# **Dust Particles in Controlled Fusion Devices: Generation Mechanism and Analysis**

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Abstract. Generation and in-vessel accumulation of carbon and metal dust are perceived to be serious safety and economy issues for a steady-state operation of a fusion reactor, e.g. ITER. This contribution provides a comprehensive account on: (a) properties of carbon and metal dust formed in the TEXTOR tokamak; (b) dust generation associated with removal of fuel and co-deposit from carbon PFC from TEXTOR and Tore Supra; (c) surface morphology of wall components after different cleaning treatments. The amount of loose dust found on the floor of the TEXTOR liner does not exceed 2 grams with particle size range 0.1  $\mu$ m – 1 mm. The presence of fine (up to 1  $\mu$ m) crystalline graphite in the collected matter suggests that brittle destruction of carbon PFC could take place during off-normal events. Carbon is the main component, but there are also magnetic and non-magnetic metal agglomerates. The fuel content in dust and co-deposits varies from 10% on the main limiters to 0.03% on the neutralizer plates. Fuel removal by oxidative methods or by annealing in vacuum disintegrates co-deposits and, in the case of thick layers, makes them brittle thus reducing the adherence to the target. Also photonic cleaning by laser pulses produces debris, especially under ablation conditions. The results obtained strongly indicate that in a carbon wall machine the disintegration of flaking co-deposits on PFC is the main source of dust.

#### 1. Introduction

Generation and in-vessel accumulation of carbon and metal dust are perceived to be serious safety and economy issues for a steady-state operation of a fusion reactor, e.g. ITER [1,2]. The major concerns are related to: (a) the risk of pressure rise and explosion under massive air or water leak on hot dust; (b) fuel retention in particles loosely bound to surfaces; (c) the degradation of performance of diagnostic components (e.g. first mirrors [3,4] and windows) or pumping components such as cryo-panels; (d) levitation of charged tritiated dust which may influence plasma performance [5]; generation of dust upon fuel removal from plasma-facing components (PFCs) [6].

The access to in-vessel components of a D-T reactor will be extremely limited. Therefore, in present-day machines one has to determine the amount, location and properties of dust. Equally important is to recognize the generation mechanisms and the impact of dust on plasma operation. The significance of such studies was recognized at TEXTOR already in mid nineties. Since then comprehensive dust surveys have been carried out on the occasion of major shut-downs which gave access to all in-vessel components [1,7,8]. The impact of in-situ PFC cleaning techniques on dust generation is also to be assessed. Oxidative fuel removal methods or annealing in vacuum disintegrate co-deposits and, in the case of thick layers, make them brittle thus reducing the adherence to the target. Photonic cleaning by laser pulses, especially under ablation conditions, also produces debris fine debris.

The intension of this contribution is to provide an account on: (a) properties of carbon and metal dust formed in the TEXTOR and Tore Supra tokamaks during campaigns comprising 4 - 24 h of plasma operation; (b) dust generation associated with removal of fuel and co-deposit from carbon PFC; (c) surface morphology of wall components after different cleaning treatments, such as photonic, thermal and oxidative.

# 2. Experimental: dust collection and analysis

The study was performed with materials retrieved from the TEXTOR and Tore Supra tokamaks after long-term campaigns. Loose dust particles and flaking co-deposited layers were collected during the opening of TEXTOR vacuum vessel in 2008. Sampling was performed from the bottom and the low-field side of the Inconel® liner, from the main graphite limiters such as pump toroidal belt ALT-II (Advanced Limiter Test II), poloidal and inner bumper, ICRF antenna grill, and from the bottom shield of the dynamic ergodic divertor (DED), as marked in Fig. 1. Vacuum pump with a series of filters was used to remove dust from the bottom of the machine. Deposits from the liner and limiters were collected using adhesive carbon stickers and ultra-fine copper nets for microscopy. Samples of co-deposits well adhered to the substrate were isolated by scraping them off from the target plates [9]. In the case of Tore Supra, samples of flaking co-deposits were obtained from the deposition zone on limiter tiles (Toroidal Pump Limiter, TPL) removed from the Tokamak in 2007 within the DITS program (Deuterium In Tore Supra).



Fig. 1. Sampling of dust in TEXTOR: deposition (1) and erosion (2) zones on ALT-II; bottom of the Inconel liner (3), main poloidal limiter (4), DED bottom shield (5), inner bumper limiter (6), ICRF antenna (7).

Observations of dust during the plasma operation were accomplished by fast cameras and spectroscopy. The material retrieved from the vessel was examined by means of scanning and transmission electron microscopy (SEM and TEM), energy dispersive X-ray spectroscopy (EDX), Rutherford backscattering spectroscopy (RBS) and nuclear reaction analysis (NRA) using a  ${}^{3}\text{He}^{+}$  beam in order to determine surface concentration and distribution of deuterium. The total fuel content was determined by means of temperature-programmed desorption spectrometry (TDS) based on the monitoring of masses 2(H<sub>2</sub>), 3(HD), 4(D<sub>2</sub>) and then higher than 18 for species of various hydrocarbons  $C_xH_yD_z$ . Samples were outgassed up to temperature of 1273 K. For samples with a high D/C ratio a long-term (70 h) annealing at 623 K was also performed in order check fuel removal effectiveness at the maximum baking temperature of the ITER divertor.

In order to study dust production caused by photonic cleaning two types of samples were subjected to laser irradiation: (i) vacuum plasma-sprayed tungsten coating (VPS-W) from the poloidal limiters used at TEXTOR [10,11] and (ii) tiles with flaking co-deposits from ALT-II. The characteristic of such layers can be found in [12]. A ruby laser ( $\lambda_{laser}$ =694.3 nm) used for ablation has a pulse duration of ~20 ns and the energy density in the range 1.5-17.5 J/cm<sup>2</sup>. Information on the composition of the gas phase was provided by a quadrupole mass analyzer (QMS) and visual light spectrometer, whereas other products are collected on a cylindrical dust catcher and studied later by means of NRA [13].

#### 3. Results and discussion

#### 3.1 Structure of dust

The collection of dust in TEXTOR (2008) has shown that the total amount of loose matter removed from the floor of the TEXTOR liner does not exceed 2 grams. The size of dust particles varies from 0.1  $\mu$ m to 1 mm. These are mainly disintegrated co-deposits detached from PFCs but larger debris (0.1 – 1mm) have most probably originated from the in-vessel mechanical work. The amount of fine matter (up to 100  $\mu$ m) is around 0.2 g. The real amount of such dust can actually be greater but ultra-fine dust could not be removed from PFC by vacuum cleaning due to a strong adhesion to substrate. The inspection of PFC indicates that most deposits stick well to the substrate. The majority of eroded carbon in TEXTOR is found in flaking co-deposits on the limiters (~ 90 g) as proven by ex-situ studies of components retrieved from TEXTOR [12]. Another place for a significant amount of deposited material is on the ALT neutralizer plates [8].

Images in Fig. 2 (a-c) show dust-forming flaking layers from the deposition zone on ALT-II limiter, liner and the main poloidal limiter, respectively. The layers on Fig.2 (a,b) are fairly flat and stratified, whereas co-deposits shown in Fig.2c have granular and columnar structure. Form of a co-deposited layer is related to the surface temperature difference on the corresponding wall components in TEXTOR: 300-350 °C in the deposition zone of ALT-II, 200-250 °C on the liner and up to 2000 °C on the surface of the poloidal limiter. As a result, the porosity and specific surface area of deposits differs. For instance, the specific surface area of columnar deposits formed on the neutraliser plates of ALT-II (maximum temperature over 2000 °C) was 24,2 m<sup>2</sup>g<sup>-1</sup> as determined with the BET method [14]. Similar granular forms have also been identified in the deposits from Tore Supra [15].

TEM studies accompanied with EDX analysis show that the major part of collected dust is composed of amorphous carbon with a presence of fine (nano-scale) crystalline carbon inclusions. Formation of crystalline carbon agglomerates may be related to the brittle destruction mechanism of PFCs during off-normal finding and then isolating a minute amount of crystalline matter from amorphous co-deposits.



*Fig. 2. Structure of flaking, dust-forming co-deposits formed on: (a) deposition zone of toroidal limiter ALT-II; (b) liner; (c) main poloidal limiter.* 



*Fig. 3. A metal droplet on ALT-II: (a) a general view of a metal splash; (b) and (c) magnified features of the re-crystallised zone with carbon co-deposits.* 

Apart from carbon dust, magnetic and non-magnetic metal agglomerates have been collected from the bottom of the liner and from the ALT-II. Large size (hundreds of micrometers) and elemental composition of these metal objects (Ni, Cr and Fe) indicate their origin from the Inconel liner or ICRF antenna grill. Some of them, as already pointed out, may originate from the in-vessel installation work, but the origin of several metal splashes found on ALT-II must be related to the transport of a molten material. An image of a splash is presented in Fig 3a. Careful analysis of such objects allows for identification of fine features, e.g. arc tracks, twisted traces and zones of re-crystallisation. It all indicates that a spherical rotated droplet "landed" on the tile and it still rotated during the re-crystallisation. The presence of an arc track on a droplet's edge gives possibility to find out the direction of magnetic field while the shape of a splash itself allows for making suggestions about the trajectory of the droplet. Angle between the velocity vector and magnetic field is  $\sim 76^{\circ}$ . While moving through plasma the spherical molten metal droplet was rotating with a rotation axis tilted relative to velocity direction and droplet's rotation did not stop after hitting the limiter. The twisted layers suggest that the original droplet was still rotating when solidifying. Carbon contamination at the tips of the protruding grains (Fig 3b, 3c), as proven by SEM and EDX analysis, appeared on top surface due to rotation, creating structures oriented in the direction of rotation. Very similar structures have been recently observed in studies of tungsten splashing [16].

#### 3.2 Fuel content and thermal release

The total deuterium content in dust and/or co-deposits isolated from PFC has been measured with TDS. Samples from different locations were heated up to 1273 K. The results for flakes from ALT-II, poloidal limiter and the antenna grill are shown in Table 1. In brief,

Location in	H	D	D/C
TEXTOR	[10 <sup>20</sup> at/g]	[10 <sup>20</sup> at/g]	
ALT-II tile	42.7	34.8	0.087
	43.8	31.1	0.079
Poloidal limiter	-	0.12	0.0003
ICRF antenna grill	8.3	6.6	0.015

**Table 1.** Retention of H and D in dust and co-deposites from various locations. The atomic density of carbon is  $4.5 \cdot 10^{22} \text{ g}^{-1}$ .

**Table 2.** Efficiency of fuel release during long term annealing of co-deposits from the toroidal pump limiters.

Limiter	T [K]	H <sub>2</sub> [%]	HD[%]	D <sub>2</sub> [%]
ALT-II	623	34.1	11.9	5.2
(TEXTOR)	1273	65.9	88.1	94.8
TPL	623	28.8	7.8	4.5
(Tore Supra)	1273	71.2	92.2	95.5

 Table 3: Deuterium-to-carbon ratio in the irradiated targets (limiters) and in the collected ablative products

I imitar	D/C ratio		
Limiter	Target	Collector	
<b>Toroidal: ALT-II</b> Deposition zone	0.08-0.11	0.02-0.05	
<b>Poloidal: VPS-W</b> <i>C-D co-deposition</i>	0.02-0.03	not detected	

the temperature of PFC surface affects not only the morphology of co-deposited layers but also the fuel content in such layers. Deuterium-to-carbon ratio in the deposits on ALT-II ( $T_{surf} = 520-570$  K) is in the range 8%-11% while on hot surfaces, i.e. poloidal limiter ( $T_{surf} > 2800$  K) D/C is below 0.1%.

Fuel inventory control and fuel removal are the major concerns for a D-T machine like ITER. One proposed scenario for fuel removal has been based on baking at 623 K. Due to engineering constrains it is a maximum baking temperature in the divertor [13]. The efficiency of fuel removal at this temperature was estimated for flaking co-deposits on ALT-II and TPL by a long-term outgassing at 623 K. The procedure comprised three steps: (i) quick temperature rise from 300 K to 623 K with a heating rate of 1 K s<sup>-1</sup>, (ii) annealing at 623 K for over 70 hours, (iii) temperature to 1273 for complete fuel desorption. Plots in Fig. 4 show the temporal evolution of fuel release during the start-up and final phases. Quantitative results in Table 2 indicate that about



Fig. 4. Example of temporal characteristic of fuel release during the long-term annealing of the ALT-II deposits from TEXTOR: desorption in the range 300K – 623K from a deposit from TEXTOR (a); desorption in the range 623K – 1273 K from deposits from TEXTOR (b) and Tore Supra (c).

5% of  $D_2$  molecules were desorbed from the samples at 623 K. Taking into account HD and hydrocarbons, not more than 10% of deuterium could be released at that temperature. In summary, the effective fuel

removal requires heating of the carbon layers to at least 1000 - 1070 K, while the release is completed at least 1273 K.

# 3.3 Fuel removal and dust generation by laser-induced ablation

Another approach to fuel removal is based on photonic cleaning of surfaces using lasers or flash-lamps. The use of a laser under ablation conditions results in fuel and codeposit removal. This is inherently associated with the generation of ablation products: solid debris and gaseous species [6,17]. Systematic studies have been undertaken in order to address these issues The procedure has comprised: (i) selection of targets from a reference graphite plate to real PFC from TEXTOR; (ii) test of irradiation conditions; (iii) developments of collectors of the ablation products; (iv) insitu monitoring of the gas phase by optical quadrupole spectroscopy and mass spectrometry; (v) ex-situ analysis of the targets and material liberated by the irradiation.

Plots in Fig. 5 are the spectra recorded during the first (Fig. 5a) and the fifth (Fig. 5b) laser pulse (energy density  $14 \text{ J/cm}^2$ ) on the ALT-II tile with a thick deposit. It clearly proves effective removal of the layer. To collect species liberated by the ablation several catchers have been developed and applied. The image in Fig. 6a shows a holder with the irradiated target (ALT-II tile) and a cylindrical catcher housing a flexible steel foil acting as collector of re-deposited gaseous species. The foil with the deposit produced during irradiation is shown on Fig 6b. Plots in Fig. 6c inform about the distribution of carbon re-deposited on the foils during irradiation of pure graphite, an ALT-II tile and a tungsten-coated limiter from TEXTOR; details of that tungstencoated limiter are in [9]. The analyses of carbon was carried out by means of NRA

with a 2.8 MeV <sup>3</sup>He beam. Deuterium content was determined in the same series of measurements. The results collected in Table 3 reveal that the released and then re-deposited gaseous species still contain fuel atoms. It is clearly visible for the products following the irradiation of the ALT-II tile. The D content on the catcher is not less then  $3.5 \cdot 10^{15}$  at/cm<sup>2</sup> and the corresponding D/C ratio of 0.02 along the inner side of the collector and up to 0.05 on the bottom of the cylinder. The irradiation of the carbon-based targets caused erosion demonstrated as a formation of craters. To assess the mass balance the laser-produced craters were studied with 2D and 3D profilometers and their volume was calculated. The efficiency of cylindrically-shaped dust collectors was estimated to be more about 90% with the main source of losses due to the opening for the incoming laser beam and a slit (see Fig. 6b) for spectroscopy measurements.



*Fig. 5.* Reduction of *H* (656.3 nm) and *D* (656.1 nm) line intensities during consecutive laser pulses at a rate 6 pulse/minute. Remaining lines at 657.8 nm and 658.3 nm belong to CII. Spectra are given for the first (a) and fifth (b) pulses.



**Fig. 6.** Dust collection in laser ablation experiments. a) a target holder with a reference graphite plate and a cylindrical collector; b) deposition pattern on a foil after irradiation of a reference graphite plate (80 pulses@14 J/cm<sup>2</sup>); c) carbon content along the cylindrical collector normalized to one laser pulse

#### 4. Concluding remarks

Dust particulates collected in tokamaks and generated by photonic cleaning of PFC have been studied. The results obtained strongly indicate that in a carbon wall machine like TEXTOR the

disintegration of flaking co-deposits on PFC is the main source of dust. Their structure (stratified or granular) is strongly related to the surface temperature of PFC on which flaking co-deposits were formed. The presence of fine crystalline matter on the bottom of the TEXTOR liner gives some indication that also brittle destruction of carbon tiles could occur. This is to be taken into account when assessing dust production in future devices if carbon PFC would be used. The issue of metal dust (beryllium and tungsten) formation is still to be properly addressed. It will be done in connection with ITER-Like Wall [18] operation of JET.

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