific surface area, morphology, 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, thickness and by SEM.

3.0 RESULTS

3.1 Cloth sample results

Figure 3 shows the total quantity of material present in the different areas sampled. In the divertor region the samples were taken from the target plate tiles from both regions of the tiles that were exposed to plasma field lines, and from areas that, although exposed to the plasma, were shadowed from the field lines. The majority of the material present in the samples was carbon. There were two locations on the vessel outer wall which showed much higher than average beryllium levels. These

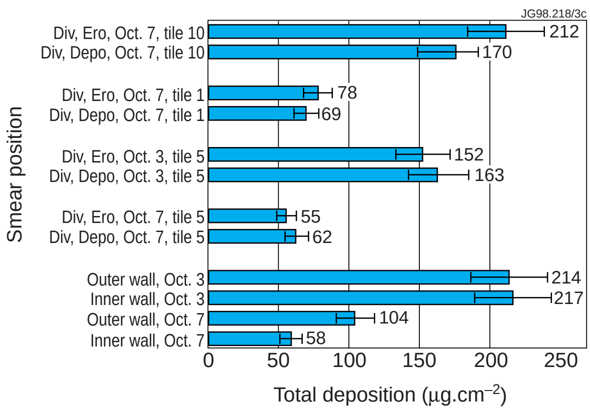

Fig 3 Total quantity of dust as function of location

areas were near to the Beryllium evaporators which have been shown to be associated with high Be surface layers [8]. Excluding these two areas the rest of the Be content in the samples are, in tokamak terms, remarkably uniform at 1±0.5µg/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 Ni level and the Be samples on the outer wall the average concentration on the smears is 120µg/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}Be. The tritium levels range from 8 Bq/cm^2 to 460 Bq/cm^2. ^{7}Be was present at the level of 1 Bq/cm^2 at the time the samples were taken three days after the end of operation (inferred by correcting the measurements for half life of the activity decay).

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. From this graph we find that the median diameter and the geometric standard deviation of the particles collected are as follows:

$$D = 27\text{mm and } s_g = 2$$

This figure is obtained from the measurements taken for three different size ranges.

The specific surface area could only be measured for the largest size fraction and is as follows:

$$4\ \text{m}^2/\text{g} \pm 50\%$$

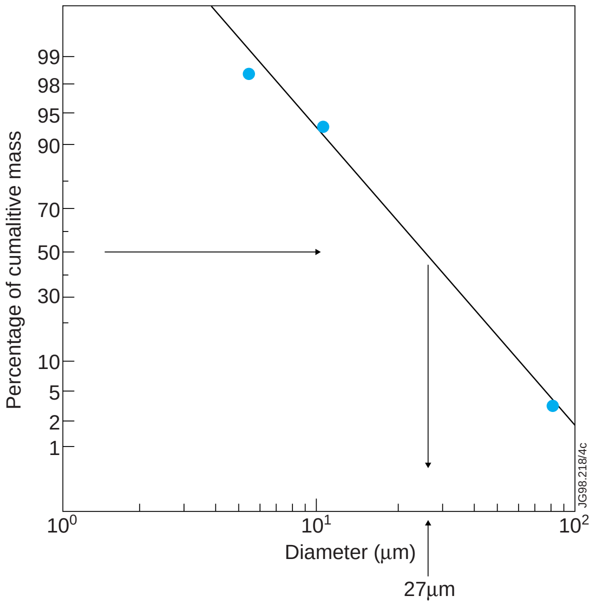

Fig 4 Dust particle size distribution

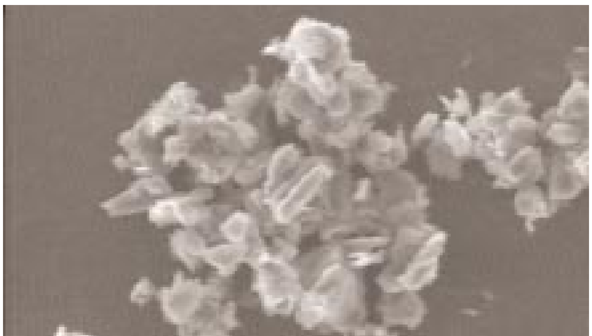

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Fig 5 Scanning electron image of dust

Scanning electron micrographs are shown in fig 5 for the particles in the largest size range. It was noticed that particles that were prepared dry for analysis tended to form agglomerates consisting of several dozen to several hundred particles. Nominally the particles are spherical but some oblong and polygonal particles were also seen.

3.2 Debris from the divertor region

Attempts were made to collect debris from the divertor region with a cyclone vacuum cleaner. In total a quantity of less than 1 milligram of material was collected. This was judged too little to analyse.

3.3 Flakes

Flakes of material with lateral dimensions of several millimetres were found upon the base of the divertor structure. These flakes which were self supporting had apparently fallen off the inner water cooled louvres on the inner leg of the divertor. Deposits were also found on the bottom of tile 3 (see Fig. 2) which appeared to be the same material as the flakes but had remained attached to the tile.

The flake material is found only in the inner leg of the divertor and is deposited in those regions which are shadowed from both conducted and radiated power from the main plasma volume. The temperature of the surfaces upon which the deposits are found 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 flakes material was high with a deuterium/carbon ratio of 0.4. The chemical composition of the flake material is shown in Table 1 and is compared with average chemical composition of the dust. This table excludes any deuterium present.

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

Table 1 Comparison of the chemical composition of flakes and dust.

The thickness of the flakes was measured at a number of positions both by SEM and optical microscopy. The films were found to be of the order of 40 μm thick. Some flakes were found which were thinner than this 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. Figure 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.

Fig 6 Scanning electron image of deposit

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

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

Thus it appears clear that a significant fraction of the deuterium and tritium used in the machine is present in the flakes. This has also been confirmed by independent results of the tritium content of the flake material [9]. The results presented here, however, are **post-mortem** analyses of the flakes several months after they were removed from the torus. SIMS analysis has also shown they 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 to 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[6].

3.4 Comparison of the flakes and dust

As shown above the chemical composition of the flake material is very similar to the dust. Table 2 compares the divertor inventories present in the dust and flakes. The divertor surface area is nominally $25m^2$.

Material	Carbon	Deuterium	Metals	Tritium	Be
Dust	30g	–	0.6g	4 107Bq	0.3g
Flakes/ Deposits	180g	$4\ 10^{24}$	1g	$1.6\ 10^{9}$Bq	1g

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

As can be seen from the table the quantity of tritium in the flakes/deposits is much higher than in the dust. 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 approx 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 tiles in this area. The results are shown in fig.7.

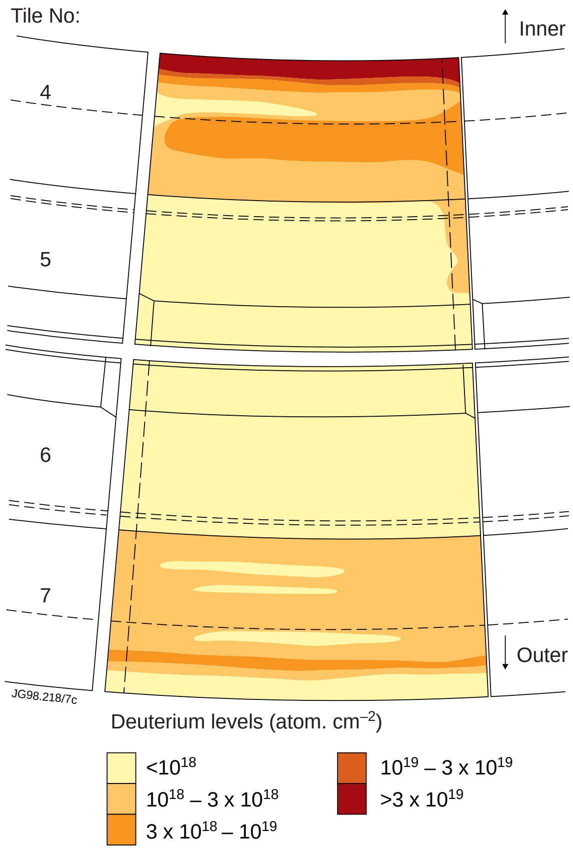

Fig 7 Deuterium map of the MkIIa base tiles

4.0 DISCUSSION

Vacuum cleaning using a cyclone device reveals that there is little loose dust/debris on the surfaces of the divertor tiles (<1mg/m^2). This is consistent with the visual appearance of the tiles and the observation that the period of operation prior to the vacuuming had shown very little first wall damage. There does, however, appear to be a significant quantity of material on the surface of the tiles that can be removed by smearing. There may also exist regions where dust may have settled that were inaccessible to the vacuum cleaner.

The MkIIa divertor at JET has areas of deposition and erosion similar to other divertors [10]. From optical examination there is an inner and outer strike zone from the most frequently used plasma configurations which has a shiny appearance (see also fig.7). Other areas, especially the inner regions of the divertor, show a matt black finish indicative of heavy deposition.

The quantities of dust present seen as a result of this investigation can be adequately explained by two mechanisms. The first is mechanical damage of in-vessel components as a result of interactions with the plasma, broken tiles, etc. For the period studied there appears to have been 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 areas that are nominally near the strike points and may therefore be expected to be relatively clean yield quite 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 Testbed at JET suggest a possible explanation for the smear results. During testing of the tiles for MkII it was discovered that if a tile was incorrectly cleaned then an anomalously high surface temperature was measured. This was linked to the instantaneous vaporisation of dust particles on the surface of the tiles [11]. 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 [11]. Continued bombardment by ion and charge exchange neutrals could explain the presence of dust in all areas exposed to the plasma.

The flake material represents a significant inventory of hydrogenic material. Given the fact the films are saturated the main determining factor in the inventory is the presence of carbon. Co-depositon of carbon has been observed before at JET and layers of similar thicknesses have been seen [4]. The difference here is that these flakes do not appear to have been formed by a process of sputtering followed by re-ionisation and then prompt re-deposition as is seen in co-deposited films. The process for the formation of the flakes appears to be by deposition of neutral particles on to the louvres.

Observations of the deposits have shown 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 better access to the louvres was obtained. The shadows suggest that this is 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 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 is an area of net deposition. The mechanism for the transport of carbon to the louvres is not known. The ion flux to the inner target for this campaign measured by Langmuir probes is $2.5.10^{26}$ deuterons[12]. 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 topmost 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 mechanism.

One mechanism which might explain the carbon on the louvres is vaporisation 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[13]. Another explanation appears to 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 [14].

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,

5.0 CONCLUSIONS

The MkIIa divertor at JET has been shown to be reasonably clean of very loose dust such as can be removed by vacuum cleaning. 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 sample 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 ~ 40µm 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. Vaporisation 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, P Casette, and M Diori. Sampling and Analysis of Erosion products inside the tokamak "JET". CEA report SPIN/10.1.1.3.4, September 1987.
2) J Charuau. Analyses of Erosion and Activation Products Collected in JET vessel during October 1989 Exercise. CEA report SPIN/10.1.1.3.4, December 1990.
3) 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. 17th SOFT, Rome, 14-18th September 1992, pp 1700-1703.
4) G M McCracken, D H J Goodall, P C Stangeby, J P Coad, J Roth, B Denne and R Behrisch, JNM 162-164 (1989) 356
5) G Federici et al, Implications of In-Vessel tritium retention/removal for ITER. This conference.
6) P Andrew at al, Tritium Recycling and Retention in JET. This conference.
7) H Altmann et al, Design of the MkII divertor with large carbon-fibre composite tiles, 18th SOFT, Karlsruhe, 1994
8) M Mayer et al, Wall Erosion and Material Transport to the Mark I Divertor of JET. This conference.
9) E Kaiser et al, JET carbon flakes: Preliminary from first studies. Forschungzentrum Karlsruhe, unpublished report.
10) J P Coad, M Rubel and CH Wu, JNM 241-243 (1997) 408
11) D Ciric et al, 18th SOFT, Karlsruhe, 1994
12) R Monk, private comummuniction.
13) S Clement et al, Power Deposition in the JET Divertor During ELMs. This conference.
14) A Rolfe et al, The Exchange of JET Divertor Modules using Remote Handling Techniques. To be published in the proceedings of the IEEE Conference on Robotics and Automation, May 1998