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# Analysis of carbon deposited layer growth processes in Tore Supra

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#### ABSTRACT

Deposited layers in Tore Supra are graphitic disordered carbons and contain spherical nanoparticles, proving the existence of homogeneous growth within the plasma edge, in an unexpected state of advancement. Evidence of homogeneous catalysis comes from the observation of graphite-encapsulated metal particles. Here, carbon particle properties such as temperature and ablation rate in tokamak plasmas are modeled with the DUSTT code, showing that growth can occur in the far scrape-off-layer during normal operation of Tore Supra. These particles can be generated either through plasma chemistry, thanks to efficient trapping in the scrape-off-layer due to sheath repulsion, or through arcing, thanks to transient optimal conditions for catalysis. Their observation on the neutralizer surfaces shows that they are then transported, avoiding destruction.

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#### 1. Introduction

Deposition of material eroded from carbon plasma facing components due to  $D^+$  bombardment causes carbon layers to be formed, attached more or less loosely to the surface, and containing varying amounts of D [1]. As a result, severe safety issues arise: (i) dust can form, due to plasma events stripping the deposits from the surface, (ii) when tritium is used in future, it will be retained permanently within these deposits [2]. During deposit formation, different types of species (pure carbon, hydrocarbon, metal, etc.) are eroded from the wall, migrate inside the plasma edge, are deposited onto surfaces, and can sometimes be re-eroded and redeposited [3]. The nature of the deposits therefore strongly depends on plasma edge conditions and on where deposits are formed. In order to control these deposits, a better understanding of the mechanisms behind them would be valuable.

This paper first focuses on deposit growth mechanisms, as revealed by transmission electron microscopy observations. The conditions of dust properties and survival in Tore Supra plasma edge are then analyzed. Finally, the discussion gives some clues to understand where and how deposits can be formed.

## 2. Ex situ characterization of deposits

Samples were scraped from carbon layers deposited on the leading edge of the neutralizers and on the vertical outboard lim-

\* Corresponding author. E-mail address: pascale.roubin@univ-provence.fr (P. Roubin). iter of tokamak Tore Supra. These deposits were thick layers (hundreds of  $\mu$ m) loosely attached to the surface, and their growth rate was estimated 20 nm s<sup>-1</sup> [4]. Their deuterium content was limited, less than 1% [5]. Their structure was analyzed in details [6–8], revealing that, contrary to what was observed in ASDEX for example [9], they were not amorphous and had local graphitic organization, though highly disordered. Scanning and transmission electron microscopies (SEM, TEM) were carried out at the CP2 M laboratory (Marseille, France) using the Philips XL30 SFEG STEM and the JEOL 2010F microscopes.

A representative SEM image of a powder grain in Fig. 1(a) shows the typical tip shape observed, all the tips being parallel. This orientation is the mean growth axis of the deposited layers and Fig. 1(b) shows that it is also that of the mean magnetic field, i.e. that of the mean incoming ion flux. Fig. 1(c) shows a tip whose top is broken, leaving an internal concentric shell structure clearly visible: this indicates that locally, growth is radial, which may be due to the local sheath electric field. It can be noted that the ion gyro-radius for D, or for example for C with Z = 4 (B = 3.4 T), the sheath length, and the scale length of the deposit structure are of the same order of magnitude (100–250 µm), and therefore local electric fields, including tip enhancement effect, could affect the deposition process. These general growth features are systematically observed for neutralizer deposits, indicating the existence of an intrinsic heterogeneous growth process on neutralizer surfaces.

Fig. 2(a) is an image of a spherical graphitic particle (nanoonion, diameter  $\sim 4-10$  nm) as frequently observed in Tore Supra deposits. The structure is most often a mix of concentric and lamellar organization, looking like onions and ribbons, respectively, if



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Fig. 1. Carbon deposits collected on the leading edge of Tore Supra neutralizers: SEM images of (a) tip shape, (b) a broken tip (the magnetic field direction is along the tip axis), and (c) photograph showing the tip orientation on the surface (for sake of clarity, surface and mean magnetic field directions are underlined).



Fig. 2. TEM images of Tore Supra deposits (a) spherical nano-onion (collected on the vertical outboard limiter) (b) metal particles embedded in graphitic ribbons and shells (collected on the neutralizers).

organization is advanced enough. Fig. 2(b) is an image showing frequently observed metal nanoparticles and graphitic ribbons or shells (typical size: 20–70 nm), also frequently observed. Metal particles can be encapsulated by these graphitic shells, revealing catalytic processes. The existence of these graphitic particles, with or without metal, reveals that, besides the heterogeneous growth onto surfaces, there is a homogeneous growth within the plasma phase in the plasma edge of Tore Supra, as already pointed out for TEXTOR [10,11].

## 3. Estimation of dust growth and destruction

Different plasma conditions were considered: MARFEs, glow discharges, and normal plasma operation. The ion density was estimated  $10^{19}$ – $10^{20}$  m<sup>-3</sup> and the electron temperature 1 eV during MARFEs, and they were estimated a few  $10^{15}$  m<sup>-3</sup> and 2–3 eV, respectively, during glow discharges. For normal plasma operation, three types of scenarios representing the Tore Supra data base are

considered here: low-energy (power level: P < 2 MW), moderateenergy lower hybrid ( $P \sim 2-4$  MW), and high-energy mixed lower hybrid and ion cyclotron resonance ( $P \sim 4-10$  MW) heating scenarios, the former being the most frequent ( $\sim$ 70%, 20% and 10%, respectively). The plasma parameters in the scrape-off-layer (SOL) display exponential profiles, with typical e-folding lengths of 3-6 cm. Followed from the Last Closed Flux Surface (LCFS) to the first wall, the plasma wets the Toroidal Pump Limiter (TPL) surface in the first 3.0 cm, wets the neutralizers in the next 3.5 cm, and finally, principally wets the bumpers. Note that the exact positions of the antennas and outboard limiters depend on the plasma scenario. Fig. 3(a) and (b) show the electron temperature and density as a function of the distance from the LCFS for the three types of scenarios, emphasizing the limiter, neutralizer and bumper positions. Typical densities at the LCFS are 0.2 and  $1.0 \cdot 10^{19} \text{ m}^{-3}$  and temperatures are from 30 to 60 eV. In the SOL, the ion temperature is  $T_i \sim 2-6T_e$  [12] whereas the fraction of carbon impurity ions  $\gamma_c$  is conservatively assumed to be close to that in the core discharge,



**Fig. 3.** Plasma and dust parameters during several scenarios of Tore Supra operation (TPL, neutralizer and bumper positions emphasized through color code): electron temperature  $T_e$  (a) and density  $n_e$  (b); calculated dust temperature (c) and destruction rate (d) with ( $T_e$ ,  $n_e$ ) plots superimposed.

estimated ~3% from  $Z_{\rm eff}$  measurements. The D neutral concentration and temperature were calculated using the EIRENE code [13] and were typically  $c_n \sim 5 \cdot 10^{16} \,\mathrm{m}^{-3}$  and  $T_n \sim 5 \,\mathrm{eV}$ , respectively.

Modeling of dynamics and transport of carbon dust in tokamaks was performed recently using computer simulations with the code DUSTT and was described in detail in [14,15]. Although both equilibrium and dynamic properties can be deduced from the code, the analysis focuses here on equilibrium properties of dust in edge plasmas, such as temperature and ablation rate. These properties were modeled taking into account kinetic and potential energy transfers due to absorption of electrons and ions, dust ablation, thermal radiation, and thermoionic and secondary electron emissions, including the effects of impurity ions and deuterium neutrals. Ablation was modeled taking into account chemical and physical sputtering by ions, radiation enhanced sublimation and thermal evaporation as described in Ref. [14-16]. It should be noted that these calculations may be not valid for very small particles (less than 10 nm) for which properties may be different to those of bulk.

Fig. 3(c) and (d) show calculations of dust temperature and dust destruction rate taking into account the  $T_i$ ,  $T_n$ ,  $\gamma_c$  and  $c_n$  Tore Supra SOL conditions. Dust stability and survival probability can be roughly estimated in Fig. 3(d) from the limits given by destruction rates: less than 1 nm s<sup>-1</sup> corresponds to very limited destruction by sputtering while more than 1 mm s<sup>-1</sup> corresponds to rapid sublimation. The region between 1 nm s<sup>-1</sup> and 1 µm s<sup>-1</sup> defines a regime where dust can dwell and the white region is a growth re-

gion. The  $(n_e, T_e)$  plots from Fig. 3(a) and (b) are superimposed in these figures, suggesting how growth and survival of carbon particles are possible during Tore Supra operation. For most of the scenarios, dust temperature remains below carbon sublimation temperature (Fig. 3(c)), and there is a regime permitting growth close to the first wall, whereas bumpers, neutralizers and most of the TPL surface are below the fast destruction regime (Fig. 3(d)).

## 4. Discussion

The TEM observation of nanoparticles has clearly shown the existence of different homogeneous growth processes in the plasma edge of Tore Supra. This growth is independent of the growth process leading to the tip shape, since nanoparticles can be found in different places in the machine where deposits do not have this shape. Moreover, these particles are not observed everywhere in the tip-shaped deposits and not necessary alone. It seems very unlikely that particles are generated during glow discharges since concentration in carbon species is very low and mean free path very large. The repartition of the deposits at the different surfaces of plasma facing components seems representative of standard operation, also probably excluding MARFEs or post-disruption phases as dominant mechanisms. On the other hand, our modeling has shown that during normal operation, carbon particles can grow far inside the SOL and dwell or survive closer to the LCFS, in particular near the neutralizers, where extensive carbon deposits are found.

Several scenarios can therefore be considered, taking into account generation processes and transport from the growth region to the deposition surface. Carbon particles can be formed through plasma chemistry processes, as in laboratory reactors when concentration in molecular precursors such as C<sub>2</sub>D species is high and when residence time is long. Negative precursors are frequently cited as a condition for this particle formation and, in plasma processes, trapping of these negative particles occurs thanks to sheath repulsion which balances ion drag [10,11,17,18]. This first type of growth could be at the origin of the carbon nano-onions. Cauliflower-like carbon particles (>100 nm), which result from coagulation and agglomeration processes in the plasma phase [11,17], were not observed, replaced here by the growth on neutralizer surfaces. A second likely process is a formation due to arcing at a metal surface. Liquid metal droplets can be formed and directly transported to the deposition surface. Carbon can also dissolve in these hot particles, then precipitate when cooling, leading to graphite-encapsulated particles. Generally speaking, metal catalysis is a way to produce graphitic particles at low temperature (down to 500 °C). Note that the calculated particle temperatures in the far SOL are from 500 °C to 1500 °C, and are fully consistent with the state of graphitisation observed. Particles observed on neutralizer surfaces are therefore probably formed close to the first wall and transport is thus fast enough to avoid destruction. The particle transport mechanisms are not known exactly and we give here estimations for the two cases, ballistic and diffusive, for a 100 nm particle submitted to a mean destruction rate of 100 nm s<sup>-1</sup>. In the case of ballistic transport, in order to survive, the particle should have a speed larger than  $\sim 5 \text{ cm s}^{-1}$  to arrive on the neutralizer (~5 cm distance): this value is significantly lower than that ( $\sim$ 10–100 m s<sup>-1</sup>) estimated by taking into account the various forces existing in tokamaks (drag of ion flows, electric force, centrifugal force, gravity, etc.) [15]. In the case of diffusive transport, the diffusion coefficient can be estimated through the Einstein expression  $D = kT_d/f$ , where k is the Boltzmann's constant,  $T_d$  is the kinetic temperature of the particle, and f its drag coefficient in the plasma [15]. In the case of the Tore Supra edge, this leads to  $D \sim 10^{-2} \text{ m}^2 \text{ s}^{-1}$ , also allowing more than 5 cm to be explored by the particle before being destroyed. Note that, not only transport, but also accumulation on the neutralizers results from the various forces acting on particles in the tokamak edge. In the case of the biggest particles which are very likely negatively charged, these forces can overcome the sheath repulsion.

#### 5. Conclusion

Homogeneous growth in an advanced state has been shown in the Tore Supra plasma edge through the existence of graphitic spherical nanoparticles, either small carbon nano-onions or large graphitic shells, with or without metal particles inside. DUSTT code simulations have shown that carbon particles can grow in the far SOL for the most frequent Tore Supra heating scenarios. Particle temperatures are consistent with graphitisation observed. This analysis therefore supports the hypothesis of generation of particles close to the first wall, due either to arcing or to plasma chemistry among species confined in the SOL, followed by transport fast enough to avoid destruction. For higher energy scenarios, both erosion rate and temperature are higher and this probably mitigates against homogeneous growth. To further clarify the deposit formation analysis, direct deposition onto surfaces of smaller compounds should be investigated in future.

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