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Journal of Nuclear Materials 290–293 (2001) 245–249

journal of nuclear materials

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Dust characterization and analysis in Tore-Supra

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Abstract

Dust produced by the last 1000 shots in Tore-Supra has been characterized. The measured mean equivalent diameter is 2.7 μm, comparable to other tokamaks. The dust surface density varies from 2000 mg/m^2 on the bottom of the vessel to 15 mg/mm^2 on the remaining surfaces. The carbon dust sampling in Tore-Supra revealed some nano-structures which have been identified as fullerenes. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Carbon; Carbon deposition; Carbon erosion; Erosion; First wall materials; Tore Supra

1. Introduction

Dust production during D/T operation in a tokamak is a safety issue mainly for the dissemination of radioactive material [1]. The redeposition of the eroded material from a carbon first wall can trap large quantities of tritium in remote vessel areas. Chemical reactivity (for example, with water vapor at high temperature) can also be an issue because of the very large effective surface area of dust.

The quantification and the characterization of the dust produced in large tokamaks is a first step for safety engineering and for the understanding of the plasma–wall interaction. Sampling has been done in many large devices and the analysis tends to give a good physical and chemical description of eroded material from the first wall [2,3]. Tore-Supra is presently the third largest tokamak in operation which has the capability to operate during long pulse with the specificity of using an ergodic divertor and only RF heating.

Tore-Supra has a circular magnetic configuration which can be ergodized at the periphery by six resonant divertor modules. The operation mode is either in a limiter or a divertor regime.

In the limiter regime, the plasma can lean on the inboard toroidal first wall (actively cooled, 22 m^2 vessel coverage with 12 m^2 of polycrystalline graphite and carbon fiber composite (CFC)) or on a set of local pumped limiters (one horizontal, outboard semi-inertially cooled and made of pyrolitic graphite, four bottom, made of CFC tiles semi-inertially cooled).

In the divertor regime the plasma is limited on the outboard by the ergodic divertor modules which are covered with 12 m^2 of CFC tiles. Some field lines are also deviated on neutralizing plates made of CuCrZr water-cooled bars covered with B_4C.

After each major opening, the vessel is fully vacuumed before final closure, and therefore the dust collected is correlated with each previous experimental campaign. Characterization of the dust in Tore-Supra was done through different campaigns with a specific aim. This paper presents the first results on dust distribution and characterization.

2. Dust collection

Dust was collected on four different occasions.

The first campaign was done in July 1998, a few weeks after shot 24 856. Dust was collected with cotton

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swipes at the bottom of the vacuum vessel, between cooling tubes. After dissolution, the material was transferred on a transmission electronic microscope (TEM) grid for observation in FZJ/IWV laboratories (Jülich).

The second campaign took place in April 1999, during the long winter shut-down after shot 26 820. The collection was targeted on flakes deposited in two types of locations. The first one (A) is directly exposed to under the parallel ion flux in the scrape-off layer, the second one (B) is shadowed from the plasma, able to collect sputtered carbon (and other) atoms from carbon tiles. Collection was performed by scratching the surface with a metallic blade, which broke off the slightly adhesive flakes. The material was analyzed in SPAM laboratories (CEA Saclay) by means of a TEM and by chromatography.

The third campaign concerned water collected from a bottom vessel port, following the plasma facing component (PFC) leak which ended the 1999 experimental campaign (shot 28 353, 17 November) by a critical heat flux. 5 l of water which had washed the vessel locally were filtered through a 200 µm mesh and the alluvium were examined optically after drying.

The fourth campaign was aimed at characterizing the dust distribution around the vessel and took place in December 1999, three weeks after the water leak. Dust was collected via 0.02 µm anopore filters by controlled vacuuming on specifically identified areas in the vessel. 79 samples were analyzed for mass increase and particle size distribution. The collection was done following a poloidal pattern (12 samples) in each of the six vessel modules (Fig. 1) and also on seven other plug-in systems. This characterization was performed at INEEL laboratories in Idaho Falls and is part of a more extensive collaboration on dust characterization in Tore-Supra.

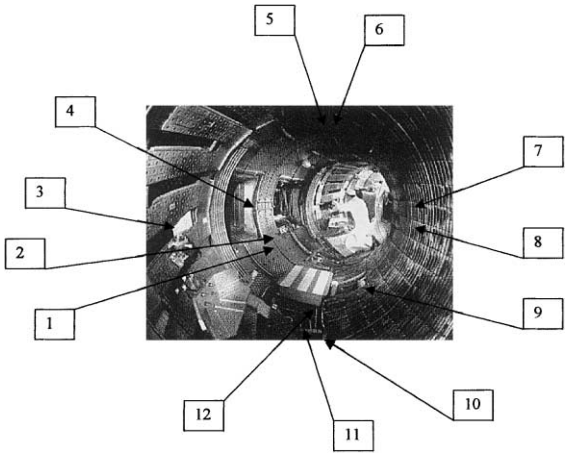

Fig. 1. Poloidal distribution for the dust sampling.

3. Microscopic examination

The material collected during the four campaigns was examined by optical means to identify the structures and characterize the particle distribution.

Fig. 2 is representative of the alluvium material collected in the water drained from a bottom port after a PFC water leak (third campaign). This material presents a very large discrepancy of size and is characterized by an equivalent diameter ($\varnothing_g$). One can identify a very large structure as a broken graphite tile ($\varnothing_g$ 6 cm, 50 g), other large parts of this tile ($\varnothing_g$ 1 cm) and the metallic washer, many other chunks of dark (carbon) and shiny (metallic) materials ($\varnothing_g$ 1 mm). Increasing the magnification reveals many particles with an equivalent diameter ($\varnothing_g$) in the range of 200 µm.

The collected flakes (from the first campaign) in Fig. 3 have also a broad $\varnothing_g$ ranging from 10 to 500 µm

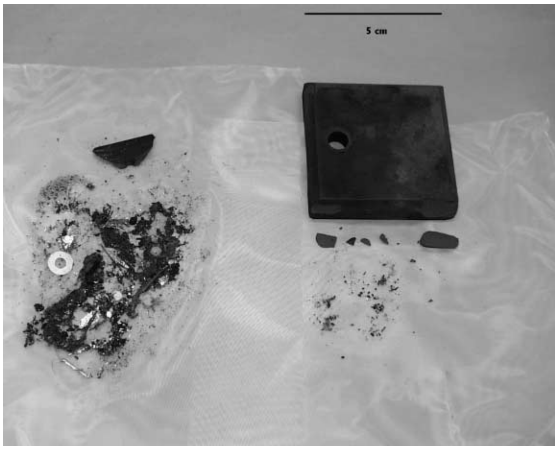

Fig. 2. Alluvium collected after a water leak on a PFC.

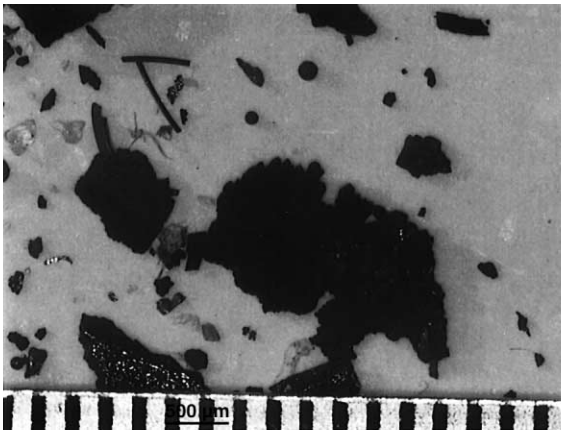

Fig. 3. Flakes collected in location A.

(optical microscopy). Smaller particulates were examined by TEM and revealed different types of structures. Aggregates of globular particles (with a rather regular $\varnothing_g = 15$ nm) are identified in the dust collected at the bottom of the vessel in the first campaign. They are assembled as filaments with an extension in the range of 200–600 nm. These microstructures have similitude with the ones obtained on the dust taken from a high heat flux (HHF) simulation test on graphite material [4].

The filamentary organization is rather similar but the globular shape is much more regular in the samples taken from Tore-Supra compared to the ones from the HHF simulation.

TEM examination on the flakes collected in positions A and B during the second campaign also revealed organized nanometric structures. Hollow large particulates, filaments and globular particles and hollow long structures are shown in Figs. 4 and 5. The carbon structure of the long particles in Fig. 5 can be described as a hollow segment with identical wall thickness, closed by spherical ends and therefore has many similarities with nanotubes. The other globular structure could probably be associated with other fullerenes. In order to ascertain this hypothesis, a specific extraction with toluene was done. Then a liquid chromatographic analysis was performed on a Waters Novapack-C_{18} column with a diode strip detector (UV–vis). As seen in Fig. 6, fullerenes (C_{60} and C_{70}) were clearly identified at 330 nm.

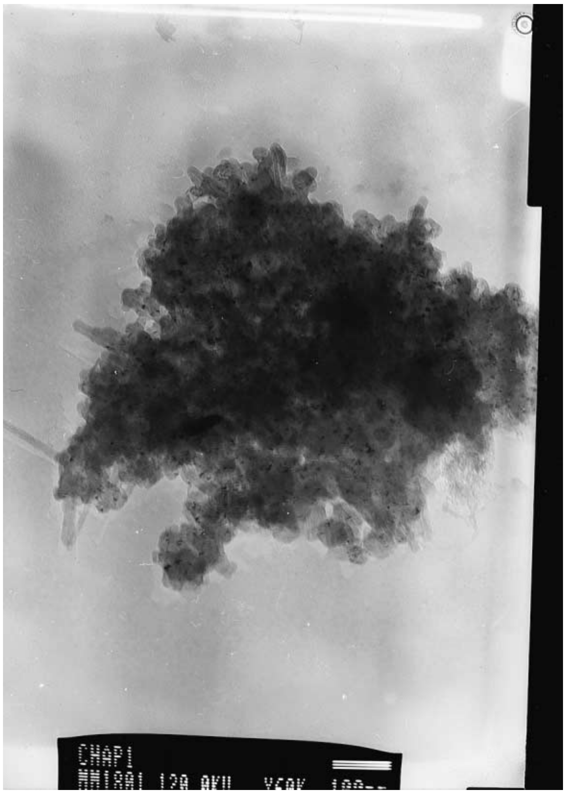

Fig. 4. TEM image of flakes: globular and elongated structures.

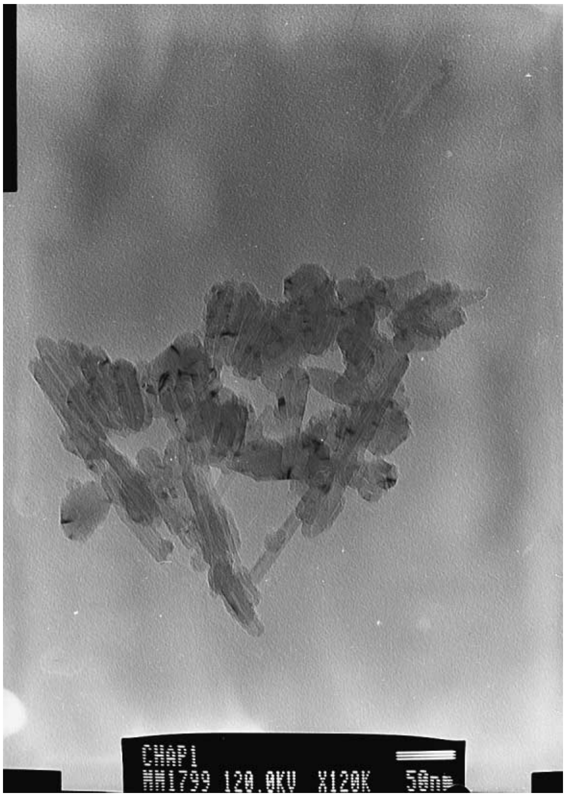

Fig. 5. TEM images of flakes: evidence of nanotubes.

The fourth campaign was more devoted to the distribution of dust in the vessel rather than the microscopic structure of the particles. The collected weight of dust on each filters has been normalized to the collection area. The bar graph in Fig. 7 gives the poloidal and toroidal distribution of this ratio. Particle size distribution was done on vessel modules I and II with an optical microscope, automatic image analysis and a statistical count base evaluation of a characteristic diameter (geometric mean diameter, GMD) with its standard deviation (GSD). These values are reported in Table 1.

4. Discussion

The dust surface density distribution is roughly toroidally symmetric (Table 1), except for an abnormal value (350 mg/m^2) in module I, location 1 which can be explained by the presence of melted copper from an LHH antenna in the neighboring port. Poloidally, the dust distribution follows gravity. On the bottom of the vessel, the density is close to 2000 mg/m^2 for location 11 (long nozzle sampling) and 500 mg/m^2 for location 12

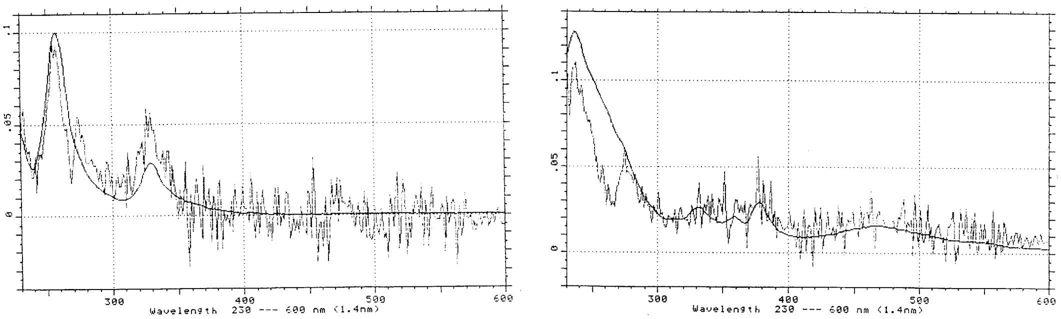

Fig. 6. Chromatographic detection of C_{60} and C_{70} fullerene structures.

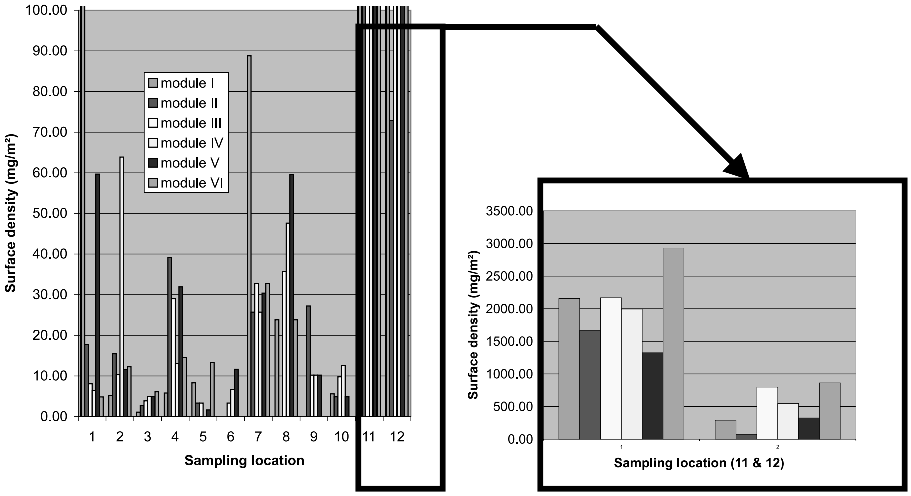

Fig. 7. Dust surface distribution in Tore-Supra.

Table 1
Mean particle diameter and standard deviation for dust collected at different locations

	Collection area (mm²)	Module I		Module II	
		GMD (μm)	GSD (μm)	GMD (μm)	GSD (μm)
1	620.0	2.2	2.8	2.4	2.4
2	1550.0	3.1	2.7	1.9	2.9
3	1800.0	3.1	2.7	3.2	2.7
4	689.0	3.0	2.9	2.5	2.6
5	1200.0	2.6	2.9	2.9	2.6
6	600.0	3.3	2.8	2.3	3.3
7	428.0	2.2	2.7	3.1	2.8
8	84.0	1.8	3.1	2.8	3.1
9	294.0	3.1	2.7	3.1	2.9
10	1430.0	3.1	2.7	3.2	2.6
11	160.0	1.3	2.4	1.5	2.7
12	480.0	2.0	2.8	4.7	2.7

(short nozzle sampling). The most realistic sampling must come from the long nozzle, the shorter one not begin able to touch the vessel between the tubes. The mean value for all the other surfaces 'seen by the plasma' is close to 15 mg/m². On the outboard wall (>200 mm from the LCMS), similar values are obtained from the panels lips and from the surface between the lips (15 mm gap) recessed 20 mm behind, suggesting a homogeneous 'vaporisation'.

Values measured on the inner first wall heat sink,between carbon tiles, on surfaces recessed 10 mm behind are higher (twice) than on the other vessel areas (except bottom), suggesting a larger redeposition close to the carbon surface interacting directly with the convective flux.

The dust collected on stainless steel surfaces (location 9) and carbon plate (location 10) have the same size (GMD $= 2.7$ μm) and standard deviation (GSD) of 2.8. The dust surface density is rather higher on the stainless-steel which is also close to a carbon interacting surface as the carbon plate (location 10) is only a remote protective tile for trapped electrons. Both locations are at the bottom of the machine and at the same distance from the LCMS.

Statistical analysis of the mean diameter for the dust particles from the fourth campaign follows a log normal distribution (from 0.02 to 100 μm). The GMD of the collected dust is very homogeneous for all the locations with a mean value of 2.68 ± 2.77 μm. This measure has to be completed by the observation on the alluvium collected from the water leak showing very large particles in the millimetre range and on the flake size in much larger range (nanometers to millimetres). The collected alluvium are certainly from a much larger surface and are weighted by the debris from the broken tile and the flakes were collected by scratching the surface because of their adherence.

The proportion of nanometric structures such as fullerenes has not been measured. Nevertheless these carbon structures are known for their very large capacity to store hydrogen and therefore could have a strong impact on the balance control of trapped species.

The GMD values measured in Tore-Supra (2.7 μm) can be compared to the GMD (count median diameter) of other dust sampling in tokamaks, thanks to the log normal distribution and the definition of the median (50% of the population). In DIID (low-*Z* PFC), the dust CMD ranges from 0.3 to 0.9 μm [5] and is rather similar to a sampling in Alcator (high-*Z* PFC), which ranges from 0.3 to 1.1 μm [6], and from TFTR (low-*Z* PFC) ranging from 1.6 to 2.7 μm [2].

5. Erosion estimate

If we assume that the carbon dust is mainly produced by the erosion from the PFC, an estimation of the total mass collected in one experimental campaign should compare with sputtering integrated on all the shots.

A total mass of 31 g has been estimated by assuming that the bottom of the vessel (15 m^2) is covered with an average of 2000 mg/m^2 of carbon dust and the remaining surface (85 m^2) with an average surface density of 15 mg/m^2. The last experimental campaign went from shot 26 915 (22 June 1999) to shot 28 353 (17 November 1999), totaling 13 400 s of plasma. An integration on all the representative shots (986) was done assuming the following formula:

$$m(g) = \left(\sum_{\text{shots}} \frac{V_{\mathrm{PL}} \langle n_e \rangle_{\max} t_{\text{shot}}}{\tau_{\mathrm{p}}} \right) Y_{\mathrm{C}} \frac{M_{\mathrm{C}}}{\aleph_{\mathrm{A}}},$$

where V_{PL} is the plasma volume (25 m^3), n_e the maximum plateau mean plasma density, t_{shot} the shot duration, τ_{p} a mean particle confinement time (200 ms), Y_{C} the sputtering yield for H and D below 100 eV on carbon (2×10^{-2}), M_{C} the carbon atomic mass (12) and $\aleph_{\mathrm{A}}$ is the Avogadro number.

The estimation of the eroded mass is 27 g which is surprisingly close to the measured (redeposition) value of 31 g.

6. Conclusions

The dust surface density varies from 2000 mg/m^2 on the bottom of the vessel to 15 mg/m^2 on the remaining surfaces.

The dust GMD collected in Tore-Supra is rather uniform (2.7 ± 2.8 μm) and comparable to other machines.

The carbon dust sampling in Tore-Supra revealed some nano-structures which have been identified as fullerenes. These structures could have a large impact on the hydrogen stored in the dust.

The total estimated carbon mass collected in vessel (31 g) is in the same range as an estimation of the eroded plasma facing material (27 g) for one testing campaign (986 shots).

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