SYNTHESIS of NANOPARTICLE of CARBON

First results

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Context

Tribology → **the science of interacting surfaces in relative motion.** → **Friction** / **Wear** → **Lubrication** → Reduces friction / Protects surfaces

Lubricant ← **Base**: **mineral or synthetic oil** + **Additives**: **friction reducer**, **anti-wear**, **anti-oxidant**....

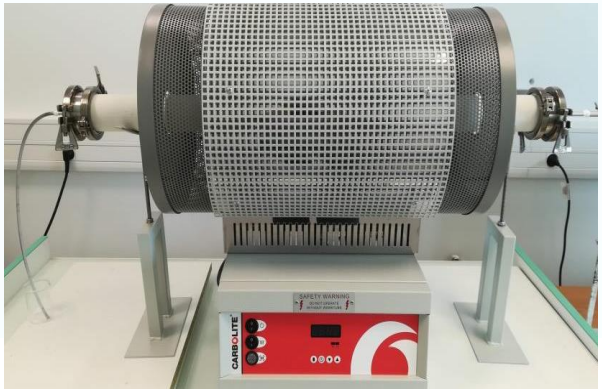
graphitic nanostructures have interesting friction-reducing properties

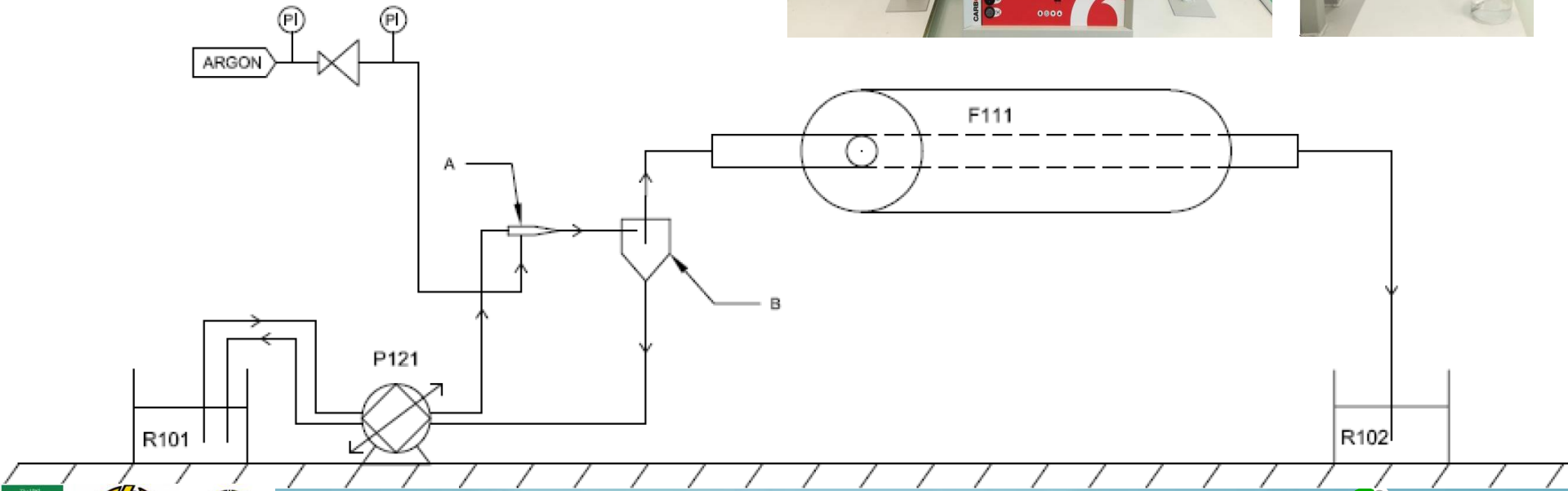
Objective :

- Synthesis of **porous carbon nanoparticles** from a sucrose solution
- Determination of their tribological properties

Technique for the synthesis of carbon nanoparticles

R101: Sucrose solution beaker
R102: Demineralized water beaker
F111: Tubular furnace STF 16/180-Carbolite Gero
P121: Peristaltic pump
A: Concentric nebulizer
B: Cyclonic chamber





Technique for the synthesis of carbon nanoparticles

Operating conditions

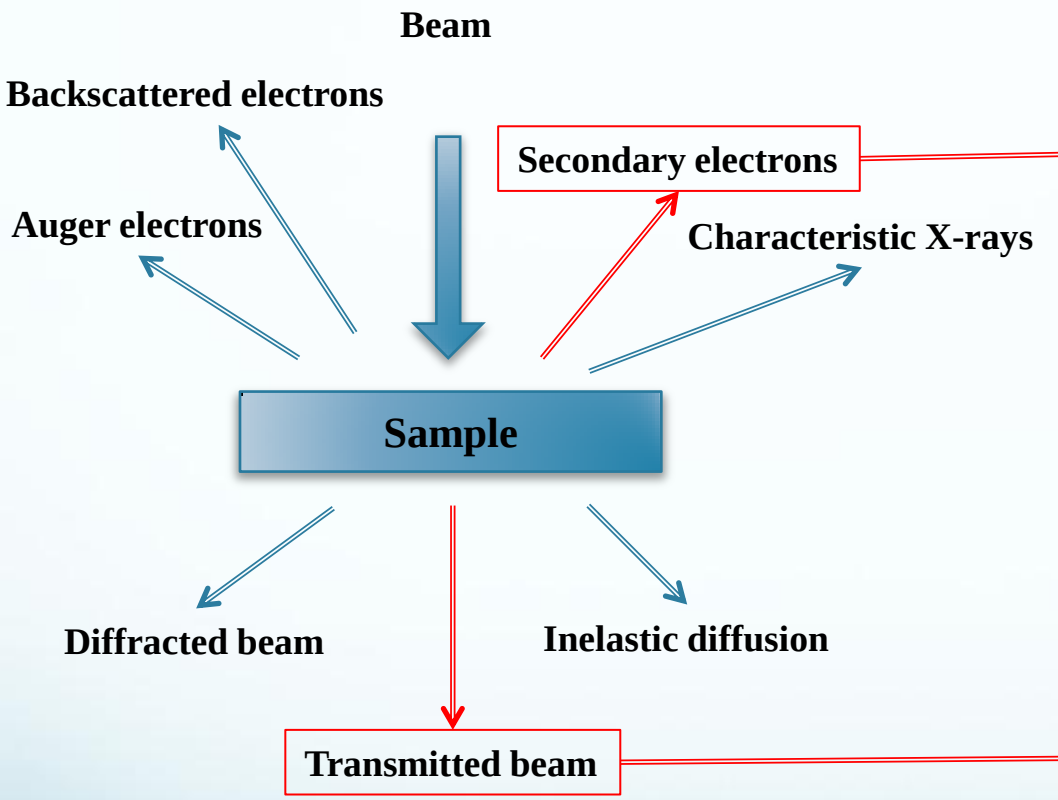
Carbonaceous source	Sucrose	C sucrose = 0.5 mol/L		
Catalyst	NaHC03	C = 0.1 mol/L	C = 0.5 mol/L	**C = 1 mol/L**
Carrier gas	Argon	Pressure from 0.5 bar to 3 bar		
Pyrolysis temperature		800°C	900°C	1000°C
Flow rate		2830.7 µL/min (80 rpm)		
Duration		From 20 mn to 60 mn		

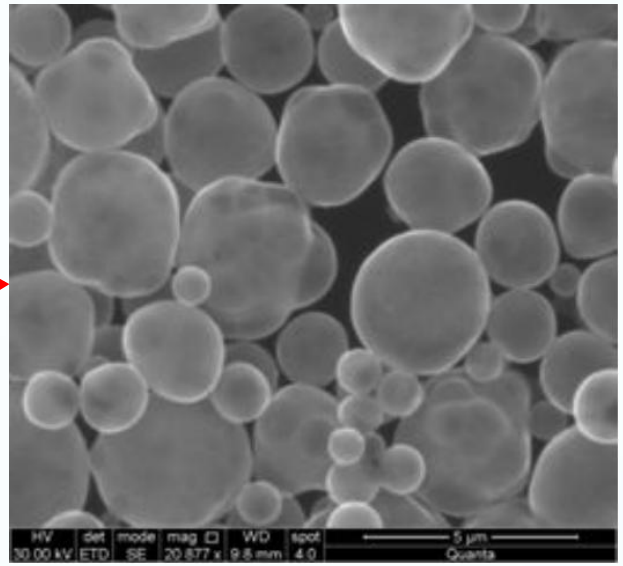
NaHCO3 acts as a porogen

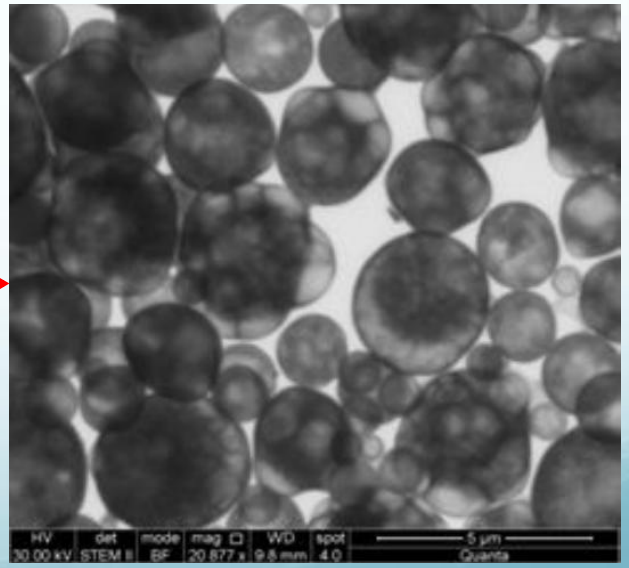
Visible particles at 1 mol/L

Observation of carbon nanoparticles

Scanning Transmission Electron Microscope







Synthesis conditions optimization

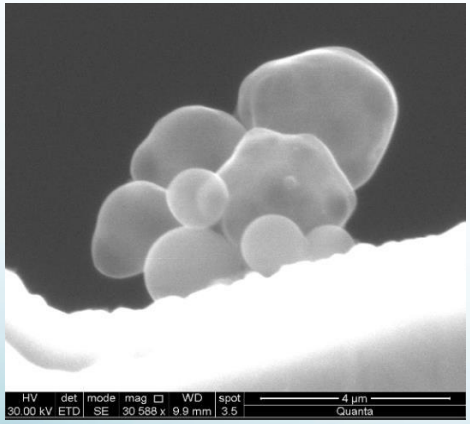
Variation of argon pressure

Pressure (bar) / In the beaker	0.5	1	1.5	2	3
On the surface	-	+	++	++	+++
In suspension	-	-	+	++	++
At the bottom	-	-	+	+	+++

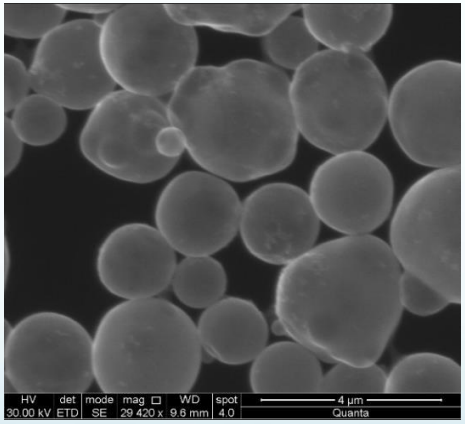
- No visible particles + Some isolated particle ++ Many dispersed particles +++ Particle cluster

Increase of argon pressure ➡ more particles

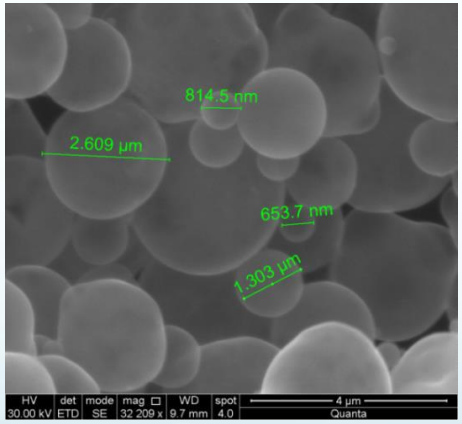
Scanning Electron Microscope



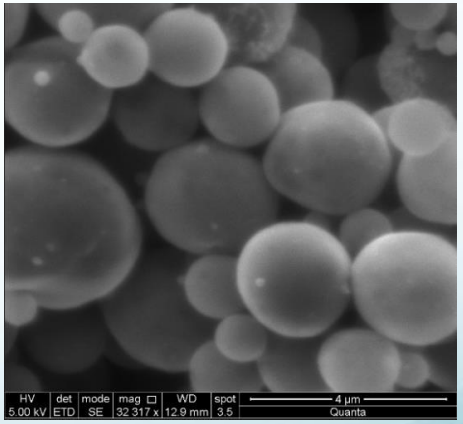
1 bar



1.5 bar



2 bars



3 bars

Particle diameter between 400 nm to 3000 nm

Spherical particle aggregates

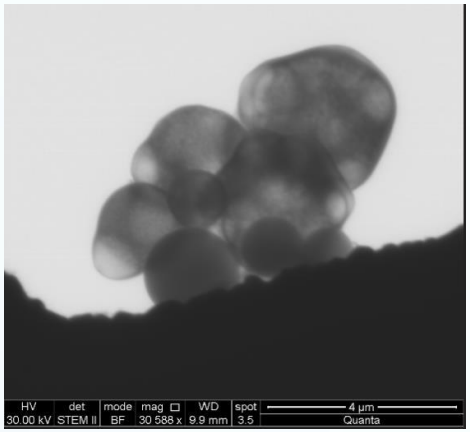
Increase of argon pressure ➡ increase of sphericity

Synthesis conditions optimization

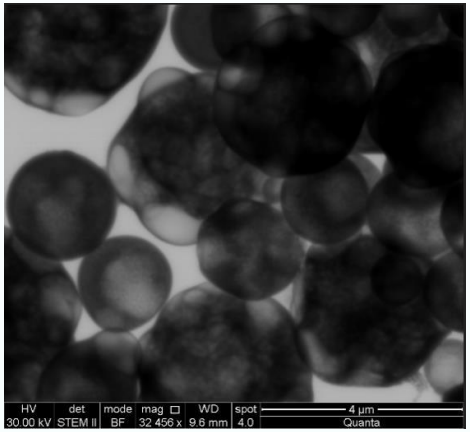
Variation of argon pressure

Scanning Transmission Electron Microscope

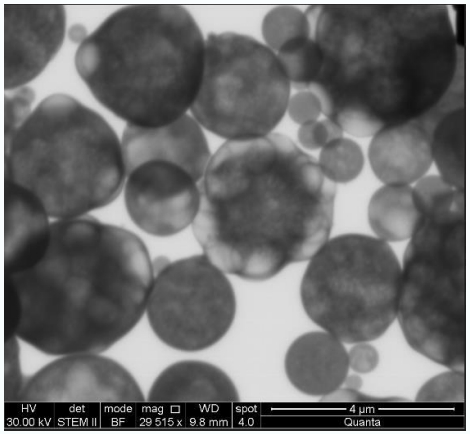
synthetized at 800°C



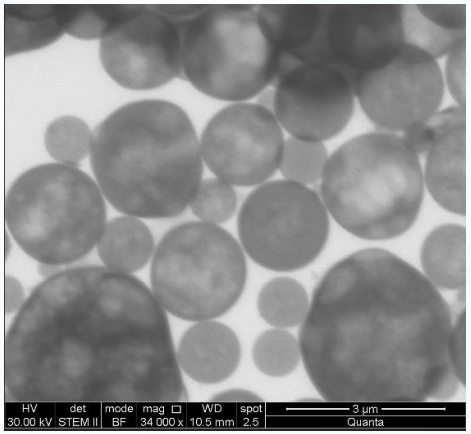
1 bar



1.5 bar



2 bars



3 bars

Difference in density ⇒ porosity

Biggest particles ⇒ aggregates of smaller structures

Increase of argon pressure ⇒ Decrease of the particles diameter

Synthesis conditions optimization

Pyrolysis temperature

Synthesis at 3 bar

Temperature (°C)	800	900	1000
In the beaker			
On the surface	+++	+++	++
In suspension	++	++	+
At the bottom	+++	++	+

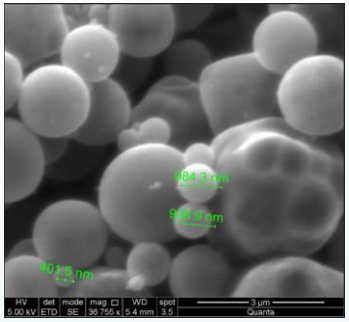
800°C

900°C

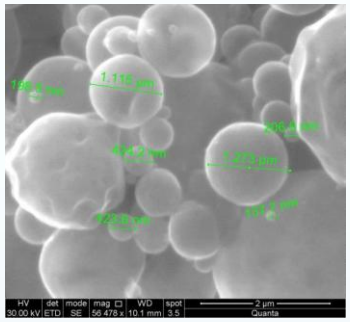
1000°C

Increase of pyrolysis temperature ⇒ Less particles

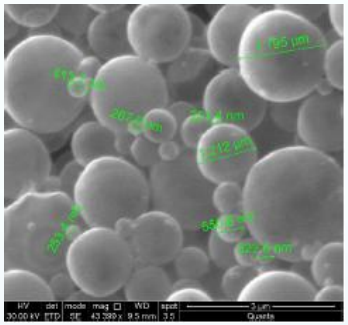
Scanning Electron Microscope



800°C



900°C



1000°C

Temperature	Higher number of particles
800°C	1000 nm – 2000 nm
900°C	700 nm – 1200 nm
1000°C	400 nm – 1000 nm

Increase of pyrolysis temperature

⇩

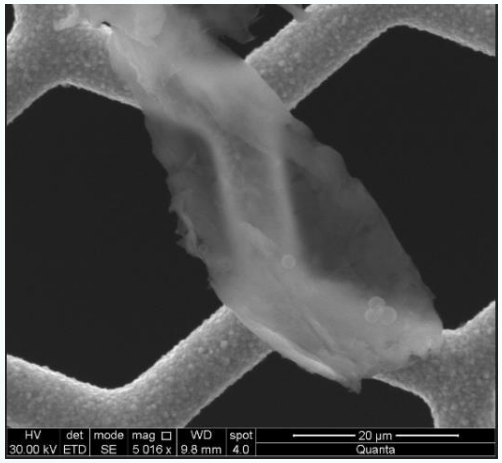
decrease in average diameter

⇩

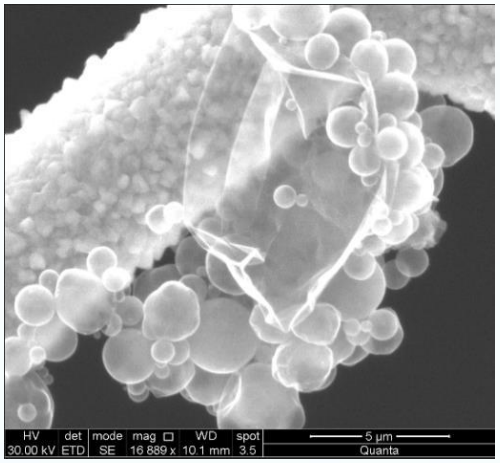
more volatile particles

Synthesis of carbon nanoparticles

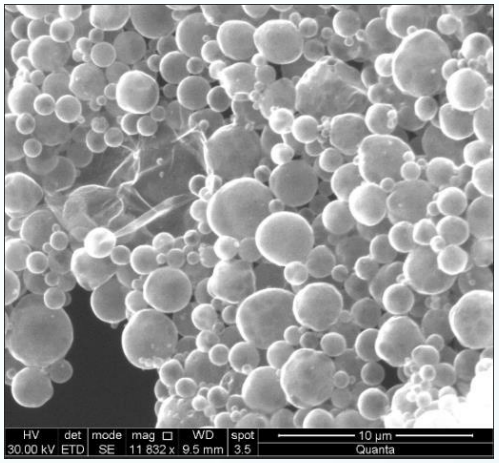
Scanning Electron Microscope



800°C 2bars



900°C 3bars

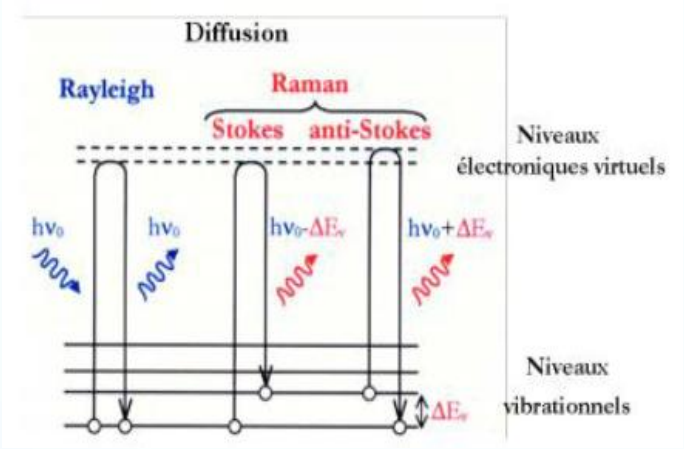


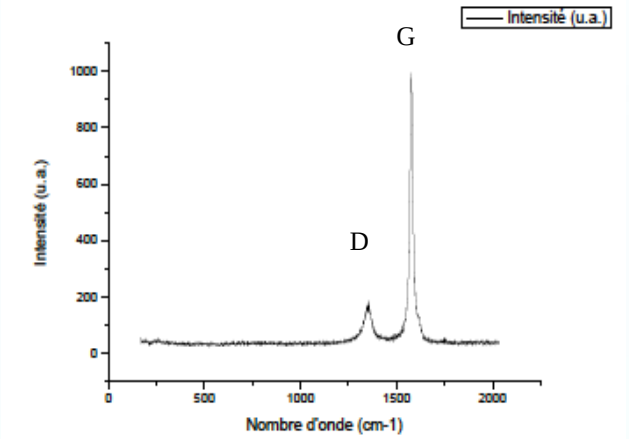
1000°C 3bars

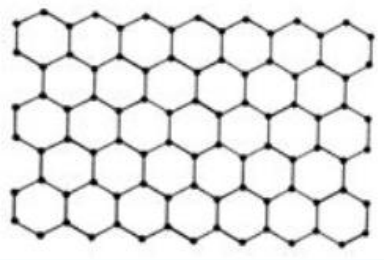
Other structure : carbon film

Particle analysis

Raman Spectroscopy



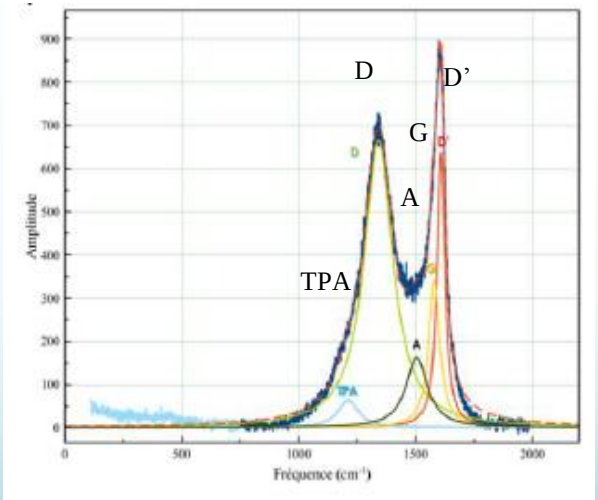


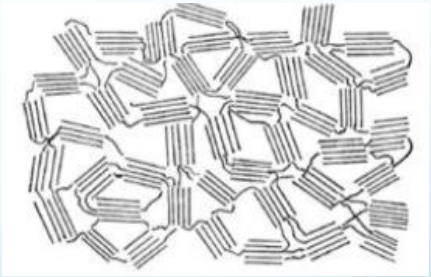


Graphite

Raman peak deconvolution*

G band : sp^2 carbon
D and D' band : sp^2 defect
TPA band : carbon skelton (transpolyacetylene)
A band : amorphous part



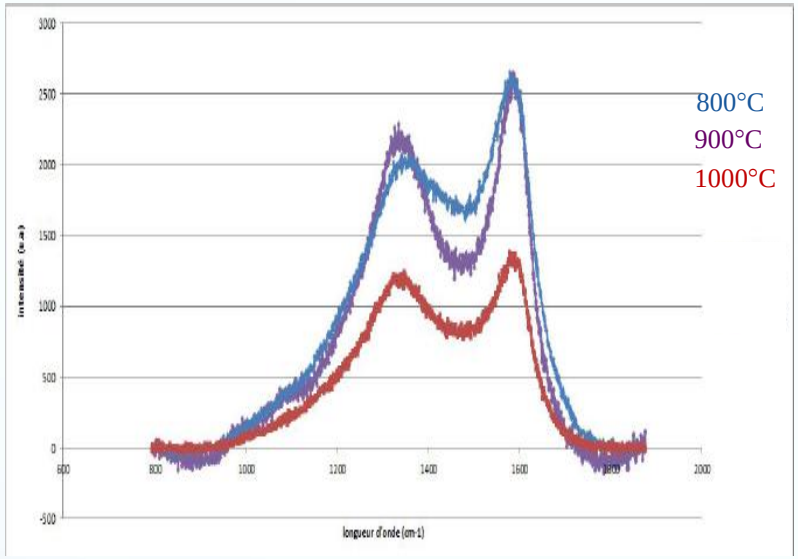


Activated carbon

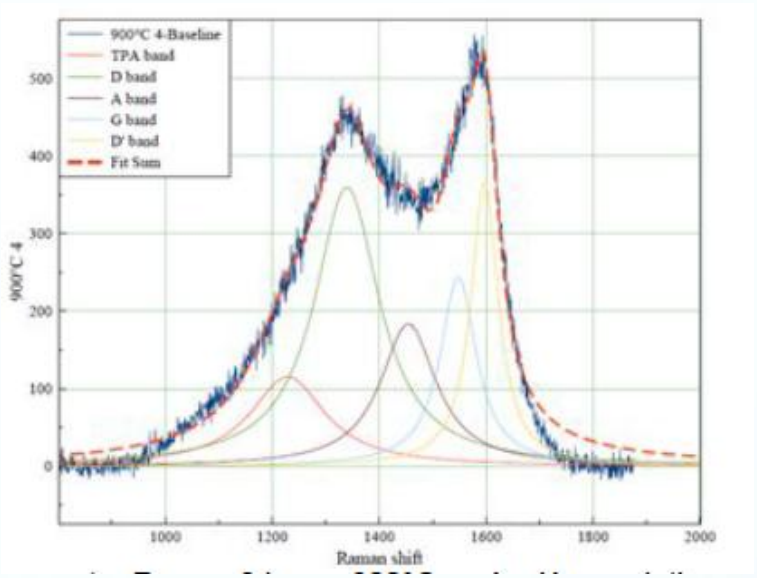
*Cheng Hu , Saeid Sedghi...Raman spectroscopy study of the transformation of the carbonaceous skeleton of a polymer-based nanoporous carbon along the thermal annealing pathway - ScienceDirect- 2015

Particle analysis

Raman Spectroscopy



Amorphous carbon



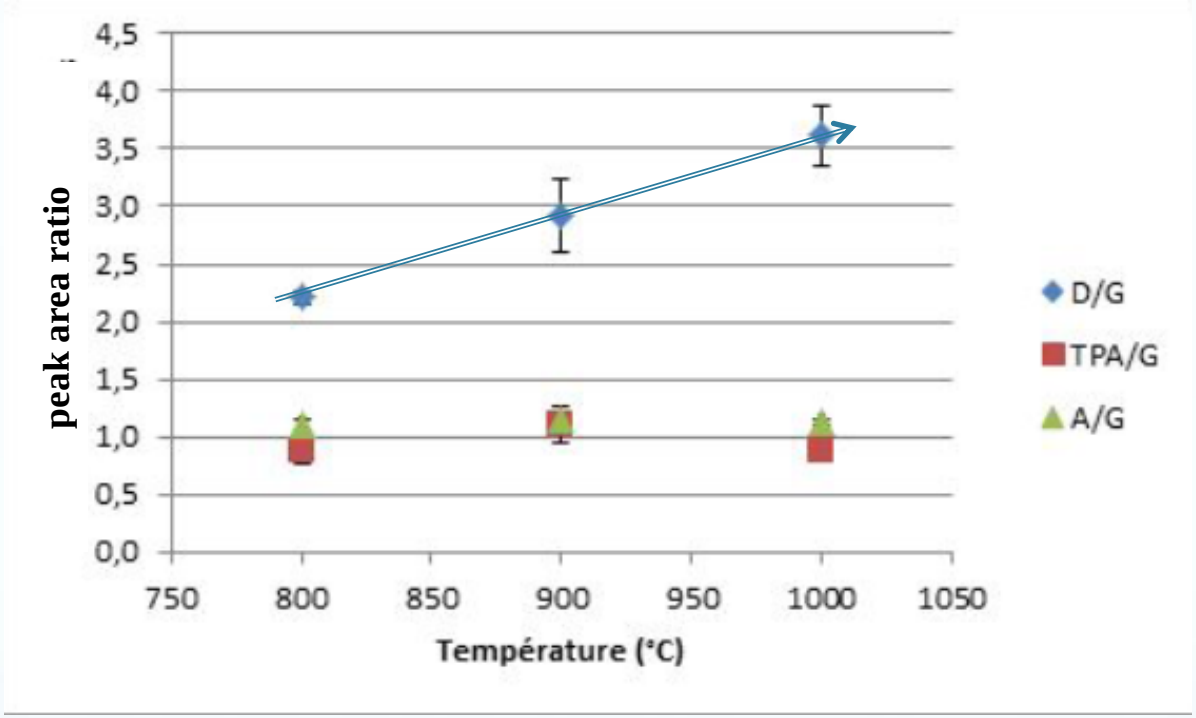
Raman spectrum deconvoluted

G band :1600 cm^{-1}
D band :1350 cm^{-1}

Beginning of low graphitization

Particle analysis

Raman Spectroscopy

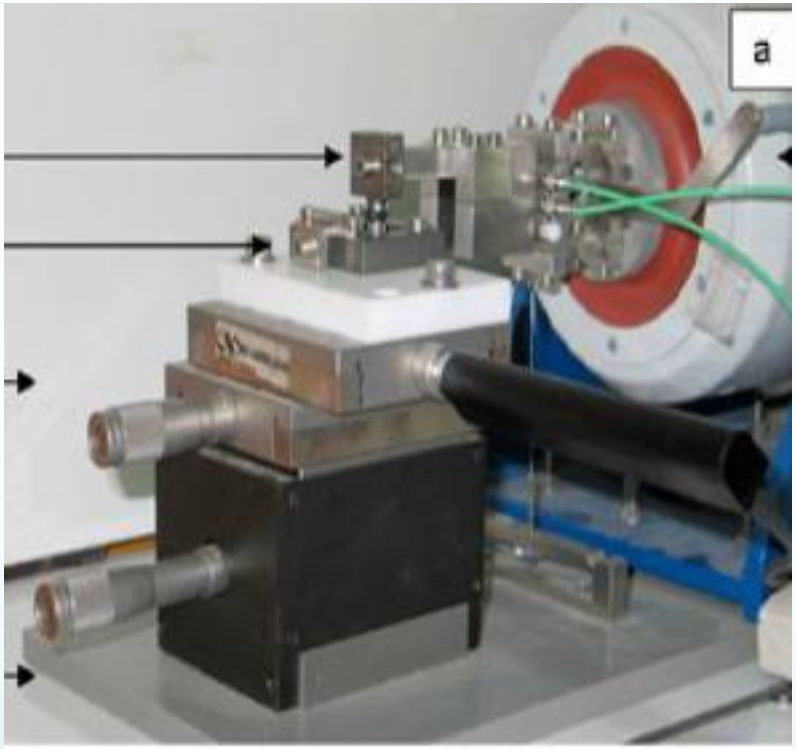


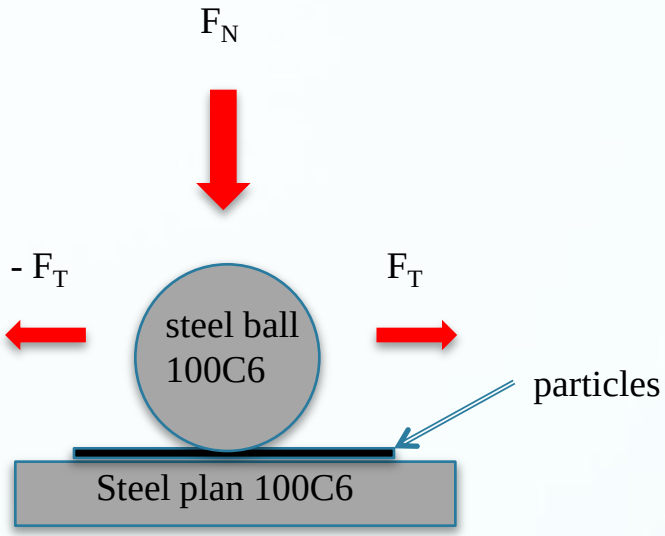
Apparent decrease in order in the carbon structure

Probably due to the increase in the number of small particles

Particle analysis

Tribological behaviour



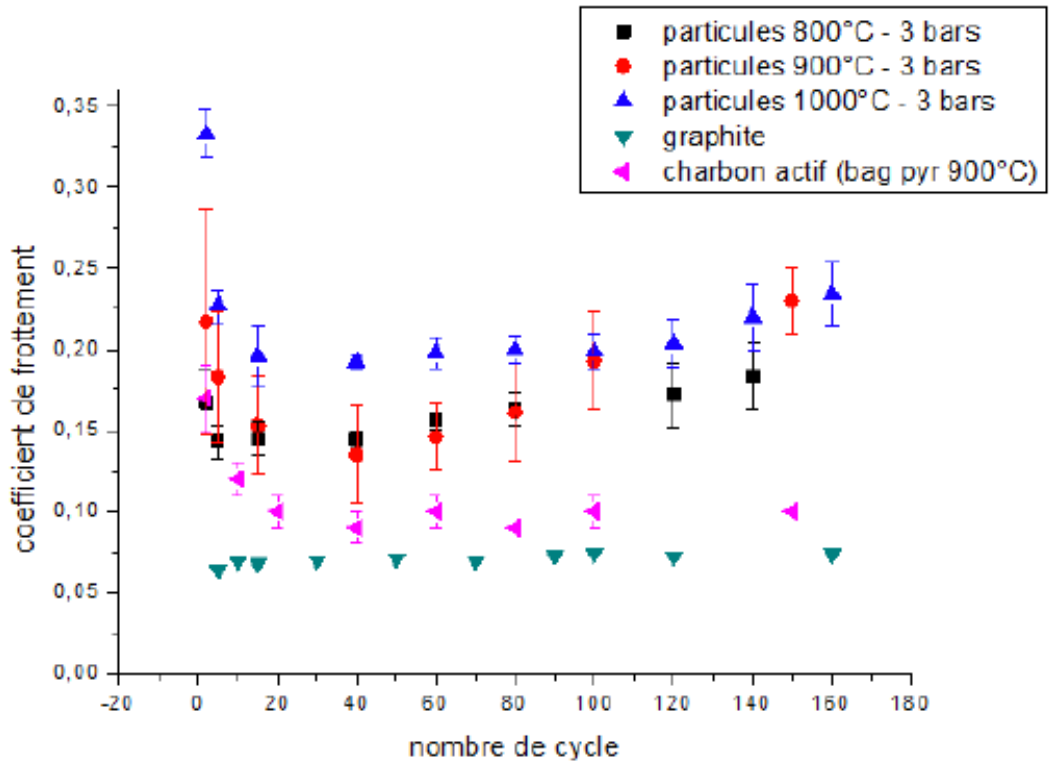


friction coefficient : $\mu = \frac{\| F_T \|}{F_N}$

- Normal force applied : 10 N
- Number of cycles : between 35 and 250

Particle analysis

Tribological behaviour



- Poor film holding
- induction period : 15 cycles
- friction coefficient :
  - graphite : **μ =0.07**
  - activated carbon : **μ= 0.09**
  - 800°C and 900°C : **μ =0.15**
  - 1000°C : **μ =0.2**

Increase of order at great distance ⟹ **Reduction of the friction coefficient**

Temperature increase ⟹ **Increase in friction coefficient**

Conclusion

Spray pyrolysis → **Synthesis of carbon nanoparticles**

Electron microscope → **Porous particles, morphology and size depend on the pyrolysis temperature and carrier gas pressure**

Raman spectroscopy → **Weakly organized**

Tribological characteristics → **$0.15 < \mu < 0.2$**

Perspective and future work → **Synthesize particles at temperatures well above 1000°C.**
Annealing in order to improve the graphitization degree

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Thank you very much for your attention.

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