Dust production from a carbon target exposed to a hydrogen plasma

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Laboratory experiments are proposed to understand the formation of dust particles collected in Tokamaks which walls are in carbon based materials. Because of a strong reactivity of hydrogen and carbon, hydrocarbon molecules are formed at the wall surface and released in the plasma edge as well as carbon by ion bombardment. Dust particles, expected in hydrogenated carbon composition, are produced in a hydrogen laboratory plasma were carbon atoms are injected by ion sputtering of a graphite target, negatively biased and located in the plasma. Hydrocarbon molecules formed at the graphite surface are released too. Preliminary results are presented about the influence of the sputtering rate and the graphite chemical erosion on their formation.

I. Introduction

Carbon-based walls of fusion devices (Tokamaks) undergo erosion through plasma particle bombardment. Redeposition of the eroded material onto the wall surface is not homogeneous and shows particulates with a wide range of size and shape: flakes, spheroids, filaments, aggregates, nanotubes\(^1\)…In these conditions, different mechanisms of dust formation have been proposed\(^2\): a) the flaking of thin films of the redeposited material, b) the condensation of C vapor originating from high heat loads and from arcing, c) the injection in the cold edge plasma of \(C_n\) clusters, d) the polymerization of negative hydrogenated carbon clusters\(^3\). The interest in their production is increasing because of safety reasons: dust particles absorb Tritium in D-T operations and make it mobile when devices are opened. Moreover, because of an increasing dust production with time, they are expected to induce Tritium loss during operation. Another aspect of their interest is that charged particulates in suspension in a sheath or a plasma produce a self-consistent redistribution of the surrounding electrons and ion fluxes\(^4\).

The aim of this work is to isolate a dust production way in a hydrogen plasma containing a) C atoms (ions) injected by ion sputtering of a graphite target and b) hydrocarbon molecules (ions) formed at the graphite surface. In this situation, the presence of the compounds \(CH_n\),
C$_2$H$_m$ are expected as well as their polymerization in C$_x$H$_y$ clusters (ions or neutral), as produced in methane, acetylene or ethylene dusty plasmas. A coagulation phase of these clusters could lead to spherical particulate synthesis in the plasma.

II. Experimental set up

The experiments are performed in a cylindrical magnetic multipolar device: 40 cm length, 30 cm diameter. The ionization is due to primary electrons emitted by heated tungsten filaments, negatively biased and located at the top of the device. Permanent magnets fixed on the outside wall increase the residence time of these electrons by magnetic confinement, allowing to work at low gas pressure (10$^{-5}$ - 2 10$^{-3}$ mbar), in a collisionless plasma. The working pressure used here is P $\sim$ 2 10$^{-3}$ mbar for hydrogen (electron density and temperature of n$_e$ $\sim$10$^{15}$ m$^{-3}$ and T$_e$ $\sim$ 2 eV, respectively) as well as for hydrogen mixed with 50% argon. In the plasma center, a graphite disc plate (10 cm diameter) is setting and negatively biased at -300 V to inject carbon atoms by ion bombardment. In improved situation, i) with hydrogen only, the ion flux density in the plate surface is $\Phi_1$ $\sim$ 8 10$^{18}$ m$^{-2}$ s$^{-1}$ corresponding to a power density of 400 Wm$^{-2}$ and ii) with hydrogen and argon, $\Phi_2$ $\sim$ 2 10$^{19}$ m$^{-2}$ s$^{-1}$ for a power density of 750 Wm$^{-2}$. Because of the sputtering effect and the presence of heated filaments, the plate temperature can reach 440 K, measured by a thermocouple. This value can be enhanced using a heating external power supply to produce graphite chemical erosion.

Several dust collectors (~ 1 cm$^2$) can be placed at different plasma locations but close enough to the carbon source and at the device bottom.

III. Preliminary results and discussion

Preliminary results are presented here, about particulate formation at low pressure. In this condition, the production rate is low. So, because of a weak density of dust particles produced in the plasma, they cannot be observed by laser light scattering. As a consequence, in a first time, we have chosen to stop every discharge after about two hours of operation, to analyze the dust collector depositions by Scanning Electron Microscopy (SEM).

For a given discharge, two kinds of particulate are observed: i) at the bottom of the device, spherical particles of diameter: 55 nm $\leq$ d $\leq$ 105 nm and ii) in the plasma volume, more or less spherical aggregates of particles i), of dimension: 750 nm $\leq$ s $\leq$ 1000 nm. The sizes of
i) and ii) particulates, depend on the experimental conditions as well as their density in the collector surface. In general, the structure of aggregates ii) is always well visible. This result suggests that at this stage of production, deposition on the particulates has not already occurred to smooth their surface.

In a general way, the dust production is increased in the following situations:

a) when argon is introduced in the discharge. Indeed, for a bias plate of -300 V, the carbon yield by H⁺ sputtering is \( Y_{\text{H}^+/\text{C}} \sim 0.01 \) and it is maximum\(^7\) while \( Y_{\text{Ar}^+/\text{C}} \sim 0.2 \). Thus, neglecting the hydrogen contribution, the carbon flux density injected into the plasma is:

\[ Y_{\text{Ar}^+/\text{C}} \phi_2 \sim 4 \times 10^{18} \text{ m}^{-2} \text{ s}^{-1} \]

adding the contribution of C and C⁺ component in the plasma.

b) when the temperature of the plate is increased (maximum of 640 K). Indeed, the graphite plate erosion can be enhanced by chemical reactions between carbon surface atoms and hydrogen atoms and ions. Their effect is to produce hydrocarbon molecules with binding energies low enough to allow desorption at the solid temperature. For instance, at 500 K, with a H⁺ flux density of \( \sim 10^{18} \text{ m}^{-2} \text{ s}^{-1} \) –conditions closed of ours- the total yield of hydrocarbon production\(^8\) (\( \text{C}_n \text{H}_m \), \( \text{C}_2 \text{H}_4 \), \( \text{C}_2 \text{H}_6 \), \( \text{C}_3 \text{H}_6 \), \( \text{C}_3 \text{H}_8 \)) is \( Y_{\text{chem}} \sim 2 \times 10^2 \), higher than the carbon yield due to sputtering by 300 eV energy hydrogen ions. Let us notice that in our conditions, the added Ar⁺ bombardment increases the plate temperature by 30%.

Because the particulate formation is assigned to the presence of radicals\(^3\) \( \text{CH}_n \), \( \text{C}_2 \text{H}_m \) produced by 1) chemical reactions between carbon atoms (ions) and hydrogen ions (atoms) in the plasma and 2) the break up of the hydrocarbon molecules (ions), released by the graphite surface, we have started a spectroscopy experiment in the visible range. The hydrogen lines or the hydrogen lines mixed with the argon lines being very intense, it is very difficult to observe the CH (\( \text{A}^2\Delta \rightarrow \text{X}^2\Pi \)) band with the Q(0,0) head band at 431.4 nm, appearing for instance, in methane plasma\(^9\). Lines of C I (\( \sim 600.11 \text{ nm} \)) are not observed too and are masked by the H₂ Fulcher band.

**IV. Conclusion**

Preliminary results about a dust production way in the nanometer size range are presented. They appear in a hydrogen plasma containing carbon and hydrocarbon compounds (neutrals or ions), injected from a graphite target by physico-chemical erosion. Spectroscopy in the visible range to analyze the gas composition seems difficult in our experimental composition. Nevertheless, in parallel, it is planed to analyze dust particles (outside the plasma) by Fourier
Transform Infrared (FTIR) Absorption spectroscopy. This diagnostic will give directly the carbon bonding type which is at the origin of the dust formation as well as information about the CH modes.

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