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Characterisation of dust and flakes generated in Fusion machines after DD and DT operations

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Abstract

Flakes and dust are generated by co-deposition in all experimental fusion machines having a carbon first wall. At JET a very large fraction of tritium is retained in form of flakes in the vicinity of the water cooled louvres adjacent to the inner divertor and in the sub-divertor zones of the machine.

The BET specific surface area performed on flakes collected after a DD campaign confirm a low value of at about 7 m^2 g^{-1}. The average tritium activity of flakes after DTE1 was estimated to be (1.17 ± 0.1) TBq g^{-1}.

At the end of RTE approximately 3.0 g tritium was not accounted for and must remain in the vessel as flakes. If they have the same content of tritium as the flakes removed up to now then a further (950 ± 80 g)of flakes must still be present in the vessel.

Charakterisierung von in Fusionsanlagen während DD- und DT-Operationen erzeugten Flocken und Staub

Zusammenfassung

Wasserstoff enthaltende Schichten sowie Flocken und Staub werden in allen größeren Fusionsanlagen beobachtet, speziell wenn die erste Wand oder Teile des Divertors aus Kohlenstoff bestehen. Innerhalb des Vakuumbehälters des JET Tokamaks wurden bis zu 40% der injizierten Tritiummenge primär in diesen Materialien zumindest temporär eingeschlossen, die durch Koabscheidung von Graphit und Wasserstoff entstehen. Die wesentlich Depositionszone für die Schichten waren die wassergekühlten Louvres neben dem inneren Divertor, von wo aus lose Schichten dann in die Sub-Divertorzonen fallen konnten.

Messungen der spezifischen Oberfläche (BET) von nach einer DD-Kampagne gesammelten Flocken ergeben einen geringen Wert von etwa 7 m^2g^{-1}. Die durchschnittliche spezifische Tritiumaktivität der Flocken wurde nach dem Ende der DTE1 Phase zu (1,17 ± 0,1) TBq g^{-1} bestimmt.

Nach Abschluss des Austausches der Ziegeln in der ersten Wand fehlten in der Tritiumbilanz etwa 3,0 g. Es wird vermutet, dass diese Tritiummenge in den Flocken enthalten sind. Entspricht deren Tritiumgehalt dem der bisher entnommenen, so müssen sich noch (950 ± 80) g Flocken innerhalb des Vakuumgefäßes befinden.

Contents

1. Introduction

Three distinct mechanisms have been identified in current tokamak experiments which are responsible for in-vessel sequestration of tritium: i) surface saturation of the plasma facing first wall components, ii) diffusion into the bulk of first wall components and iii) re-deposition of eroded carbon with incorporation of tritium into the deposited films. Of these, the highest contribution arises from re-deposition. At JET for instance, all through the vacuum vessel, where plasma interacts with graphite or CFC (carbon fibre composite) tiles, carbon is constantly being eroded and re-deposited in colder regions which are shaded from ion fluxes[1], such as the gaps between tiles, the louvres and ducts to the pumping system. At the louvres, which are thin, toroidally stacked continuous plates that block the direct line of sight between the inner coils and the divertor tiles, the deposits were present at the top edges of their back surfaces as well as on surfaces facing the centre of the machine.

Major erosion takes place near the divertor strike points. Erosion mechanisms under normal operation conditions with hydrogenic particles includes physical sputtering ($T < 600$K), chemical erosion ($600 < T < 1200$ K) and radiation-enhanced sublimation ($T > 1500$K) [1]. Thermal sublimation is only expected at temperatures above 2000 K during disruptions and runaway current events.

The chemical erosion yield (C/H) vary with the quality of the target (highly or poorly orientated pyrolitic graphite) as well as with the energy and the nature of the impinging particles H^0 or H^+. As a rule, it increases with ion energy and displays a small hydrogen isotopic effect. The temperature dependence of the chemical erosion yields are similar for all types of incident hydrogen particles and show a maximum at approx. 750-800K [2, 3]. Hydrogen isotopes react chemically with the eroded carbon. There is strong evidence from laboratory experiments and from the operational experience gained with tokamaks that chemical erosion of graphite surfaces produces hydrocarbons, the spectrum of the reaction product being analogous for all particle interactions. Indeed, the main reaction product is methane (50-75%), and other less important products are C_2H_x (C_2H_2, C_2H_4, C_2H_6, etc.) (20 - 35%) and C_3H_y (C_3H_6, C_3H_8, etc.) (5 - 15%). A fraction of the products is probably generated via C^+, CH_x^+ and H^+ ions impinging on the divertor and producing C_2H_x, radicals via a polymer-like C:H intermediate. These eventually lead to the formation of hydrogen-rich loose carbon layers and well-adhered layers of low D/C ratio in shadowed areas of the machine. The co-deposition process can go on indefinitely without

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[1] *The divertor support structure is water cooled (~20°C). Divertor tile surfaces are at intermediate temperatures (e.g. ~200°C when the torus wall is at 320°C).*

any apparent self-limitation and in the end contribute significantly to the build-up of tritium inside the vessel. Calculations for ITER yield for tritium a co-deposition rate in the grams per 400 s pulse range [4]. In addition to co-deposition, small amounts of tritium may be sorbed reversibly on dust during prolonged direct exposure to this radioisotope, i.e. < 4 GBq g^{-1}, the amount increasing with temperature [5].

Thick carbon deposits containing high levels of hydrogen isotopes have been observed in JET as well as in TFTR after exposure of first wall materials to discharges [6, 7, 8]. In the vacuum vessel of JET the deposition was found to be asymmetric, i.e. while heavy deposition occurs at the inner divertor region there is comparatively little at the opposite side. Examination of a complete set of poloidal tiles revealed the highest deposition was on the plasma shaded lower edge of inner tiles 3 of the Mark IIA divertor [6, 7]. The edge deposition showed two distinct regions, one closer to the plasma-exposed surface of the tiles having comparatively low tritium concentrations and another region, further away from the surface, of thick deposit with a clear tendency of spall off (see Fig. 1 in ref. [7]). Inner base tiles 4 were also characterised by strong deposition and flakes falling or hanging from the side shadowed from the plasma (see Fig. 5 in ref. [7]). In fact, the deposits on tile 4 and at the end of tile 3 were well-adhered when you first remove the tiles, but almost complete spalling has occurred in storage since that time. An analogous observation was also made in TFTR. The worst areas for loose material were on the plasma exposed regions of tiles 4 and 7, where material come off on sticky tape and hence demonstrating that these deposits are loosely bound. An earlier estimate at the time of RTE suggested that 35% of the flakes were collected by a vacuum cleaner [9] but at that time the piles of flakes under the divertor had not been seen.

From Scanning Electron Microscopy (SEM) of cross sections of the tile surface it appeared that the deposited material has a thickness of several tens of microns. Spalled-off deposits constitute what has been identified as dust and flakes. Dust and flakes naturally accumulate on lower horizontal surfaces. In addition to flakes and deposits on louvres and protecting coils, large amounts of debris were observed with an endoscope, mostly in the sub-divertor region of JET. They have accumulated in this region during the operation of the MKIIA divertor (1996-1998).
It is estimated that 6-8% of the total D fuelled into the machine is presently trapped in deposits on tiles as well as in dust and flakes in the inner divertor region.

At TFTR approx. 15% of the observable tiles showed flaking after the end of plasma operations, appearing predominantly on isotropic carbon [10, 11]. Dust accumulated at TFTR mostly on the floor of the vacuum vessel. Typically about 0.5 g were collected from an area of about 100 cm^2 [12].

In ASDEX Upgrade, brownish (D/C = 0.4) as well as transparent (D/C ≈ 1) layers and flakes were found under the divertor structure after the 1999 experimental campaign (3000 s of plasma operation) [13].

Because appropriate diagnostics are not available, it is not possible at present to quantify dust inside an operating machine. This would, however, be important because dust may constitute a safety hazard during an accidental air ingress. Co-deposits, flakes and dust are also of much concern because of the associated progressive increase of tenaciously held in-vessel tritium inventory. Furthermore, detailed knowledge of the distribution of tritium and other hydrogen isotopes within the vessel constitute an important database for modelling carbon transport phenomena from regions of high flux of incident particles to regions of low flux. It also represents fundamental information needed for the selection of suitable plasma facing materials and the development of concepts to mitigate the negative effects of co-deposition.

At JET, even after a clean- up period of three months pulsing with deuterium following DTE1, (First D-T Experiment), the tritium retention by the vessel was still high. Indeed, 6.2 g of tritium out of the 35 g injected into the torus (18% of the fuelling) was still outside the Active Gas Handling System (AGHS) and therefore presumed to be in the JET vacuum vessel [9].

In this report experimental results on the properties of flakes retrieved from JET before and after the DTE1 campaign are presented and the implications for the total tritium inventory presented. The current state of knowledge on the physical and chemical properties of flakes and dust collected from operating machines is compared and discussed.

2. Characterisation of flakes from JET (physical properties)

2.1. Particle size

In-vessel dust and flakes from the divertor region are in principle discernible by their respective sizes (see Table 1). Most flakes from the pre-DTE campaign of JET were found to have dimensions of the order of 0.5 mm with a thickness of about 20 μm (occasionally flakes having lengths up to 20 mm were also collected). Metallic particles consisting of Fe, Ni and Cr (components of inconel) were also detected among the flakes [14]. SEM showed that the flakes have a distinct layered structure, each layer being several μm thick. Using SEM analysis, a thickness twice as large, i.e. 40 μm, was measured by Peacock et al. [6] for some pre-DTE flakes. In a later publication, Peacock et al. [15] reported that dust obtained via vacuum cleaning of the JET torus after the pre-DTE campaign had a median diameter of 27 μm. For this measurement, the collected material was filtered through three membranes of successively smaller pore size and the median diameter determined from the three fractions.

The thickness of highly tritiated DTE1 flakes varied within a range of 46-60 µm and, as in the case of the pre-DTE flakes, they showed individual sub-layers with a thickness that varied from 8 to 30 µm [16, 17]. Their width was in the mm range (at times single massive pieces of 15 x 50 mm were also observed). The majority of the dust particles (< 90 µm) collected with a cyclone vacuum cleaner from the JET machine after the DTE1 campaign was found to be in the range 1.5 - 10.5 µm [17]. Patel et al. [18] observed the dust to be of highly irregular shape and gave a mass median diameter of 6.4 µm for particulate sizes ranging from 4 up to 34 µm. On the other hand measures performed at the Hot-cells in FZK on JET flakes showed that the bigger part of the particles had an average diameter between 375 and 410 µm (see Fig. 1).

In figures 2 and 3 the pictures obtained by optical spectroscopy for sample 2-5 of pre-DTE flakes are shown. As it is illustrated in the pictures metallic occlusion are visible as well as the layer-type structure of the flakes witnessing the existence of a successive deposition mechanism.

Summarising, JET flakes have sizes in the mm range and a thickness of < 60 µm.

Typically, dust had accumulated at TFTR at the bottom of vertical diagnostic viewing tubes. Carmack et al. [19] examined TFTR dust with an optical microscope and found the count based median diameter ($d_{50\%}$) to range from 0.8 to 3.0 µm. Together with the dust, a few flakes of mm scale were collected after operations at TFTR ceased [10]. But it was only after subsequent openings of the machine that massive flake formation took place at the bumper limiter.

From a safety point of view, dust particles of less than 10 µm are considered respirable aerosols with a potential of settling in the alveolar region of lungs. They therefore require particular attention.

2.2. Density

The density of a single JET flake collected after the DTE1 campaign was determined with a commercial helium pycnometer to be 1.6 g cm^{-3} [16]. This value is high but significantly lower than that measured at the FZK with the same technique for the CFC material of JET tiles, i.e. (2.08 ± 0.01) g cm^{-3}.

2.3. Particle BET surface area

BET (Brunnauer, Emmett, Teller) surface areas of 675 and 1000 m^2 g^{-1} respectively, have been reported for flakes and dust collected from the vacuum vessel of JET after the DTE1 campaign (see Table 1) [16, 17]. In contrast, dust produced from a JET tile was shown to have a BET surface area between 11 and 36.4 $m^2 g^{-1}$ for fine particles with sizes between 4.5 µm and 80µm [20]. For the large size fraction of the pre-DTE flakes, Peacock et al. [15] reported a specific BET surface area of 4 ± 2 $m^2 g^{-1}$. The latter is in line with the measurements done at the TLK on pre-DTE JET flakes sample 3-4. Indeed, the sample had a mass of 155.9 mg, an average tritium activity of 1MBq g^{-1} and a BET surface of 7.2 $m^2 g^{-1}$. It thus appears that both values given in refs. [16] and [17] are exceedingly high, i.e. by more than two orders of magnitude. It is therefore considered necessary to verify these measurements by additional experiments. A corresponding effort is presently in preparation at TLK with flakes both from the DTE1 campaign. However, some technical problems have to be solved, such as weighing dusty material having a high specific tritium activity without excessive contamination of the glove box and the introduction of liquid nitrogen into the box. Contamination is mainly expected to occur from electrostatic effects on particles in the dry glove box atmosphere, as charged particles tend to adhere to the walls of the glove box and on the equipment. Also of concern are significant chronic releases of tritium from flakes [21] and dust spread during transfer operation of flakes from the cyclone pot (used for the collection of flakes from the louvres in JET) into the BET apparatus. Procedures required for these operations have been written and the development of a strategy for the disposal of flakes and the contaminated equipment after completion of the planned measurements is in progress.

Dust samples collected from different locations in TFTR were found to have BET specific surface areas in the range 7-27 $m^2 g^{-1}$ [19, 22]. An average value of 13.5 ± 9.5 $m^2 g^{-1}$ obtained from fourteen single measurements is provided by Anderl et al. [22]. To transfer the flakes from the transport vessel into the BET apparatus they were first suspended in alcohol. The implied assumption is that alcohol has no influence on the surface properties of the flakes. After letting the alcohol slurry settle most of the alcohol was removed and assayed for tritium by liquid scintillation counting (LSC). The remaining alcohol was evaporated. The sample was then heated at 60°C under an argon stream and thereafter baked at 260°C for about 4 hours. Both krypton and nitrogen were used for the determination of the adsorbed gas volume. The obtained specific surface area values were larger than those of dense carbon spheres. From this it was concluded that TFTR dust probably consists of agglomerations of smaller particles.

In general, dust from JET or TFTR has larger BET surface areas than flakes, which is in accordance with expectations.

3. Characterisation of flakes from JET (chemical properties)

3.1. C/H/D/T composition

Flakes and dust from JET were found to consist mainly of carbon (> 70%), oxygen (2-15%, for dust up to 34%), iron (0.1 - 2%), chromium (2%), nickel (0.1 - 3%), aluminium (0.3-5 %) beryllium (traces up to 8%) and hydrogen isotopes [14, 16, 17]. Another independent quantitative analysis yielded 96, 0.5, 0.6 and 3 wt. % for carbon, beryllium, metals and oxygen, respectively [6], which is basically consistent with the data given above. As shown by this data, flakes and dust from JET do not differ greatly in their chemical composition.

An elemental analysis performed on JET flakes yielded an atomic composition of 85.9, 3.2, 9.7 and 1.2% for carbon, protium, deuterium and tritium, respectively. This analysis surprisingly did not give any value for the oxygen content. Expressed in atom %, one obtains 14.1% hydrogen isotopes in carbon and expressed in weight % the value is 2.4. The D/T ratio in the flakes is calculated to be 8.1. Ion beam analyses showed the deuterium content in JET flakes to be rather high, i.e. D/C = 0.75 [9] which is very close to the (0.73 ± 0.2) D/C ratio measured for an amorphous C:D layer by different laboratories using different techniques [23].

An analysis of deuterium and tritium in TFTR dust samples gave values of $(6.1 \pm 2.1)\mathrm{xl}0^{21}$, $(3.5 \pm 1.0)\mathrm{x}10^{19}$ and $(1.5 \pm 0.8)\mathrm{x}10^{18}$ atoms for carbon, deuterium and tritium respectively [22]. The average D/C and T/C ratios for TFTR dust were found to be 0.0062 ± 0.001 and 0.00026 ± 0.0001, respectively, from which a D/T atom ratio of 24 can be obtained. This ratio differs only by a factor of three from that reported for JET flakes. In another investigation, similar D/C and T/C ratios of 0.0058 and 0.00023 respectively were published for TFTR dust. From these data one obtains 25.2 for the D/T ratio, which is comparable to that given in ref. [19].

Expressed in atom % one obtains 0.6% hydrogen isotopes in carbon for TFTR dust. This result is lower by a factor of approx. 20 from that for JET flakes.

3.2. Thermal release of tritium from flakes and dust

Tritium release rate measurements were carried out in a flow system at temperatures increasing progressively from ambient up to 1100°C using helium carrier gas containing 0.1% vol. H_2 (1 mbar). A typical run carried out with flakes from the JET D-D campaign comprising two consecutive linear temperature ramps (7.4 °C min^{-1} from 20 to 500°C and 25.5°C min^{-1} from 500 to, 1100°C) is depicted in Fig. 4. It shows the liberated tritium in Bq, the release rate of tritium in Bq s^{-1} and the cumulative liberation of tritium in Bq. As shown by the results, temperatures in excess of 300°C are necessary to initiate a significant liberation of tritium and very high temperatures are required for a quantitative discharge of tritium. Fig. 5 illustrates for three flake samples under the linear ramp conditions given above that the maximum release rate takes place at approx. 860°C (see also Table II). The reproducibility of these measurements is rather good. It was apparent that even at a constant temperature of 1100°C, a slow tritium liberation occurs. The rate, however, becomes progressively slower.

Investigations in another laboratory (JET) of the thermal release of tritium from JET flakes collected after the DTE1 campaign were carried out by thermal differential analysis (TDA). As is evident from Fig. 6, a significant liberation of tritium starts only at temperatures above 300°C, which is consistent with previous observations gained with flake samples from D-D campaigns. Remarkably, even when a small flake specimen is heated progressively from ambient temperature up to 800°C with a rate of approx. 4 °C min^{-1} for a total time of nearly three hours, only 89% of the tritium inventory is released. This conclusion was reached after carrying out a subsequent run, in which the previously baked-out specimen was fully combusted under air at 900°C [16].

An analogous tritium release study with carbon dust specimens collected from TFTR is in line with the observations made with JET flakes, i.e. the maximum tritium (and deuterium) liberation rates are registered at 780°C when the dust is subjected to a linear heating ramp of 10°C min^{-1} [22]. For the measurements, dust samples were transferred from the sampling vessel into the flow tube using the same ethanol-slurry technique already described in section 2.2. A comparison of tritium release rates from JET flakes and TFTR flakes is shown in Fig. 7. It shows similar thermal release behaviour in spite of rather different sample origin and different initial tritium concentration. This is an indication that the co-deposited material from both machines is chemically analogous (see also Table III).

One conclusion from the results presented above is that hydrogen isotopes are tenaciously held in flakes and dust from JET and TFTR, being releasable only by heating to very high temperatures for an extended period of time. Debris from JET and TFTR show essentially no differences in their thermal stability.

An important observation concerning the tritium content of flakes and dust retrieved from JET is that it is more than an order of magnitude higher than that collected from TFTR (see Table I). There are two important reasons for the difference in content. Firstly, the flake composition reflects the entire history of the deposit, so that the ratio of D/T depends on the ratio of D fuelling to T fuelling of the tokamak. An order of magnitude more tritium was used in JET than in TFTR. Secondly, the ratio of carbon to total hydrogen isotope content of deposits depends on their location and temperature history. TFTR is a limiter machine, and all deposits are subject to bombardment by energetic particles from the plasma which limits the H/C ratio. Furthermore, the TFTR flakes are mostly from the sides of limiters that may reach high temperatures during plasma discharges – the permissible H/C ratio reduces above about 300C. Similarly, low H/C ratios are found in all deposits in the main chamber of JET. However, in JET the majority of the flakes are located in areas shadowed from all energetic particles, and the temperature is controlled by proximity to the water-cooled divertor structure. In both cases, a large initial retention of tritium during plasma operation (respectively 40% and 90%) as well as a large long-term tritium retention (respectively 17% and 51%) [8] was observed.

3.3. Chronic release of tritium from flakes at room temperature

At the inner corner of the JET Mark II A divertor there is a region of heavy deposition, the deposits tending to spall-off and eventually lead to the formation of flakes typically a few mm across and < 60 μm thick. In section they comprise a number of layers which reflect their progressive growth history during successive plasma discharges constituting a historical record of the machine operation.

Presumably some layers contain tritium and deuterium and others, either closer to the outer surface or deep in the bulk, only deuterium. A small fraction of these flakes was shipped to the Tritium Laboratory Karlsruhe (TLK) for chemical and physical characterisation.

3.3.1. Flake collection after the DTE1 campaign

To remove the flakes toroidally from the machine a dedicated, remotely operated vacuum cleaner fitted with a cyclone pot and provided with a stainless steel brush capable of collecting material down to 2 μm was introduced into the vacuum vessel (Fig. 8). The sample pot, which was machined from a single aluminium block, has a free volume of about 200 cm^3 and is sealed with a stainless steel ball valve (Fig. 9). This sample pot containing the flake sample was purged thoroughly with argon before closing. The pot itself was sealed inside a primary pink PVC plastic bag containing only traces of air. This primary bag was in turn sealed inside a secondary aluminised bag containing a flax bag filled with a silica gel desiccant. The secondary bag was further sealed inside of a tertiary aluminised bag containing a palladium pellet catalyst of the type used at Tritium Retention Systems of TLK, which was then placed in a perforated plastic bag (Fig. 10). The palladium catalyst is capable of oxidising hydrogen to water at ambient temperature and has also a high adsorption capacity for water. The adsorber noble metal catalyst was intended to retain molecular hydrogen possibly escaping from sequestration in the flakes. This whole arrangement was placed inside a vacuum-tight stainless steel transport vessel. Before closing the vessel, the atmosphere of the vessel was replaced by argon by sweeping with this gas for a period of approximately 90 min. Plentiful bubble wrap packing served to reduce free motions of the bags inside of the vessel. In a safety assessment at JET, which extended over several weeks, the leak-tightness of the transport vessel was verified.

By the collection procedure described above, approximately 90% of the visible flakes were removed from the louvres. A very large fraction of the flakes, however, still remain in the sub-divertor zones of JET. This was verified and documented by inspection with an endoscope. These flakes, which are only accessible with a specially developed vacuum device, have immobilised a high tritium inventory. According to present knowledge, they contain the vast majority of the tritium remaining in the machine.

3.3.2. International transport of the flakes

On the 30th of April, 1999, approximately 1 g JET flakes containing more than 1 TBq of trapped tritium were transported successfully from Culham, England to the Tritium Laboratory Karlsruhe (TLK) in Germany. The complete configuration of the transport vessel is shown schematically in Fig. 11. The solid, heavy vacuum-tight transport vessel used to contain the flakes was made of stainless steel and had a lid sealed with a metal O-ring. For shipment the vessel was fixed inside a strong wooden box. There is a three bag "barrier" between the cyclone pot containing the flakes and the transport stainless steel vessel (Fig 9 to 11).

The stainless steel vessel was equipped with two valves on the lid to allow safe purging prior to removing the bags from the vessel. When purging with argon was performed, a down-stream ionisation chamber detects only minimal releases of (molecular?) tritium. From the experience with this transport method it can be concluded that the concept selected at JET is adequate and sufficiently safe.

3.3.3. Determination of tritium off-gassing at ambient temperature

After an extended interim storage of the JET flakes at TLK over a period of 21 months they were subjected to a series of tritium release tests. All release measurements were carried out in a glove box (volume 6.7 m^3) equipped with a dedicated conventional Tritium Retention System, an Overhoff tritium monitor and a moisture sensor. After rupturing the aluminised bag containing the flax bag with palladium catalyst and the aluminised bag containing the silica gel, the tritium content in the catalyst and in the silica gel was determined by dropping the solids in water and measuring the tritium in the water by liquid scintillation counting until solid/liquid equilibrium was established, which took about one week (5 days). From the rise in activity in the glove box

(TRS was off) and the activities found in both solids the total liberated tritium was calculated to be 0.42 TBq. This activity, which corresponds to roughly one third of the activity originally present in the flakes (see section 2.4.), led to an exceedingly high contamination of the glove box. It was therefore necessary to introduce the pink PVC bag into an acrylic tube provided with a cap on both sides. Under these conditions the activity in the glove box diminished slowly down to acceptable levels below 10^8 Bq m^{-3}. To assess the chronic release of tritium from the flakes, the acrylic tube was opened on both sides periodically and the activity released into the glove box as well as changes in moisture concentration in the glove box registered. Experimentally it was shown that at all times the pink bag and the cyclone pot were almost fully transparent to the movement of tritium. This was demonstrated during the first run in which, after the first tritium release measurement into the glove box, the pink bag was ruptured and then the ball valve was opened. Neither of these last two operations had any significant influence on the tritium level in the glove box.

The ambient temperature chronic release of tritium was observed in spite of the fact that tritium on tiles and in flakes or dust is retained thermally in an extremely stable way. The average chronic release rate was estimated to be $(1.8 \pm 1.1)\times10^{10}$ Bq $month^{-1}$ g^{-1} of flakes over a period of two years. As one would expect, due to the decrease in tritium inventory in the flakes, the rate of tritium release shows a tendency to decrease over time.

To investigate whether oxygen or humidity have an influence on the room temperature release of tritium, in some runs the atmosphere of the glove box was fully replaced by nitrogen, but this did not appear to have an impact on the tritium release rate. This could be consistent with a radiation-induced mechanism for the tritium liberation.

The concomitant increase of moisture with every tritium release into the glove box suggests that the tritium is mostly released as water. In fact, the moisture concentration in the glove box correlates well with the concentration of tritium if it is assumed that all tritium is present as water. A further argument supporting this observation is that almost all tritium liberated from the flakes was found in the silica gel and in the palladium catalyst and only a very small amounts migrated, probably as molecular tritiated hydrogen, through the aluminised bags into the stainless steel transport vessel. A further evidence of this effect was observed during calorimetry at JET on a set of flakes which was observed to be quite moist [24].

As apparent from Fig. 9, the flakes can easily be accessed through the large bore of the ball valve. Visual inspection through the glove box window showed the JET flakes to be black and not at all adherent to the pot walls.

3.3.4. Evaluation of chronic tritium releases

Chronic releases can be rationalised on the assumption that the beta particles break carbon/hydrogen bonds in the co-deposited material generating water with oxygen atoms existing on the particle surface [25]. If the energy absorbed by one gram flakes from the 1.1 TBq of tritium is compared with the experimentally determined rate of product formation, a G value of 0.08 is obtained, which is common to radiation-induced gas evolution from polymers [26].

G is defined as the number of atoms, or molecules reacted or produced after absorption of 100 eV.

Andrews et al. [27] noted that chronic releases of tritium following DTE1 were more than twenty times greater than anticipated from the pre-DTE campaign. In addition, tiles from the inner divertor had 10 times higher out-gassing rates than other tiles. They attributed this to the presence of co-deposited films and flakes in low temperature zones that are less stable to an exposure to humid air than plasma-exposed material. A review of this enhanced chronic release observed at JET in the light of the discovered radiation-induced liberation of tritium from flakes and tiles in a highly contaminated machine may contribute to improve the understanding of this phenomenon.

4. Tritium content of flakes and dust

4.1. Pre-DTE flakes

Several thermal release tests were performed with flakes obtained from JET after completion of D-D campaigns. In such campaigns tritium is produced via the nuclear reaction

$$\mathrm{D + D \rightarrow T + H + 3.98\ MeV} \qquad (50\%)$$

For an evaluation of the tritium content, a few flake specimens were subjected in a conventional flow apparatus to two consecutive linear heating ramps, i.e. 7°C min^{-1} from 20 up to 500°C and 15°C min^{-1} from 500 up to 1100°C, respectively. The carrier gas used in these experiments was helium-containing 0.1 % hydrogen; the flow rate was kept constant at approx. 30 cm^3 min^{-1}. The tritium released under these conditions into the gas phase was integrated using an ionisation chamber installed downstream. The average specific tritium content in the samples was determined to be (15.6 ± 3.0) MBq g^{-1} ([14], see Table I and II). This value is in reasonable agreement with the tritium content reported by Peacock et al. [15], i.e. 9 MBq g^{-1} (see Table I). The significant weight loss registered during the flow tube measurements, i.e. 24 to 65% (see Table II), can be attributed either to an oxidation of carbon with residual concentrations of oxygen in the carrier gas or to a volatilisation of carbon by reaction with hydrogen. A gas/solid reaction of graphite with hydrogen leading to the formation of methane has been registered at 785°C and 1.6 mbar [28]. The desorption of adsorbed water is considered to be of minor importance. The dust obtained after the pre-DTE campaign only contains 1.3 MBq g^{-1}, i.e. about one tenth of the flakes [15].

4.2. DTE flakes

Approximatively 150 g of flakes collected in two cyclone pots (56.8 g and 97.5 g) were retrieved from JET during 1998 after the DTE1 campaign during a difficult remote operation using a modified vacuum cleaner provided with a cyclone dust separator. The average tritium content of these flakes was determined calorimetrically at JET to be 1.17 TBq g^{-1} [16, 25]. During the initial stage of the measurement a rapidly decaying heat source was noticed leading to a tremendous out-gassing rate, consistent with the oxidation of hydrogen, which only ended after all oxygen present in the vessel was consumed. That phenomenon could be evoked to explain the much higher out-gassing rates observed at JET in the early stages after the flake's collection (Table IV). In another separate determination, the tritium content of three flake samples was obtained by heating specimens under air at 800°C and collecting the liberated tritiated water in bubblers containing water (see Fig. 12) [16]. Under these conditions an initial rapid release of tritium takes place, which is followed by a much slower but steady liberation over a period of many hours. The data suggest that a fraction of the tritium is particularly strongly bonded and only liberated after the flakes are fully oxidised. Based on these three measurements the average tritium content of the JET flakes was estimated to be (1.17 ± 0.11) TBq g^{-1}, in good agreement with the calorimetric data.

Approximatively 1 g of these flakes was also send to the Tritium Laboratory Karlsruhe (TLK) to determine their chronic tritium release at room temperature and also assess the tritium content by calorimetry and full combustion. During a period of 35 months (including 20 months of storage while the flakes were prepared at JET and sent to TLK) the chronic release rate was measured in TLK. It was determined that during that period a total amount of $4.96\ 10^{11}$ Bq (13.4 Ci) of tritium was released by off-gassing.

The tritium content of the flakes was measured by calorimetry and this has established that 0.93 g of flakes still contain 6.03×10^{11} Bq of tritium (16.3 Ci). These figures give a total tritium inventory of 1.18×10^{12} Bq of tritium per gram of flakes (31.9 Ci), or 3.3 mg of tritium per gram of flakes, and an average off-gassing rate of $2.1\ 10^{7}$ Bq h^{-1} g^{-1} of flakes.

Table IV shows a comparison between tritium activities and release rates measured at JET and in the TLK for the 3 flake samples.

4.3. Closing the tritium balance at JET

According to these measurements, the average tritium activity is (1.17 ± 0.1) TBq g^{-1} flakes. At the start of RTE there were 6.2 g of tritium missing. During RTE, 2.5 g were released into the air on venting and continuous release into the gas phase and recovered by the AGHS. This leaves 3.7 g, of which 0.5 g is in the collected flakes and approximatively 0.2 g is in the divertor and wall tiles. Therefore, at the end of RTE, approximately 3.0 g was not accounted for. This must remain in the vessel as flakes. If they have the same content of tritium as the flakes removed then a further (950 ± 80) g of flakes must be still present in the vessel (see Table V). This amount of flakes refines an earlier estimation stating that about 1 kg flakes were still present in the vessel after the DTE1 campaign [29].

Indeed, recently it was possible to validate this supposition as, by using an endoscope inspection of the sub divertor volume, large quantities of flakes were found in this area. We can hopefully close the tritium balance at JET assuming that this material has the same specific tritium activity as the recovered and analysed flakes, however this needs to be confirmed as their tritium content is not yet measured.

It should be also noted that the tritium content for the 0.93g of flakes gives a T/D ratio of 1.5%, close to the 2.3% of the total gas fuelling used for the entire DTE1 experimental campaign [9]. In other words, the tritium ratio in the plasma seems to be reflected in the tritium level in the flakes. Consequently, we may suppose that the tritium level in the flakes would be much higher for a 50:50 D-T fuelling mixture, which is the relevant range for a D-T "*all carbon*" fusion machine, and therefore, will lead to a tremendous tritium inventory in the vessel.

4.4. Tritium in dust and TFTR flakes

The "dust" from JET after DTE1 was collected by vacuum cleaning the plasma-facing surfaces of the divertor tiles, since any loose material from the vessel should collect on these, the lowest accessible surfaces in the machine. The tritium content in JET dust was obtained by total combustion and collection of the released tritium through a hot copper oxide catalyst in a water bubbler [17]. The values were found to vary within the range 0.56 to 1.27 TBq g^{-1}. The higher value is characteristic of the JET flakes, and is probably because some flakes were accidentally collected by the vacuum cleaner from the adjacent area at the base of the louvres

Skinner et al. [11] liberated the sequestered tritium by baking TFTR flakes at 500°C for an hour. They trapped the tritium in water and assayed it by liquid scintillation counting. The specific activity of the flakes was determined to be 0.11 TBq g^{-1}. Carmack et al. [19] measured the deuterium and tritium concentration in TFTR dust using thermal desorption spectroscopy (TDS). The average tritium concentration was found to be in the range 20-40 GBq g^{-1}. The D/C and T/C ratios of dust on filter samples were 0.0081 and 0.00044, respectively.

From the above it can be concluded that the tritium content of dust from JET or TFTR is lower than that of flakes (about an order of magnitude for JET).

5. Conclusions

- Flakes and dust are generated by co-deposition in all experimental fusion machines having a carbon first wall. At JET a very large fraction of tritium is retained by deposition (and therefore potentially in form of flakes) in the vicinity of the water cooled louvres adjacent to the inner divertor and in the sub-divertor zones of the machine (possibly 96 %).
- Generally, the hydrogen isotope content (tritium included) of dust is significantly lower than that of flakes. Their chemical composition, however, is rather similar.
- The concentration of hydrogen isotopes (tritium included) in the debris of JET is substantially higher than that of TFTR.
- There was some uncertainties concerning BET specific surface area of dust and flakes. New measurements performed on flakes collected after the pre-DTE campaign confirm a low value of at about 7 m^2 g^{-1}. More measurements on DTE flakes are needed to verify the presently available data.
- Hydrogen isotopes (tritium included) are tenaciously held in co-deposits, flakes and dust from plasma shadowed zones. To fully liberate by thermal means the immobilised tritium, heating to very high temperatures, i.e. > 500°C, over an extended period of time is necessary. With respect to thermal release no important difference between the flakes and dust from JET and TFTR was apparent.
- Tritium is released slowly but chronically at room temperature from flakes most probably by a radiation-induced mechanism.
- The multi barrier concept developed at JET for the international transport of flakes highly contaminated with tritium was demonstrated to be very reliable. The transport vessel was shown to be adequate for the safe unloading and transfer of the flakes from the vessel into a glove box via the air-lock.

Table I. Physical and chemical properties of dust and flakes.

Property	JET/pre-DTE		JET/DTE1		TFTR		Ref.
	Flakes µm	Dust	Flakes µm	Dust µm	Flakes µm	Dust µm	
Particle size [µm]	< 40 x < 20 000 (most about 20 x 500)		-				6
a) flakes: thickness x width							
b) dust: diameter		27 #					15
			46 to 60 x < 50 000	(1.5 to 10.5) x > 1000			16, 17
	375-410						This report
				(mass median diameter 6.4 µm)			18
						0.8 to 3	19
Density [g cm^{-3}]	nd		1.6				
BET surface area [m^2 g^{-1}]	4 ± 2 ##						15
		11 - 36.4					20
			675				16
				1000 ± 250			17
					nd	7 to 27	19, 22
						(average 13.5 ± 9.5)	22
	7.2						This report
Tritium content [GBq g^{-1}]	0.0156						14
	0.009						15
		0.0013					15
			1170 ± 110				16, this report
				560-1270			17
					111		11
						20 to 40	19
			1060 ǂ				26
			1270 ¥				26

nd not determined

\# *mass median from three fractions*

\# # *measured for the largest size fraction*

ǂ *This value was obtained for the first part of the flakes weighting 56.8g (see also Tab. IV)*

¥ *This value was obtained for the second part of the flakes weighting 97.5g (see also Tab. IV)*

Table II. Tritium release properties of flakes collected from JET after the D-D campaign

Sample	Initial weight g	Final weight g	Temperature of max. release rate °C	Weight loss %	Total tritium release 10^6 Bq	Specific tritium content 10^7 Bq g^{-1}
2.1	0.3318	0.2248	865	32.2	6.03	1.82
2.2	0.4052	0.3085	865	23.9	4.70	1.16
3.1	0.4171	0.2468	841	40.8	6.29	1.51
2.3	0.0310	0.0108	-	65.2	-	1.75

Table III. Comparison of the thermal release of tritium from flakes, dust and other specimens obtained from plasma-exposed tiles

Specimen	Plasma	Carrier gas	First linear ramp [°C min^{-1}]	Second linear Heating ramp- [°C min^{-1}]	Holding temperature °C	Release commences at °C	Maximum Release at °C	Reference
Gr-JET-2.11 flake	D-D	He + 1 % H_2	7.4 (RT to 500°C)	25.5 (500 to 1100°C)	1100	350	800 (after 117 min)	12
TFTR filter 2 dust	D-T	Ar+ 1 %H_2 (0.1 l min^{-1})	10 (RT to 1000°C)	-	1000	300	780 (after 110 min)	19
JET flake	DTE1	An	4.4		800	200	500* (after 90 min)	13

** at 800°C only 88.6 % of the tritium was liberated.*

Table IV. Comparison of off-gassing data

	Material	Date removed from JET Machine	Mass (g)	Date of analysis	Facility	Specific activity ($TBq\ g^{-1}$)	Method used to determine activity	Off gassing rate for total sample ($GBq\ h^{-1}$)	Off gassing rate per gram of material ($GBq\ h^{-1}\ g^{-1}$)	Method used to determine off-gassing rate
1	Dust	RTE - 1998	0.45	Oct 1998	AEAT	1.20	Off-gassing / / Combustion	2.67	5.9[1]	Open pot in air atmosphere for10 h
2	Flakes	RTE - 1998	56.8	Nov 1998	JET	1.06	Calorimetry	150	2.6[2]	Air purge over contnd. flakes for 38 minutes
3	Flakes	RTE - 1998	97.5	Jan 1999	JET	1.27	Calorimetry	150	1.5[3]	Argon purge over contained flakes for 50 minutes
4	Flakes	RTE - 1998	0.93	Dec 2000	TLK	1.18	Off-gassing / / Combustion	0.023	0.021[4]	Closed, bagged pot stored for 21 months

Notes on off-gassing measurements

1. This rate 5.88 $GBq\ h^{-1}\ g^{-1}$, was measured after 9 hours but was not constant. It is a fifth of the highest rate that was measured between 2 and 5 hours [17].
2. The cyclone pot was air purget to reduce the airborne activity prior to transferring the flakes to the calorimetry container. Purging was stopped when a constant activity level was achieved [26].
3. The cyclone pot was purget with argon to remove oxygen prior to transfer to the calorimetry container. Purging was stopped when constant activity level was achieved [26].
4. This figure assumes the 420 GBq contained in the Al bag and silica gel accumulated uniformly over 21 months (see pg.11). More likely, given the poor containment of the pot and the inner PVC bag, is that the material initially off-gassed at a high rate with oxidation occurring. Off-gassing would cease when the silica gel became saturated and equilibrium were reached between the material and it's surrounding atmosphere. The TLK work suggests that after the easy and rapid, release of the surface bound tritium a steady low off-gassing rate is reached. To verify the last statement it was suggested to grind the material and look if it releases or not significant quantities of tritium [30].

Table V. Closing the tritium balance at JET (MK IIA)*

35 g tritium were introduced to the JET torus from May to November 1997 during DTE1

11.5 g retained during DT operations
6.2 g still outside the AGHS after clean-up by pulsing deuterium

2.5 g recovered during the gas phase (venting) and
0.5 g recovered in 150 g flakes
~ **0.1 g** was estimated to be trapped by the 480 divertor tiles**
0.1 g was estimated to be trapped by the rest of the inner and outer walls

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3.2 g recovered
3.0 g remains in the vessel most probably as flakes

Average tritium activity (1.17 ± 0.1) TBq g^{-1} or 3.3 mg tritium per g of flakes i.e.

(950 ± 80) g of flakes must be present in the vessel.

Supposition recently validated by using an endoscope inspection of the sub-divertor volume where large quantities of flakes were found.

** JET Report on FT task JW1-1.1 (N.Bekris, P.Coad)*
** * estimation based on hundreds analysis of a complete set of 10 divertor tiles*

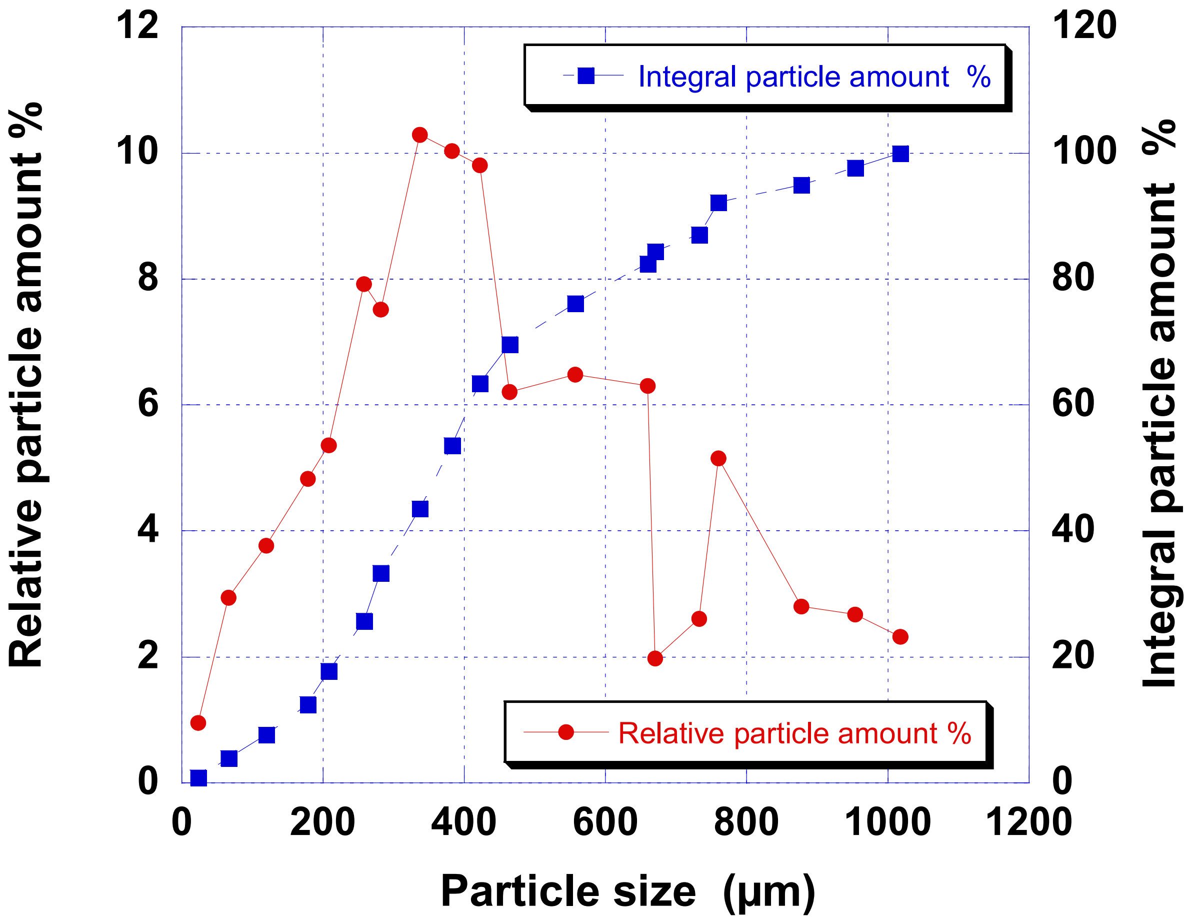

Fig. 1. Size distribution of dust retrieved from the vacuum vessel of JET after DTE1 campaign.

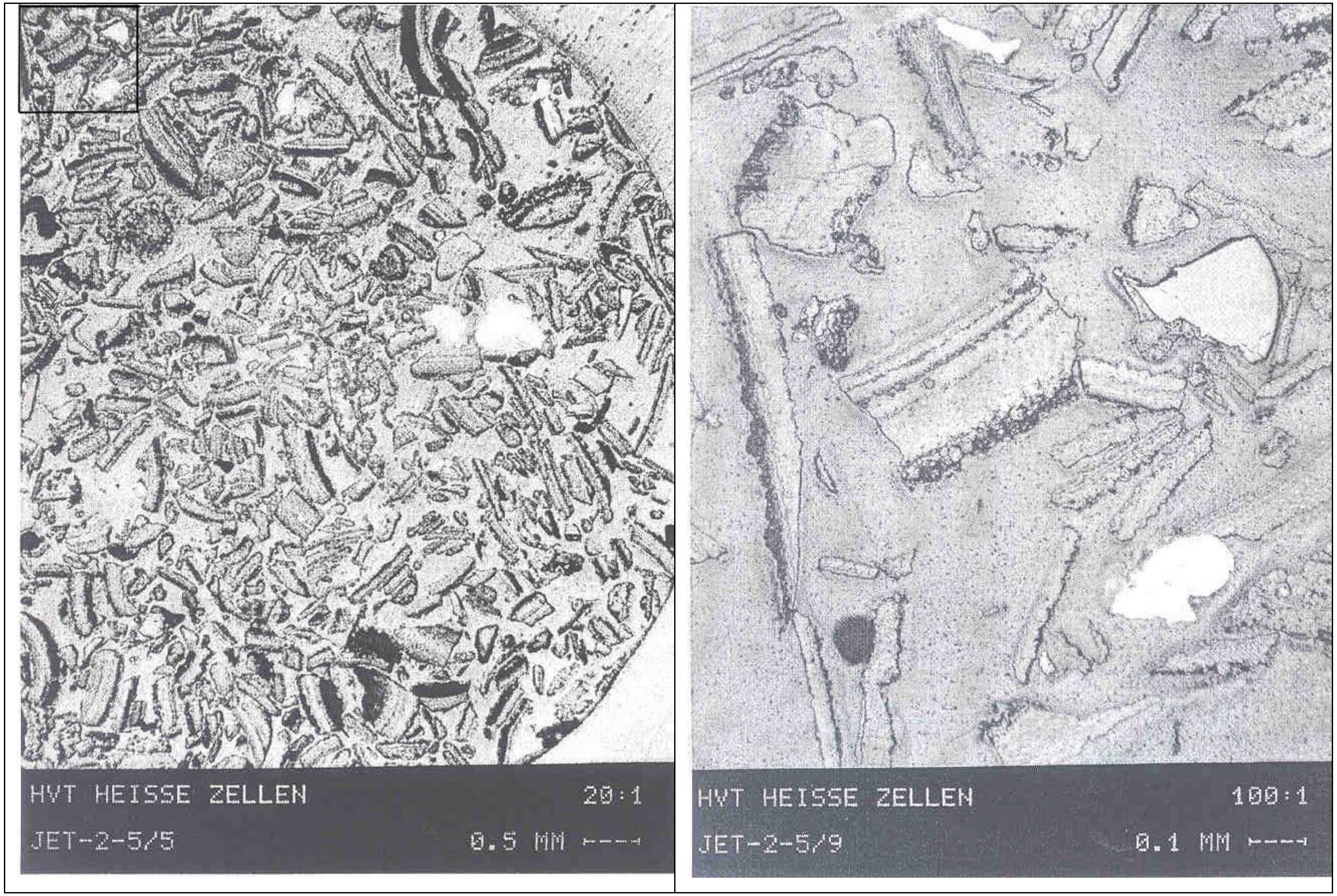

Fig. 2. Pictures obtained by optical microscope. On the left the flakes sample is magnified 20 times while the picture on the right is a further 5 time magnification of the black square appearing on the upper left corner of the picture a. Metallic occlusions are then visible as white bubbles.

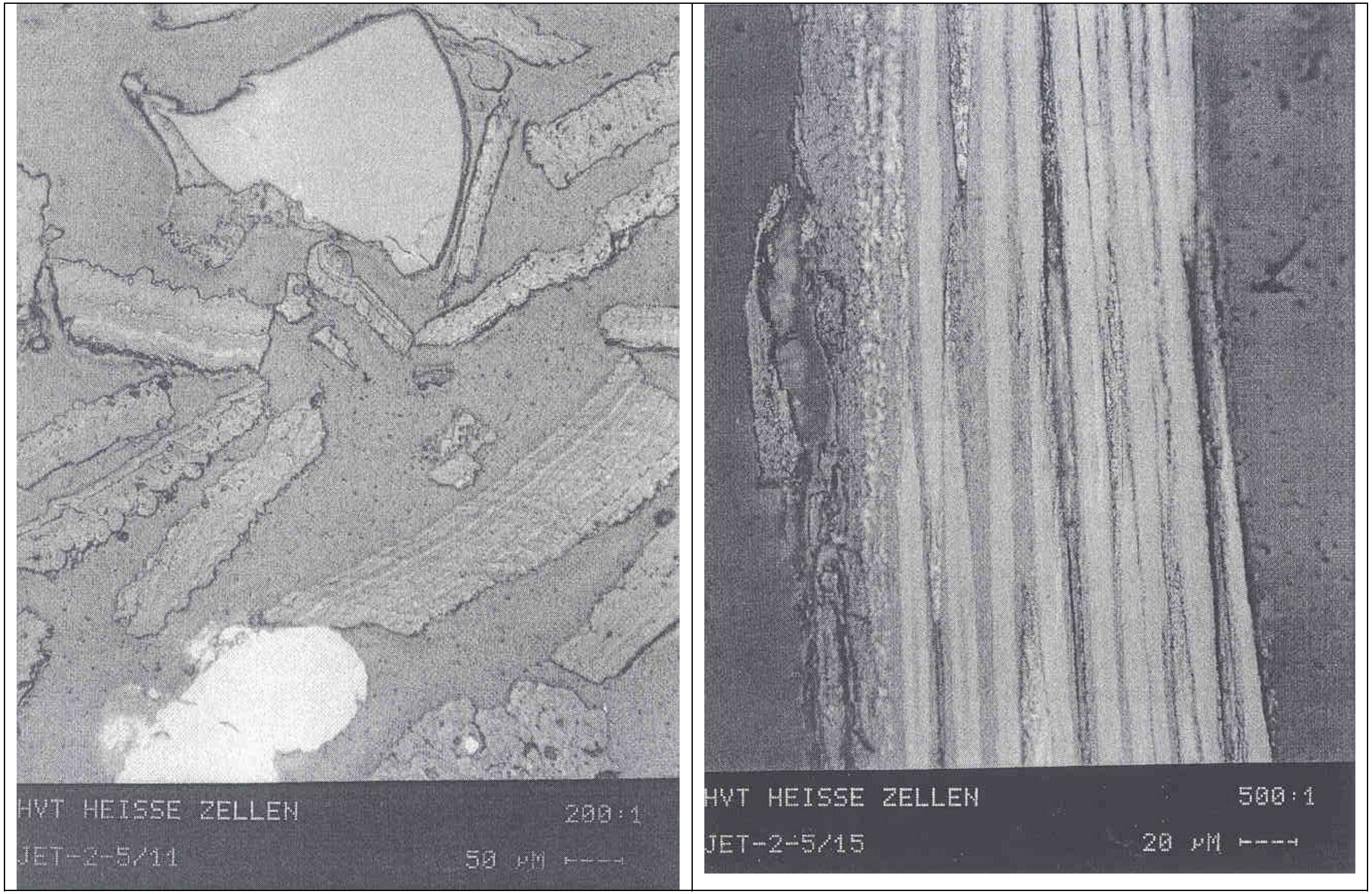

Fig. 3. Pictures obtained with an optical microscope using magnification at 200 and 500 times. The layered structure of the flakes is visible at 500x magnification.

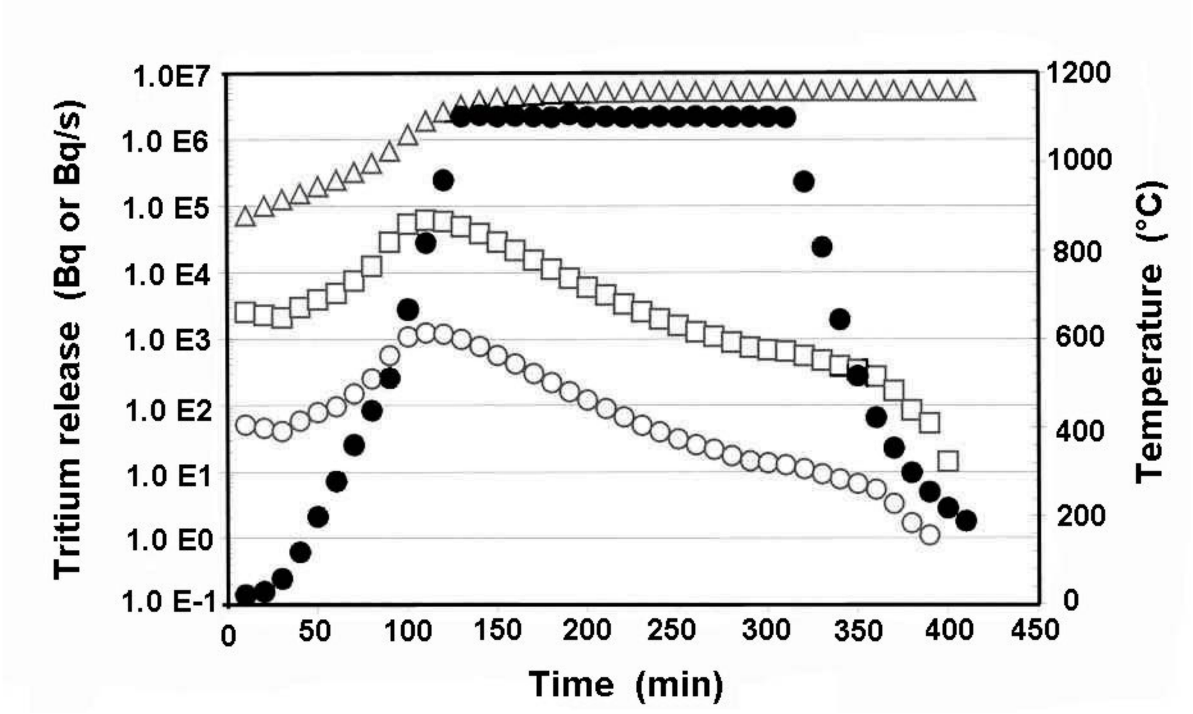

Fig. 4. Tritium release from JET flakes: • linear temperature ramp, ☐ released tritium in Bq, Δ cumulative tritium release in Bq, and o tritium release rate in Bq s^{-1}.

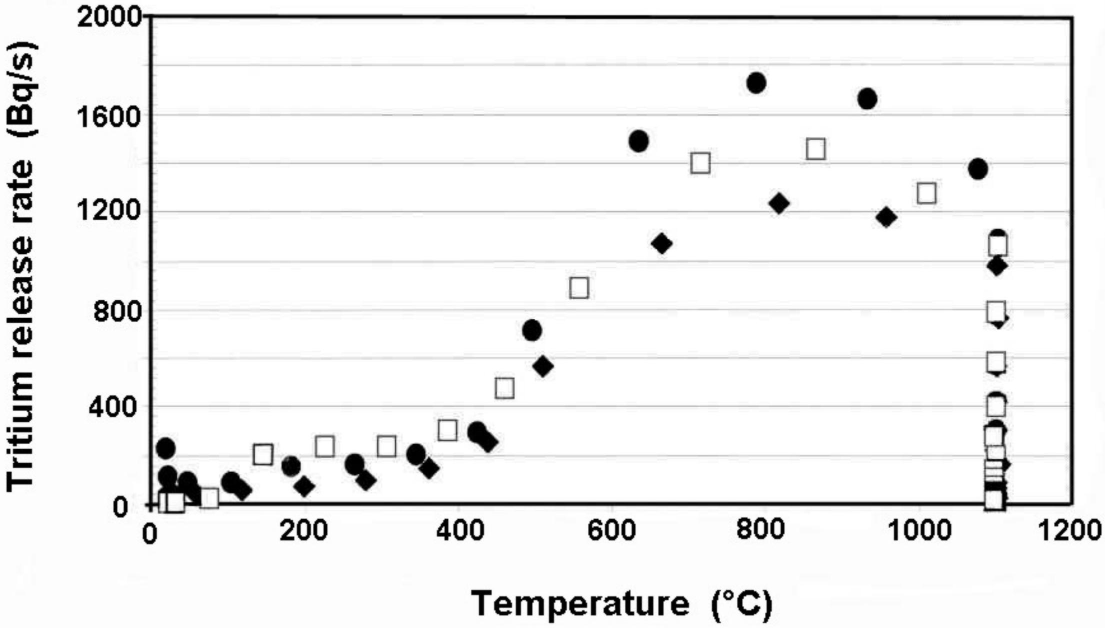

Fig. 5. Tritium release rates from three flake specimens (□ ● ♦) collected after JET D-D campaigns as a function of temperature. The temperature is then held at 1100°C, and the release rate drops gradually.

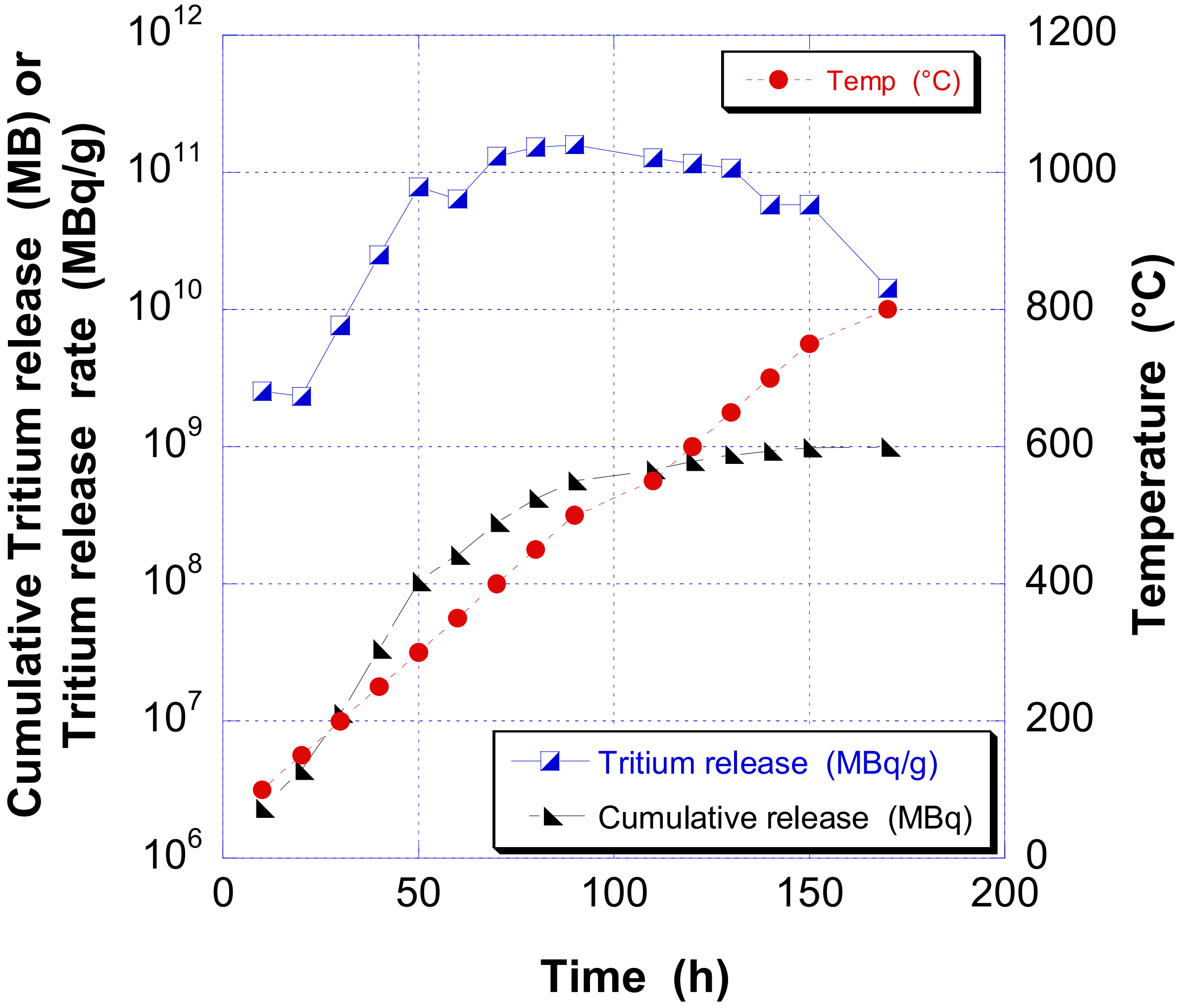

Fig. 6. Thermal release of tritium from a flake (weight 0.9 mg) retrieved from JET after the DTE1 campaign. The specimen was subjected to a linear heating ramp of 5°C min^{-1}.

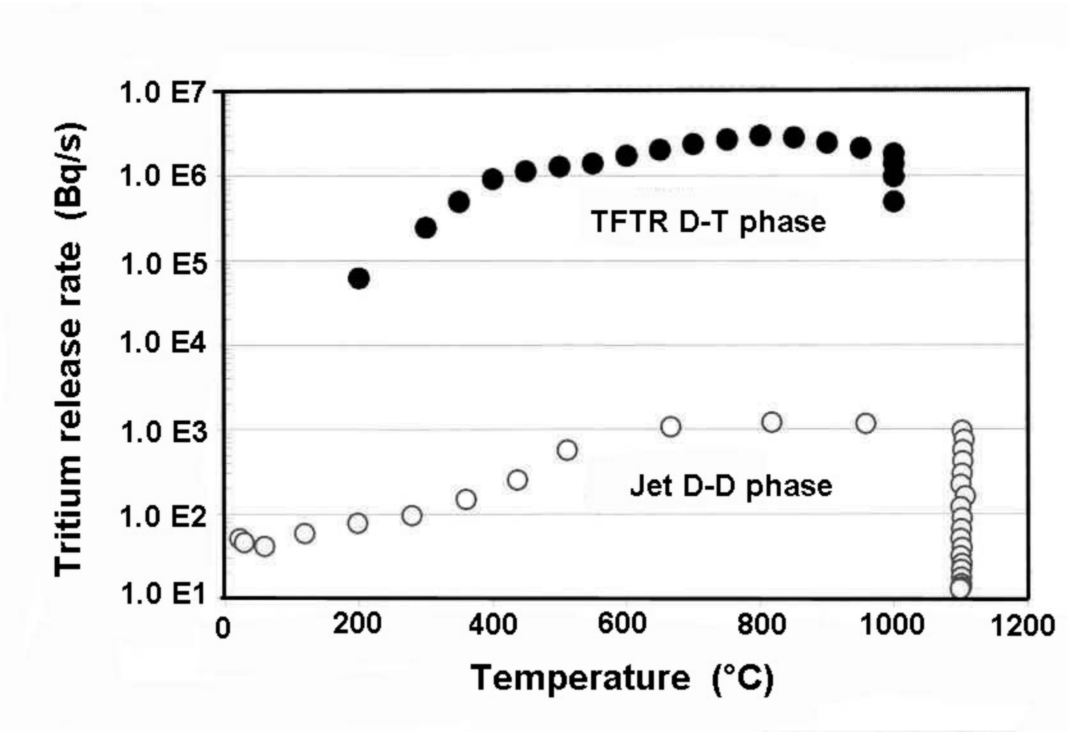

Fig. 7. Effect of the temperature on the tritium release rate from JET and TFTR flakes. At the end of the temperature ramp the temperature is held at the highest value, and the release rate gradually decreases.

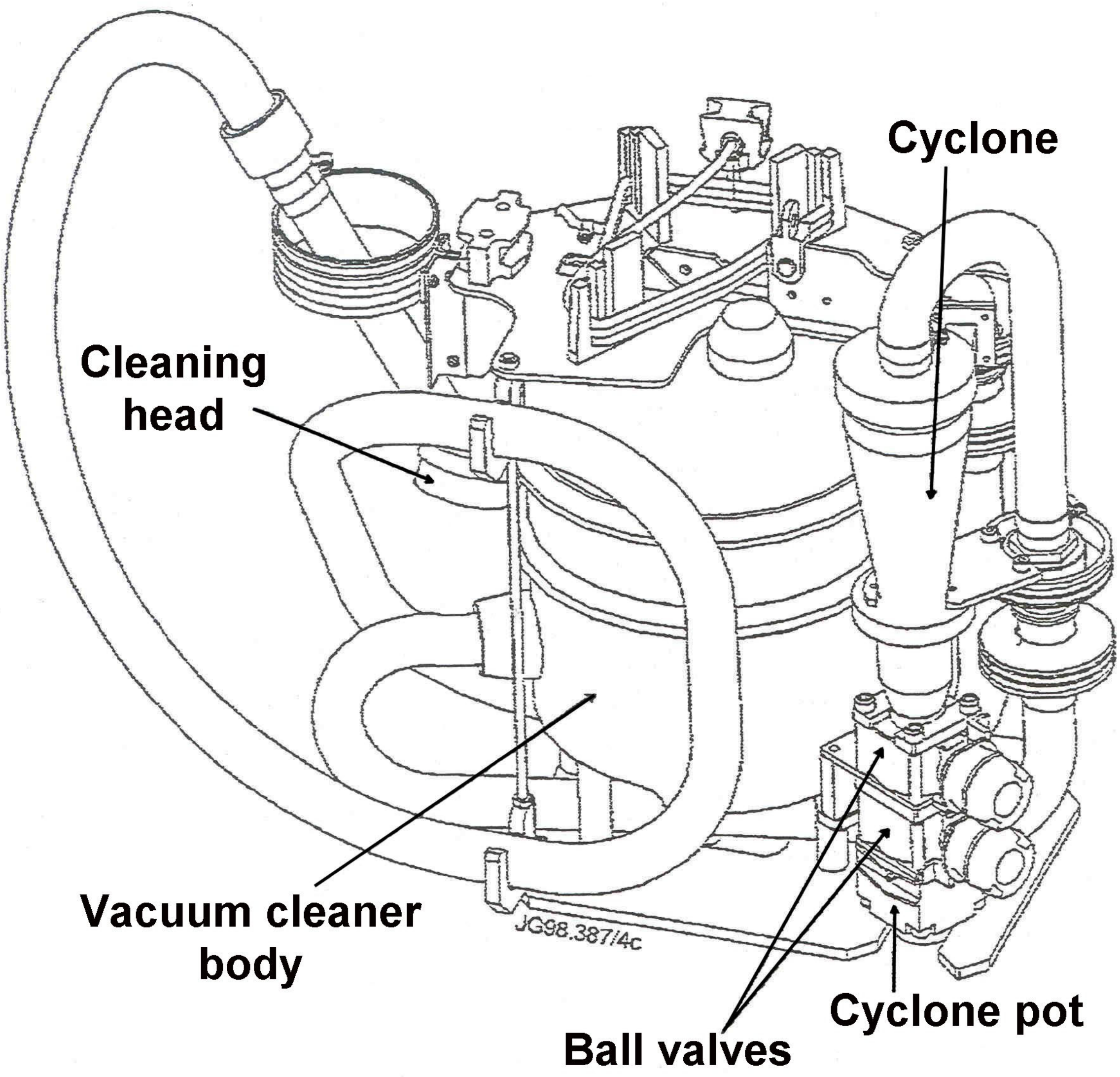

Fig. 8. Vacuum cleaner used in JET for the collection of flakes after the DTE1 campaign.

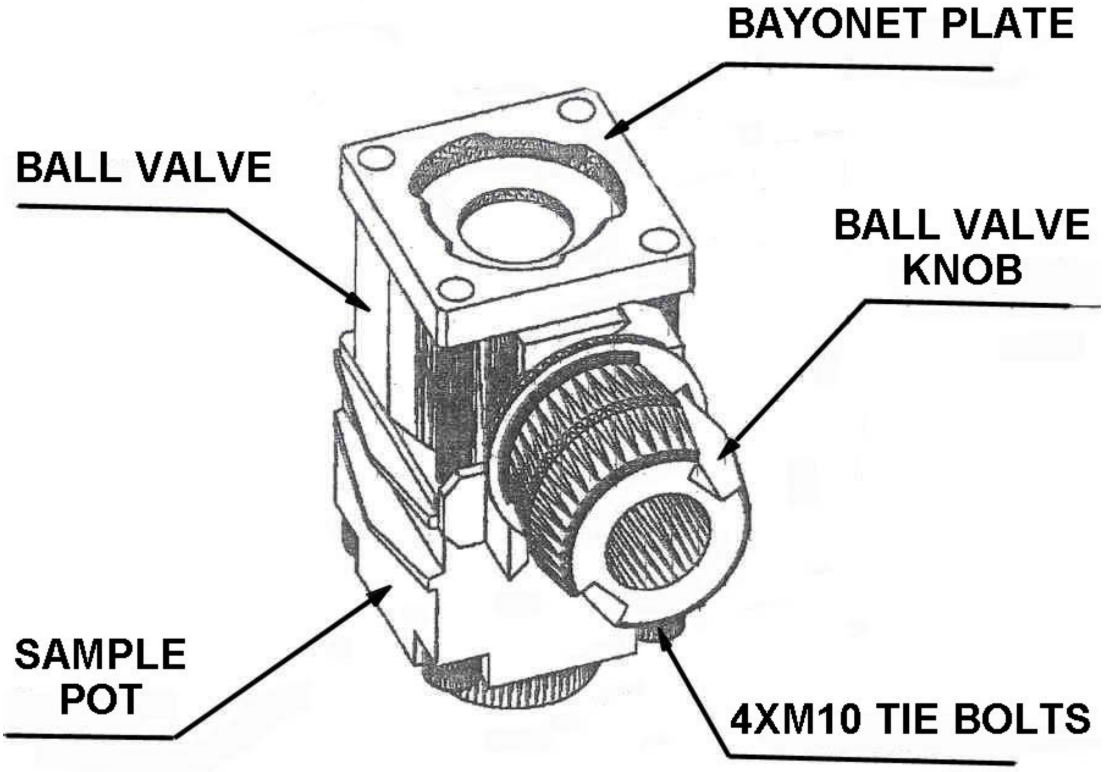

Fig. 9. Schematic representation of a sample pot.

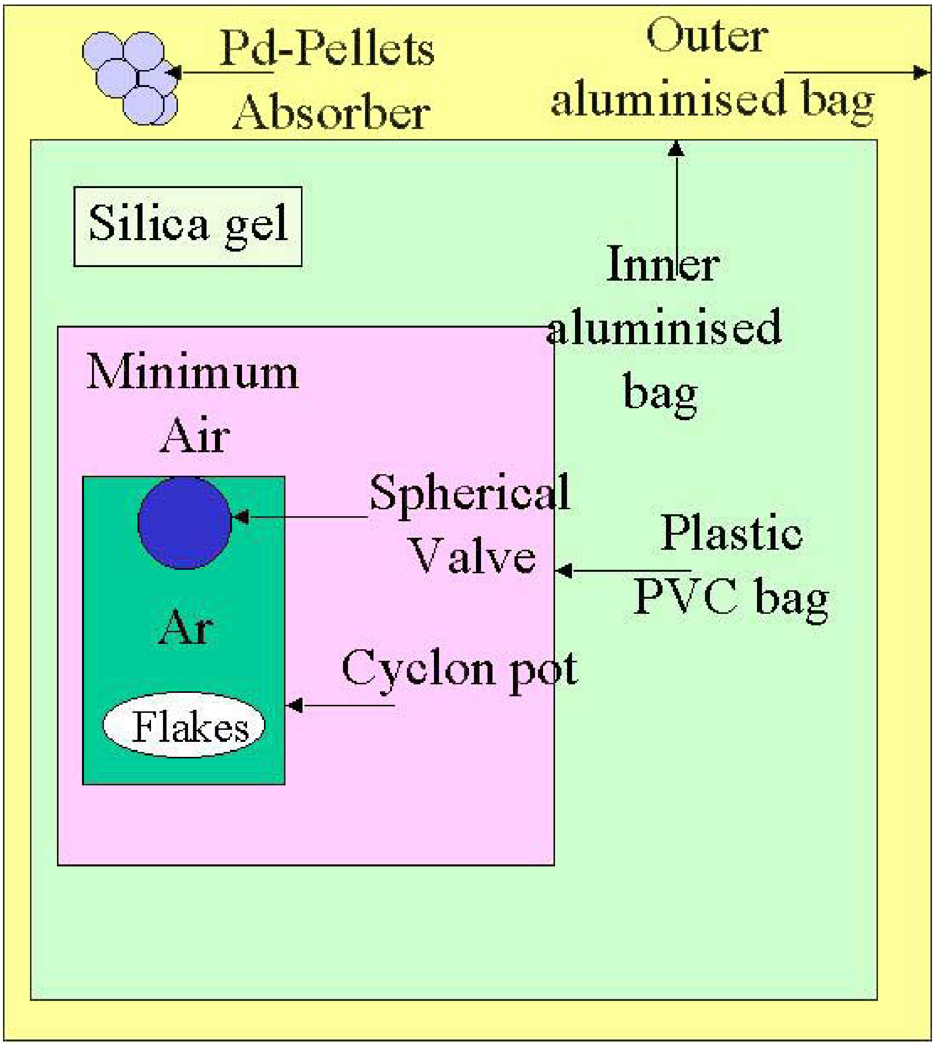

Fig. 10. The three bags packaging of the JET flakes.

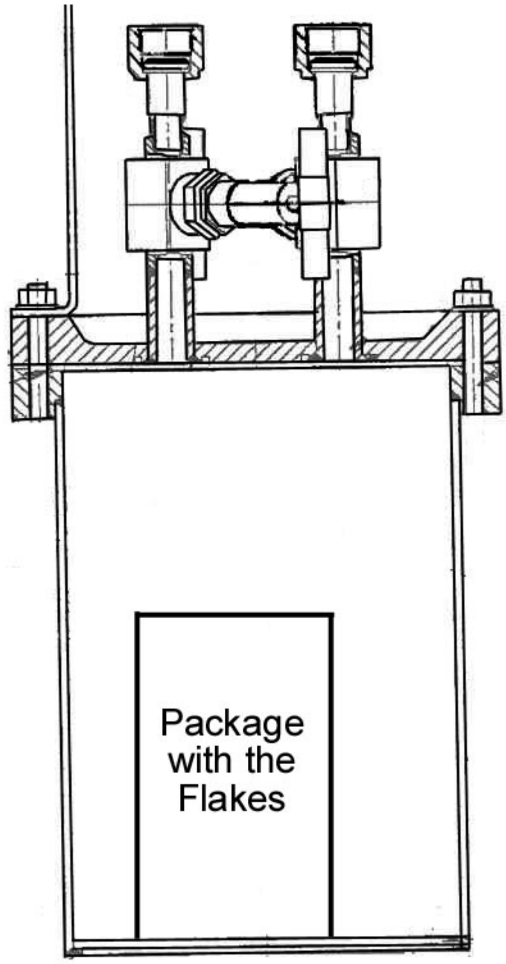

Fig. 11. The transport concept for the JET flakes.

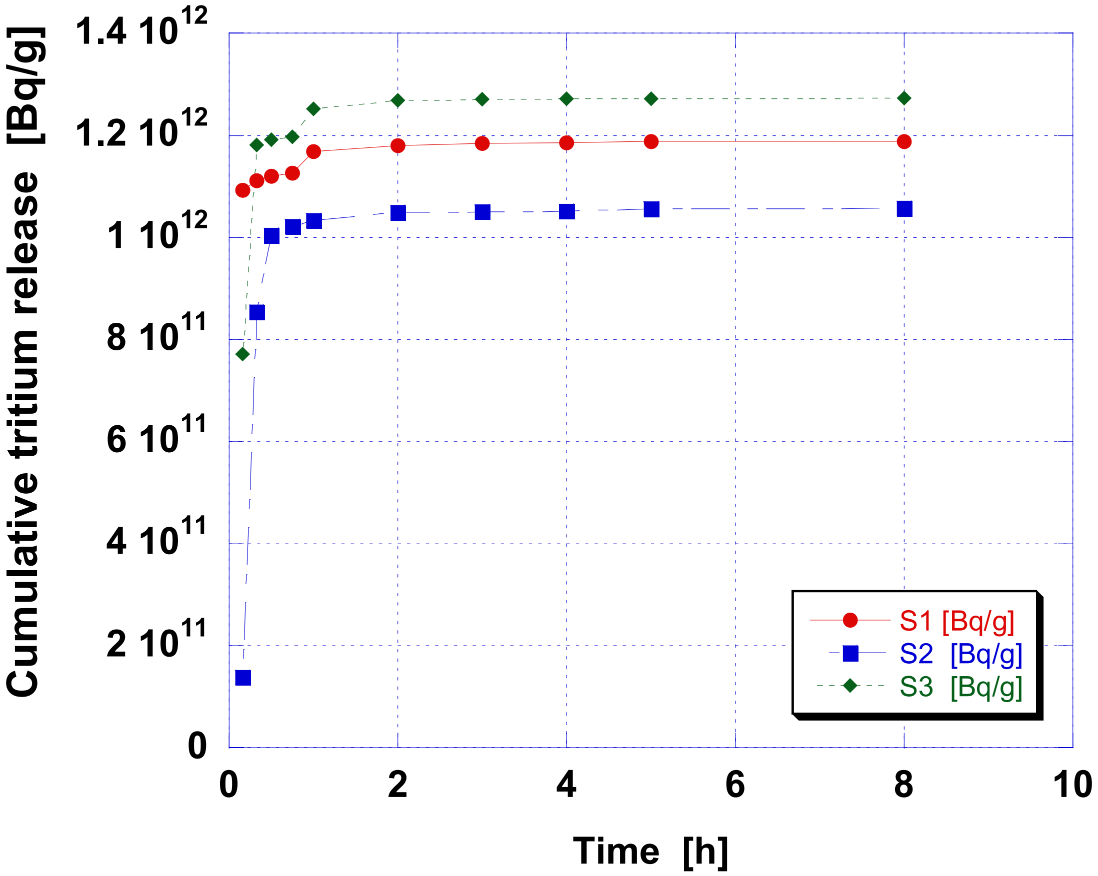

Fig. 12. Tritium content of three single flakes samples determined by heating under air at 800°C.

6. Literature

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