Dust collected in MAST and in Tore Supra

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Nanoparticle growth in laboratory plasmas

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Outline

- **Dust produced in the MAST tokamak**
  - quantitative analyses (mass)
  - qualitative analyses (shape, structure, composition)

- **Dust collected in Tore Supra neutralizers**
  - qualitative analyses (shape, structure, composition)

- **Carbonaceous nanoparticles growth in laboratory plasmas**
  - graphite cathode *sputtering* in DC discharges
  - microwave discharges in \( \text{Ar/CH}_4/\text{H}_2 \)

- **Conclusion** (similarities, differences)
Dust collected in the MAST tokamak

M6 campaign (2006-2007) = 2 x 2000 shots de 0.3 s

\textit{PFCs}: graphite, stainless steel

8 locations of collection by vacuuming below mid-plane. \textbf{3 groups of location}:

- \textbf{erosion-dominated locations} swept by strike points (foot of the central column, tiles)
- \textbf{private flux region} (dome)
- \textbf{shadowed locations} (toroidal gaps, ports, under Langmuir probe, upper surface of magnetic coils)
Dust collected in the MAST tokamak

- **Quantitative analyses**
  - outer toroidal gap of tiles + inner toroidal gap of tiles, \( m = (29.6 + 4.8) \text{ mg} \)
  - ports, \( m = 4.2 \text{ mg} \)
  - dome, \( m = 4.1 \text{ mg} \rightarrow 1.86 \text{ mg/m}^2 \)
  - tiles, \( m = 3.9 \text{ mg} \rightarrow 0.52 \text{ mg/m}^2 \)
    
    Total mass > 46.6 mg (2 \( 10^{18} \) atomes/s)

  ⇒ **Consistent with dust transport and formation**
  
  - dust transport towards shadowed areas
  - sweeping of the outer strike point towards the tile outer toroidal gap (dust transport)
  - private flux region = dome
  - eroded-dominated regions = tiles

- **Qualitative analyses:** SEM, TEM, HRTEM, EDX, IR absorption spectroscopy, Raman microscopy
SEM, TEM, HRTEM show everywhere:

1) metallic particles (nm to µm size)
   - arcing on vessel and magnetic coils (stainless steel)
   - metallic impurities during plasma ignition on inductive coils

2) carbonaceous grains (~ µm), irregular shape heterogeneous structure (amorphous to graphite-like)
   - coming from redeposited layers when they contain metallic impurities
   - otherwise, pulled out from divertors during disruption

3) carbonaceous nanoparticles, different structure (amorphous, onion-like)

   Evidence of homogeneous growth:
   - consistent with divertor plasma: dense and cold

Nanoparticles (5-10 nm)
Raman micro-spectroscopy

- for a given location, heterogeneous structure: amorphous carbon to disordered graphite
- the most amorphous grains located on the dome surface (private-flux region)

IR absorption spectroscopy

Dust in the outer toroidal gap of tiles (shadowed area):
- CD, CH, aromatic C=C, C≡C

Plan: Raman micro-spectroscopy currently done - correlation between the features deduced from spectra and the corresponding surface area collection
Dust in the Tore Supra tokamak

Deposits collected on the leading edge of neutralizers (PhD M. Richou)
• self-similar tips
• parallel tip-axes
• concentric shells
• porosity network

heterogeneous growth with specific features
- locally graphitic structure
- onion-like particles similar to carbon black
- graphite-encapsulated metal nanoparticles

**evidence of homogeneous growth in the edge plasma**
Nanoparticles produced in tokamak edge plasmas, divertor plasmas (TEXTOR, TS, MAST …)

Tokamaks with graphitic PFCs:

- sputtering (physical erosion)
- CH$_4$ release (chemical erosion)

2 stages of growth in cold plasmas:

1) **molecular precursors** (complex chemical pathway) $\rightarrow$ **nucleation**

2) growth of nanoparticles
2 examples of carbon nanoparticle growth in laboratory plasmas:

- **From cathode sputtering in DC discharges**
  - experimental set up
  - molecular precursors, nanoparticle growth

- **From hydrocarbon species in microwave discharges**
  - experimental set up
  - molecular precursors, nanoparticles growth
Glow discharges

• Argon = 0.6 mbar
• \( V_{\text{pol}} \sim -600 \text{ V} \)
• \( P \sim 100 \text{ W} \)
• \( J \sim 10 \text{ A/m}^2 \)

\[ N_e = N_i \sim 10^{10} \text{ cm}^{-3}, \quad T_e = 3 \text{ eV dans la LN} \]

⇒
• Emission spectroscopy
• Laser extinction and diffusion
Cathode sputtering

Thompson’s model (1968):
Sputtering from collisional cascade

EDF of sputtered carbon atoms
at the graphite surface, $\theta = 0^\circ$

$E_i = 100 \text{ eV}$

$E (\text{eV})$

$\Rightarrow$ Mean energy $\sim 11 \text{ eV}$

- cooling mechanism produces supersaturated carbon vapor $\Rightarrow$ condensation and carbon cluster formation, $C_n$

- modeling of neutral, negative cluster growth: $C_n, C_n^-$

K. Hassouni et al (LIMHP)

Taking into account collisions C-Ar
$= \text{carbon cooling mechanism}$

Thermalization with argon (200 °C)

Decrease of the sputtered atom energy
Experimental growth law

- Growth by cluster deposition

- Agglomeration of nanoparticles, 6-15 nm size + deposition
- Agglomeration of nucleus of 2-3 nm size + deposition

- Durée de la décharge (s)
- Taille (nm)
Microwave discharges

Bell jar reactor

**Ar/CH\textsubscript{4}/H\textsubscript{2} mixture**

- [CH\textsubscript{4}]: 3%, [H\textsubscript{2}]: 1%
- Total pressure: 200 mbar
- Microwave power: 400-600 W

\[ N_e \sim 10^{11} \text{ cm}^{-3}, \quad T_e \sim 1 \text{ eV} \]
Mechanism of Poly-Aromatic Hydrocarbons (PAHs) formation

Linearization

\[2 \text{H} + \text{C} = \text{C} = \text{C} = \text{C} + \text{H} \]

Cyclization

\[\text{H} + \text{C} = \text{C} = \text{C} = \text{C} + \text{H} \]

Hydrogen Abstraction Carbon Addition (HACA)

Condensation of 2 pyrene (A4) molecules

\[\text{nanoparticle nucleus} \]

\[\text{(1) Wang et Frenklach., Comb.Flame (1997)}\]
- **HRTEM micrographs**

- **In-situ IR absorption spectroscopy (diode laser):** presence of C≡C

- **Ex-situ IR absorption spectroscopy:** presence of PAHs with 3, 4 aromatic rings

- **Chromatography in gas phase:** presence of PAHs with 2, 3, 4 aromatic rings
Conclusion (1/2)

Nanoparticles produced in different plasma conditions have similar structure

1) carbon sputtering discharges:
low pressure, low input power

2) Ar/CH₄/H₂ discharges:
higher pressure, higher input power

3) Tore Supra (limiter tokamak)
4) MAST (divertor tokamak)
But, in the considered laboratory plasmas, molecular precursors are different:

1) carbon sputtering discharges: $C_n$ , $C_n^-$

2) Ar/CH$_4$/H$_2$ discharges: complex chemical pathway $\Rightarrow$ PAHs

Confirmation:

1) Chromatography  
2) IR absorption spectroscopy  
3) Mass spectrometer (plan)

Molecular precursors in Tore Supra and MAST?

1) IR absorption spectroscopy: $\Rightarrow$ TS neutralizers deposits : flat spectra  
   $\Rightarrow$ MAST: one spectrum (CD, CH, C≡C, C≡C)

2) Mass spectrometer (Tore Supra)

3) Chromatography analyses?