Diagnostics in situ d'un procédé plasma de dépôt de couches minces nanocomposites par injection d'une solution colloïdale dans un plasma basse pression

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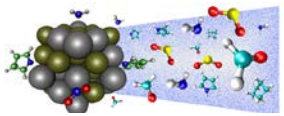

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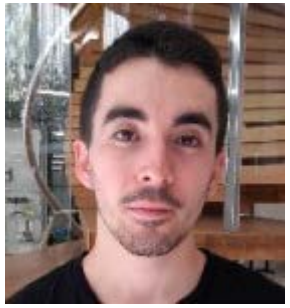

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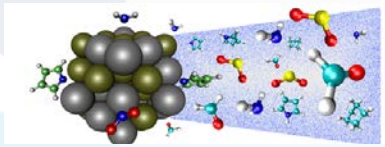

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Outline

- Introduction/context : nanocomposite films deposition by injecting a colloidal solution in a PECVD plasma (misty plasma, aerosol assisted plasma deposition)

- Study of TiO_2-SiO_2 nanocomposite films deposition
  - Time resolved OES and *in situ spectroscopic* ellipsometry
  - Evaporation of solution droplets in the plasma
  - n_{meta}, T_e, $n_e(t)$ in Ar/ solvent plasmas

- Optimisation of ZnO-SiO_2 nanocomposite films deposition by TROES

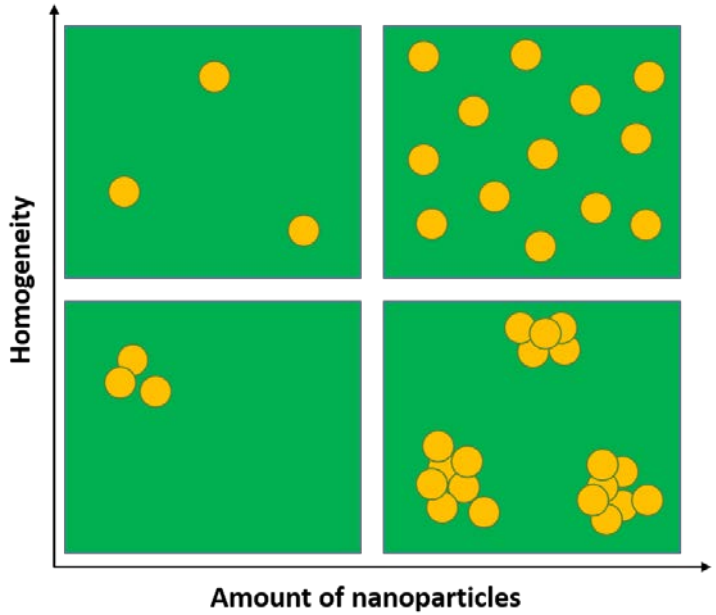
- Conclusion / Perspectives

Main issues for nanocomposite thin films synthesis

Nanocomposite (NC) films => multifunctional materials by combining properties of interest from two separate materials

General Issues for their synthesis:

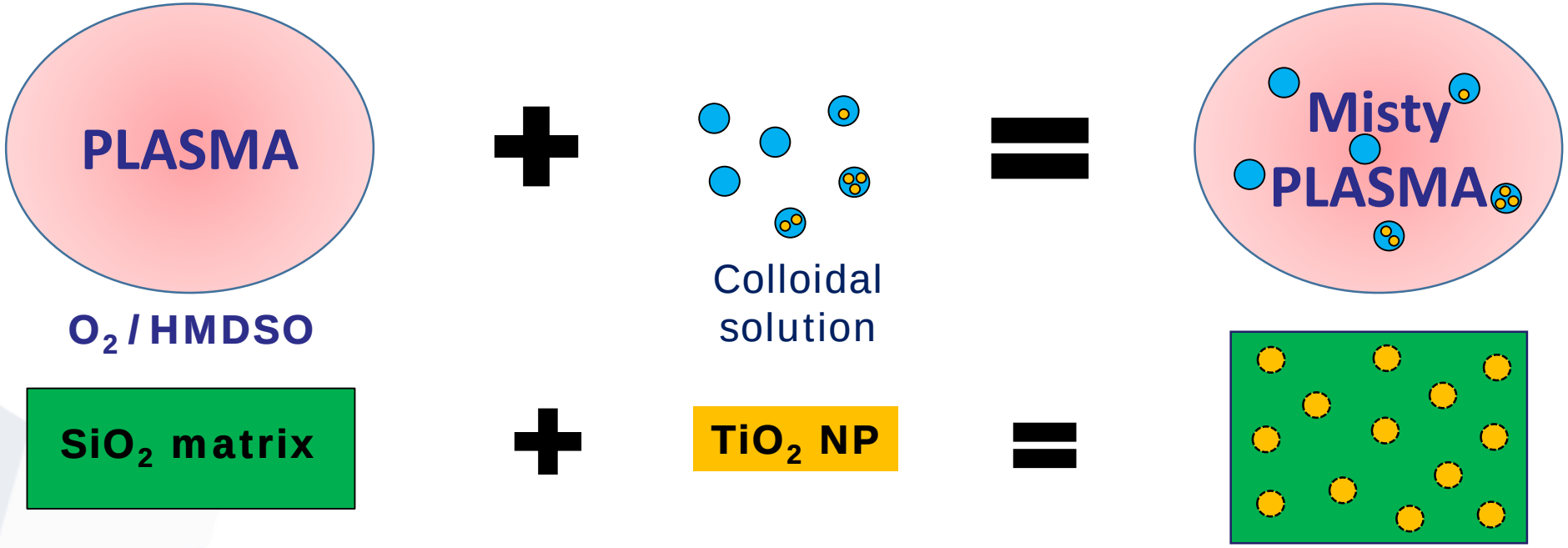
- to control of the density of NPs in the matrix
- to avoid the agregation of the NPs
- to obtain an homogeneous repartition of the NPs in the matrix



Additional challenge in plasma routes:

- **Confinement of NPs (negatively charged) inside the plasma (detrimental to the incorporation of NPs inside the matrix)**

Plasma route chosen for TiO_2-SiO_2 NC films deposition

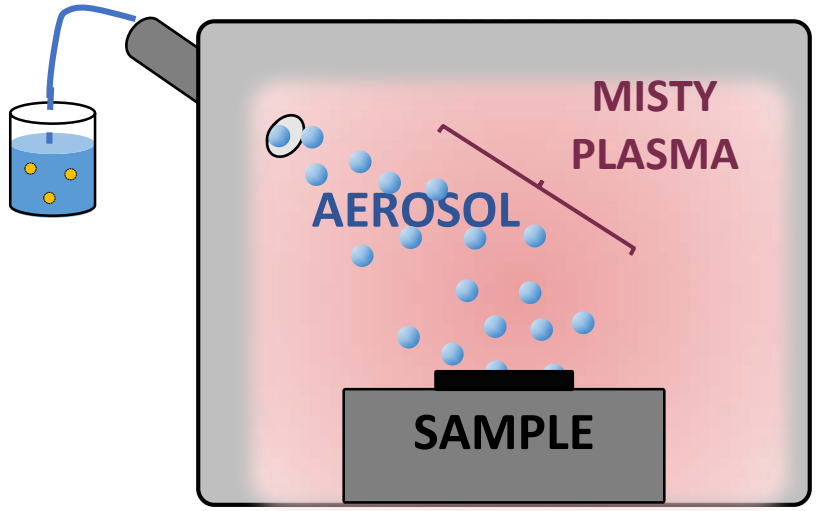


Ideal scenario :

Progressive evaporation of the solvent in the plasma so that the NPs are transported to the substrate inside the liquid droplets with almost no liquid as they reach the surface

Misty plasma for NC thin film deposition

COLLOIDAL SOLUTION (NP dispersed in a solvent)



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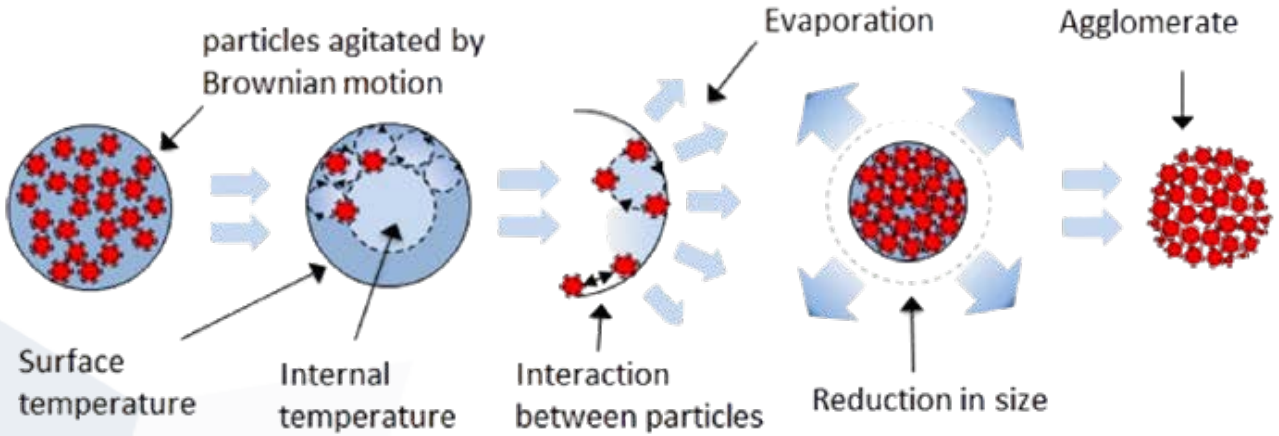
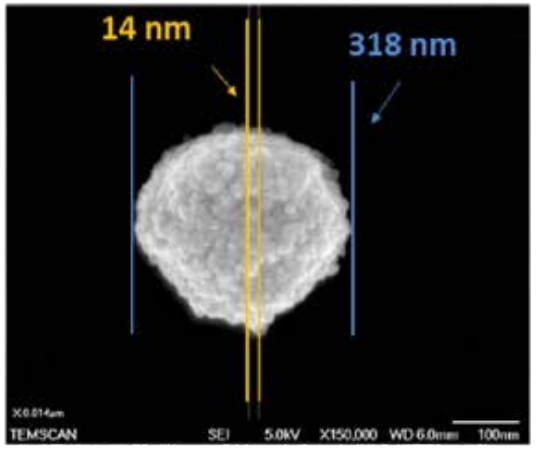
Progressive evaporation of the solvent in the plasma so that the NPs are transported to the substrate inside the liquid droplets with almost no liquid as they reach the surface

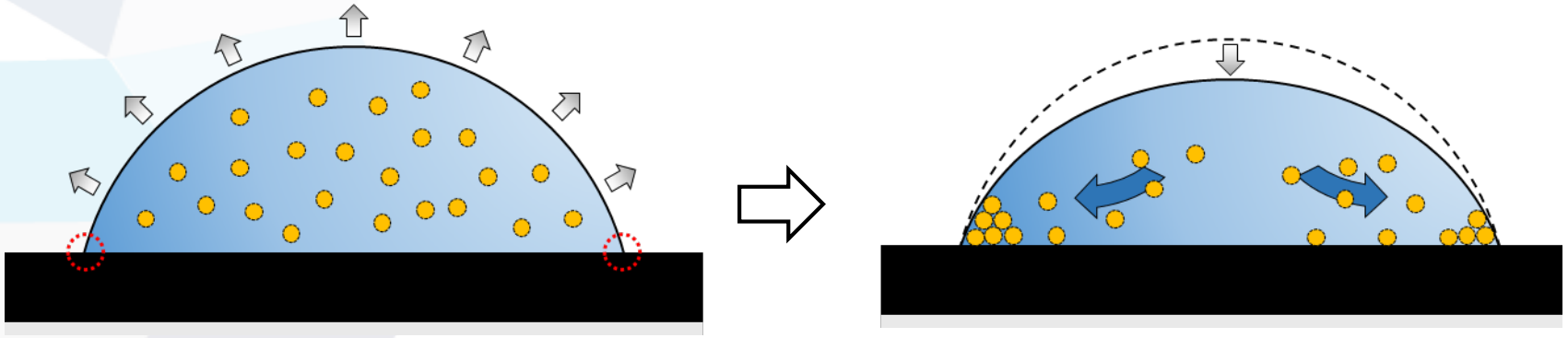
Misty plasmas

Profili *et al.*, J. Phys. D 50, 075201 (**2017**)

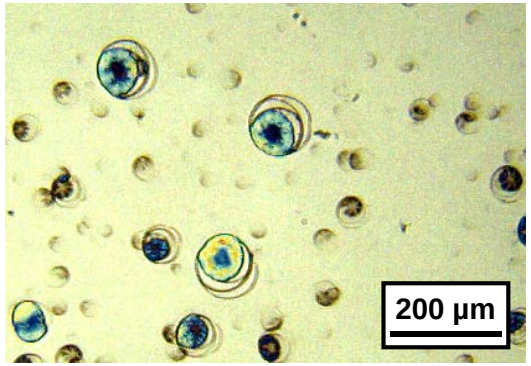




$\tau_{vap} < \tau_{travel}$



« stick and slip »



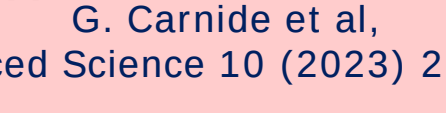
$\tau_{vap} > \tau_{travel}$

Context / NC films by plasma aerosol assisted deposition

low pressure plasma

M. Mitronika et al

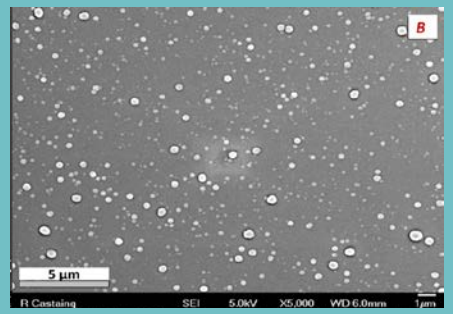
TiO_2 NPs in a SiO_2 matrix
for self cleaning superhydrophilic films



ZnO NP in a carbon based matrix
for super-hydrophobic coatings

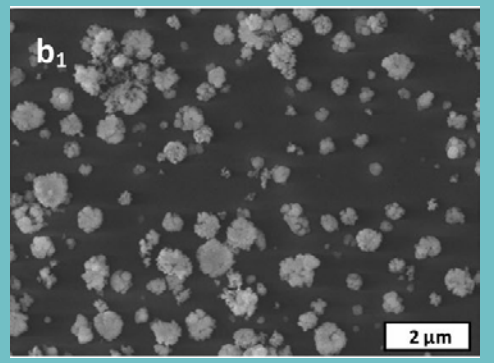
G. Carnide et al,
Advanced Science 10 (2023) 2204929

atmospheric pressure plasma

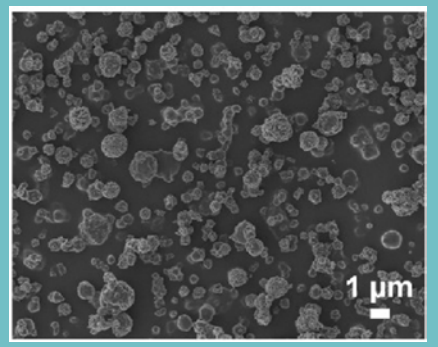


J. Profili et al.,
J. Appl. Phys. 120 (2016) 053302

TiO_2 NP in a carbon based matrix



P. Brunet al,
Plasma Process Polym. (2017)14:e1700049



F. Fanelli et al,
Langmuir 30 (2014) 857–865

University of Bari Aldo Moro

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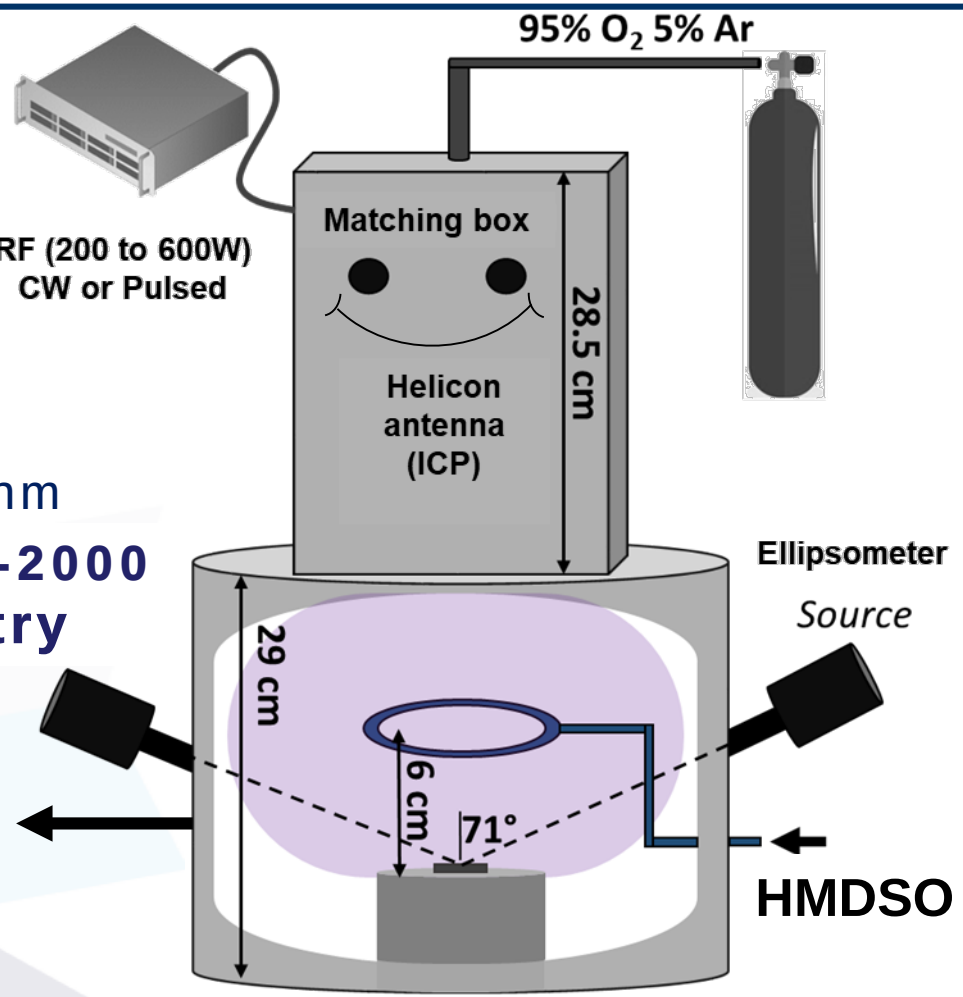
Low Pressure RF ICP PECVD reactor (IMN)



245-1000 nm

Woollam M-2000
Ellipsometry

p : 3 mTorr
O_2/Ar : 24 sccm

ICP Plasma Source

Diffusion plasma Deposition chamber

Substrates : Si, PET/ITO, PS

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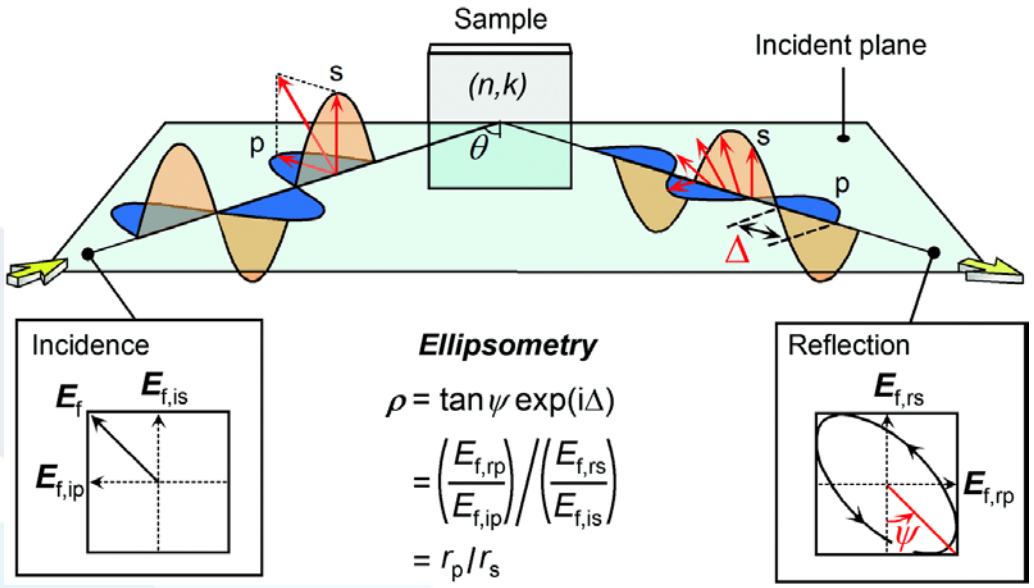
Time resolved spectroscopic ellipsometry

Analysis of the change of polarization of light



$$Is = sin(2\Psi)sin(\Delta),$$
$$Ic = sin(2\Psi)cos(\Delta)$$

Kinetic ellipsometry: time variations of Ψ and Δ at a given wavelength
=> ellipsometric trajectories

Spectroscopic ellipsometry: optical model of the film used to analyse the ellipsometric data
best fit => n(λ), k(λ) and film thickness

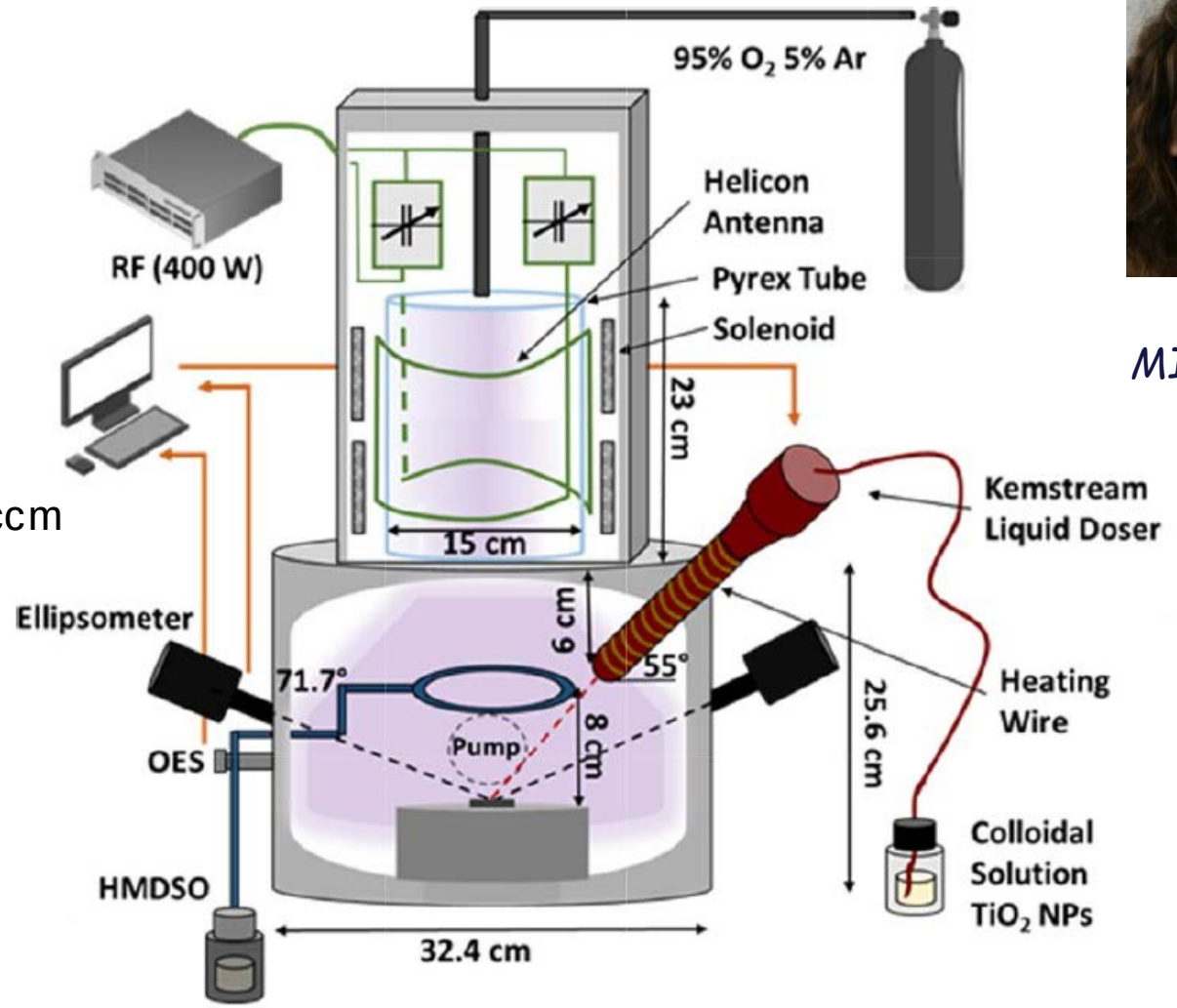
Time resolved spectroscopic ellipsometry: spectroscopic data recorded every 2 s
=> **ability to monitor film thickness and index *in situ* along the film growth**

Hybrid PECVD/DLI deposition process

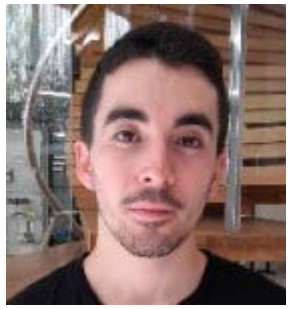
Plasma conditions:

- Pressure: 3 mTorr
- O_2 flowrate: 24 sccm
- Power: 400 W

- HMDSO flow: 0.11 sccm

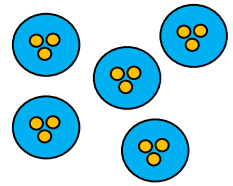


Maria MITRONIKA



Simon CHOUTEAU

DLI: Pulsed liquid injection



Droplet diameter ~ 40 µm

NP diameter ~ 4 nm

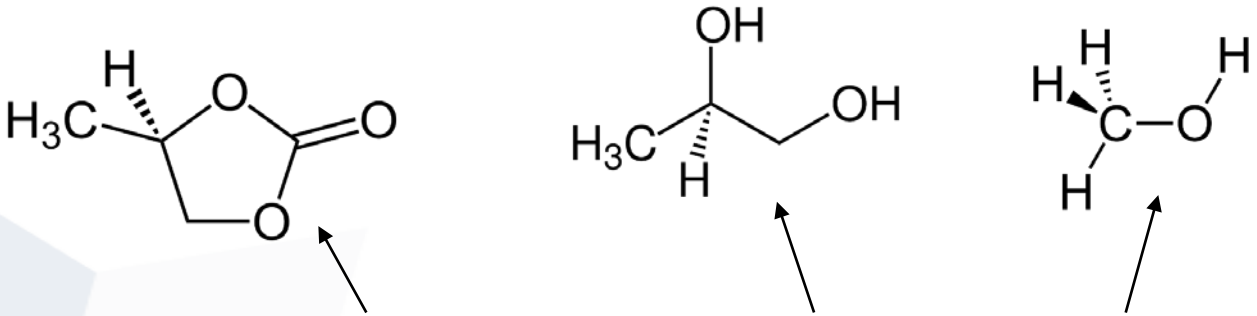
~ 1µL/pulse

Mitronika et al. *J. Phys. D: Appl. Phys.*, 2021, 54, 085206

Colloidal solution

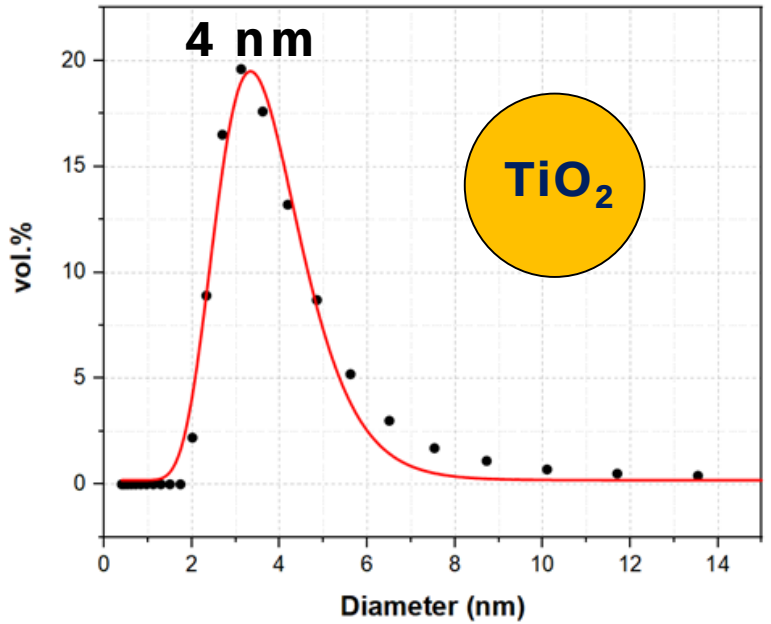
solvent composition :

PC/PG/methanol : 20 / 55 / 25 (volume fractions)



	Propylene carbonate PC	Propylene glycol PG	Methanol
Boiling point (°C)	242	188	65
Vapor pressure (Pa)	4	11	12300
Volatility	-	-	+ + +

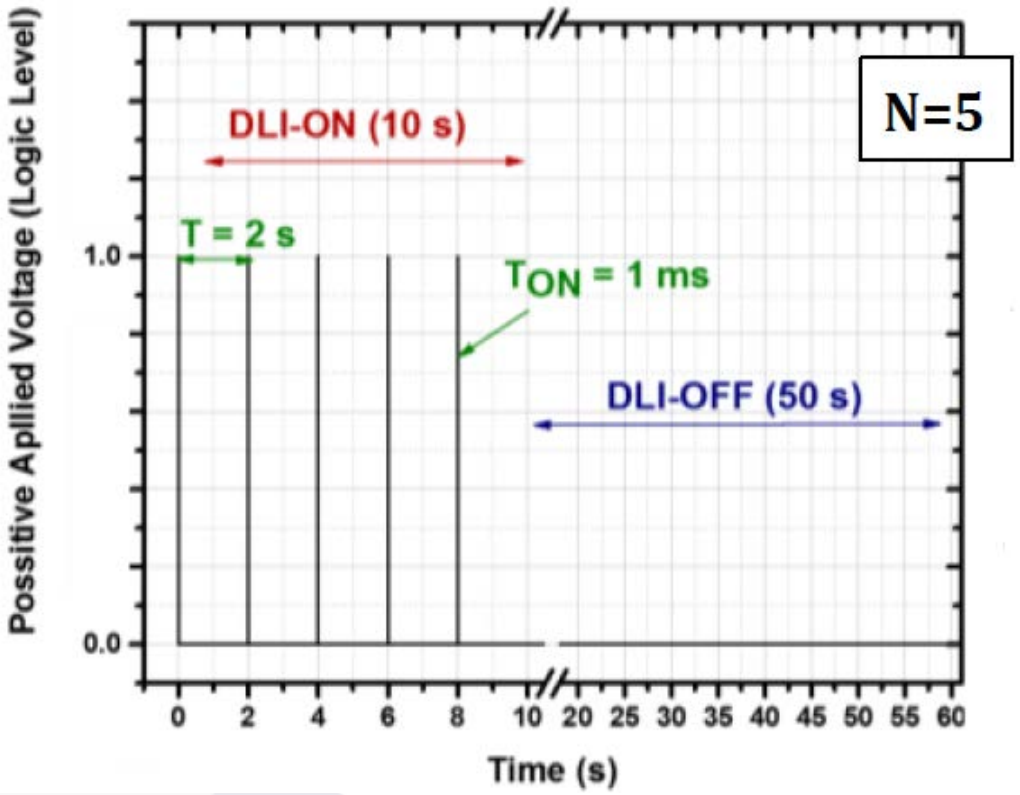
anatase TiO_2 NP size distribution

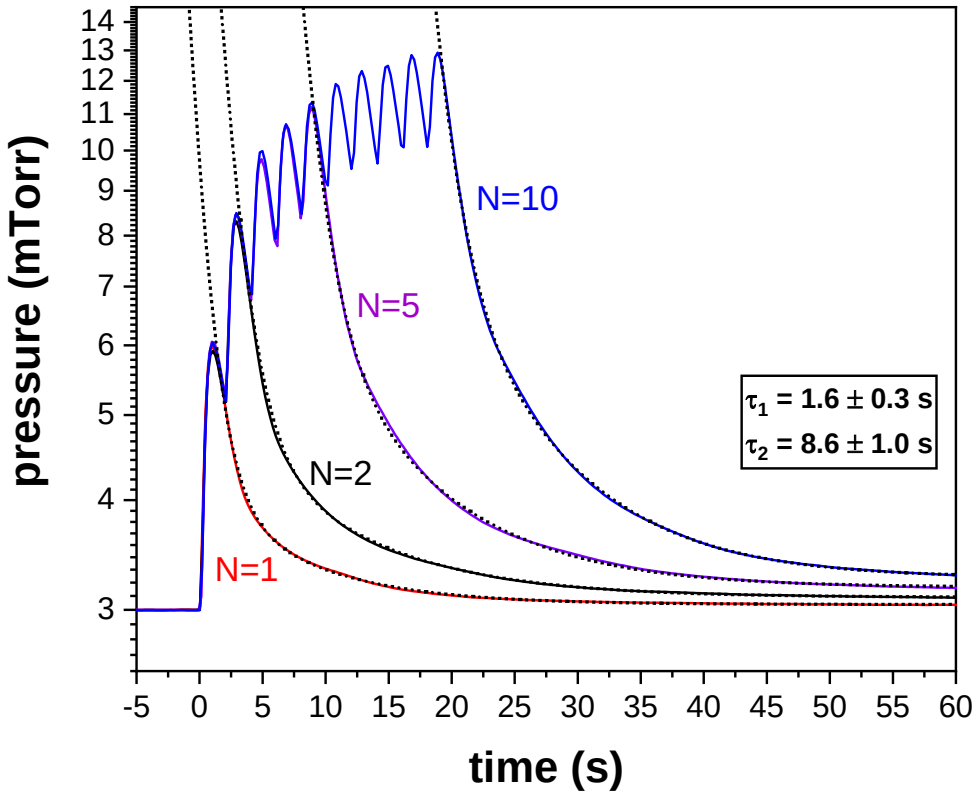


A. Karpinski et al., Sol. Energy Mater. Sol. Cells. 116 (2013) 27–33

Pulsed Liquid injection

Iterative mode of injection : Sequence of N pulses (1ms each, 2s apart) per minute



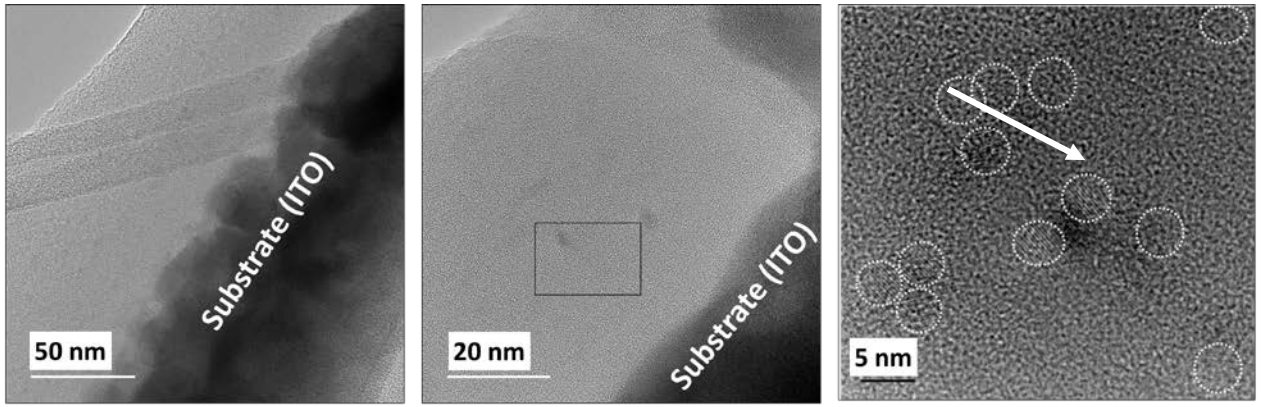


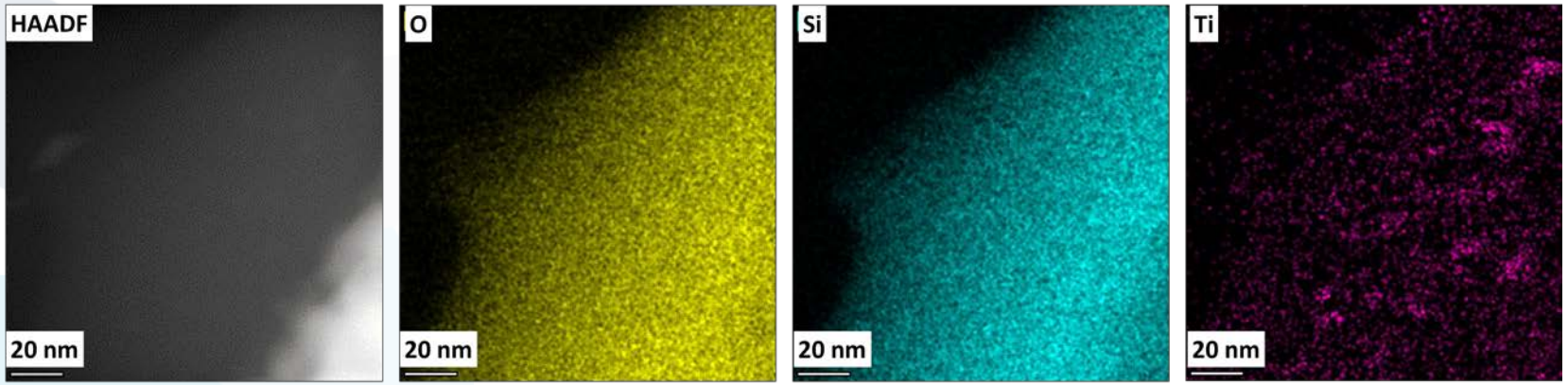
Characteristic decay times of the pressure consistent with the solvent evaporation time

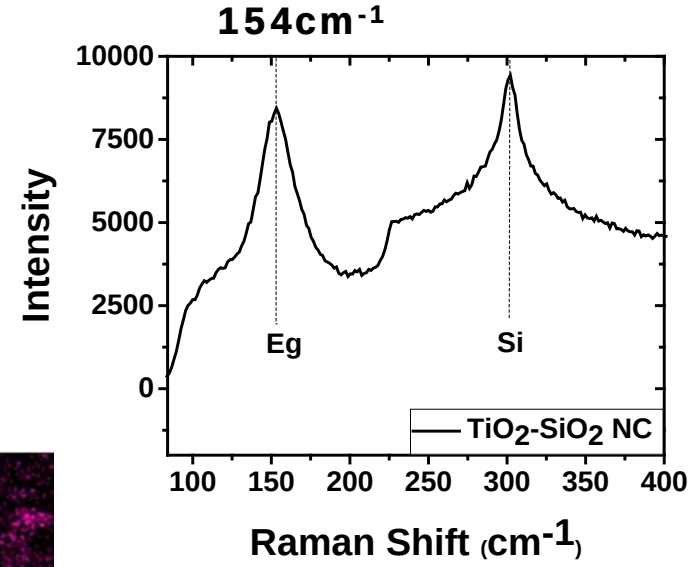
TiO_2-SiO_2 NCs thin films – Structural Analysis

N = 10

TEM







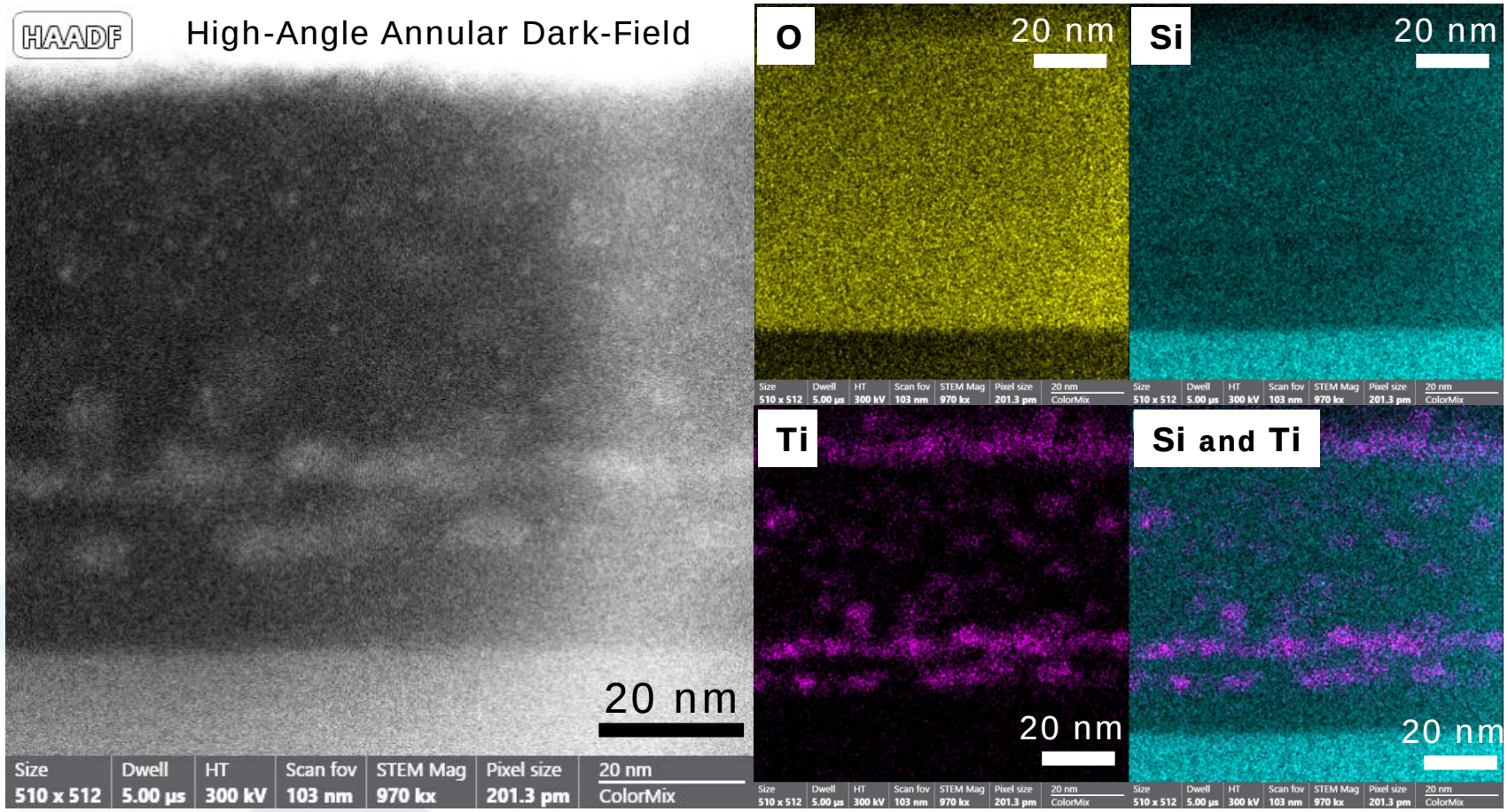
anatase Eg band peak of TiO_2 bulk at 144 cm^{-1}

shift to 154 cm^{-1} => nanoscale TiO_2

NPs distributed inside the SiO_2 matrix,
NPs not agglomerated (1-2 NPs in proximity).
Raman: Nanometric anatase phase maintained.

Structure of the TiO_2-SiO_2 NC film

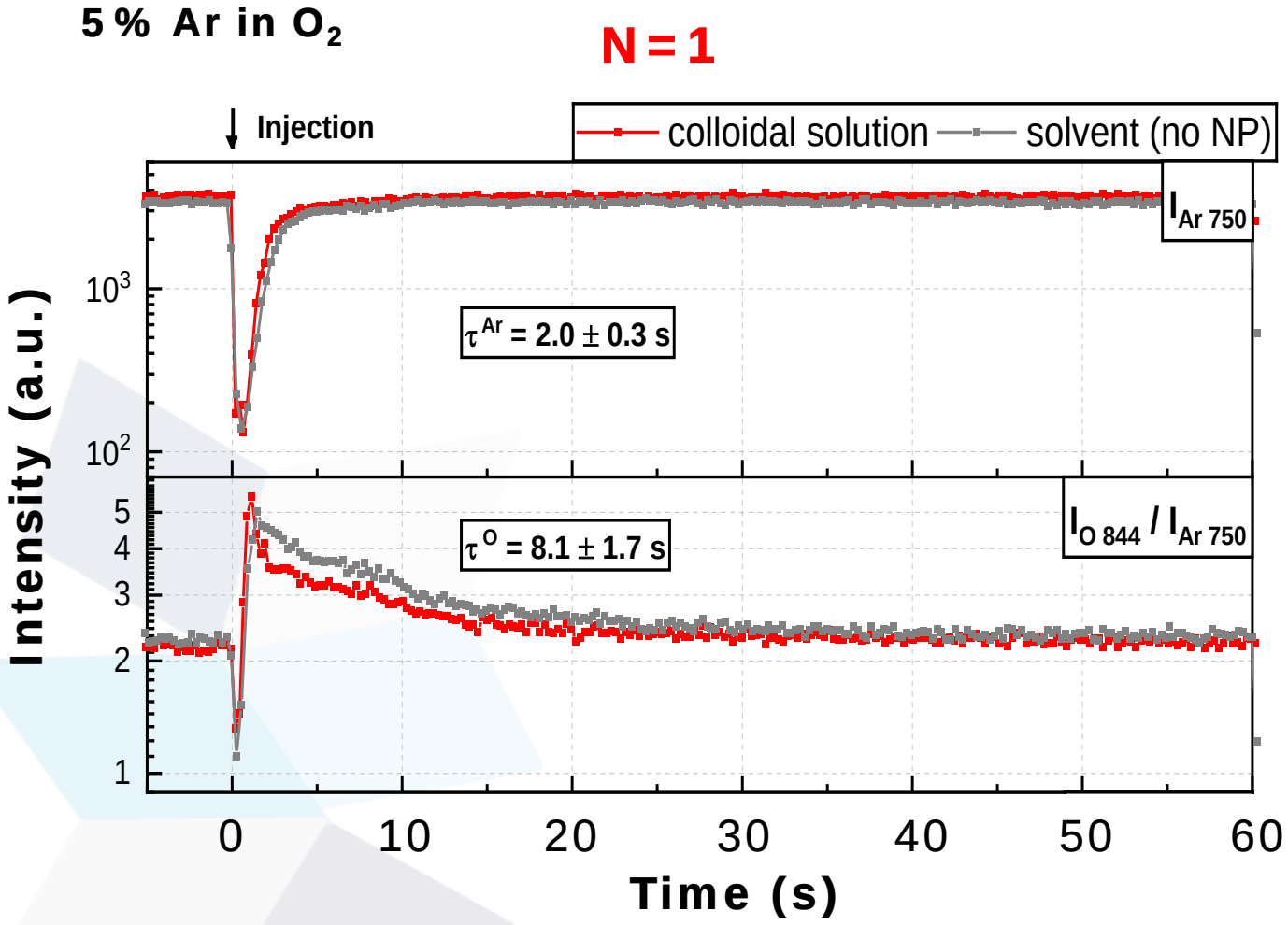
STEM cross section, HAADF and EDX



Conditions: 75% MeOH solution, 60 min deposition at N=10

- Si and O homogeneously distributed through the film
- Ti-rich areas from *coffee rings* clearly visible
- Their height roughly match a single NP (5 nm)

Time resolved optical emission spectroscopy (N=1)



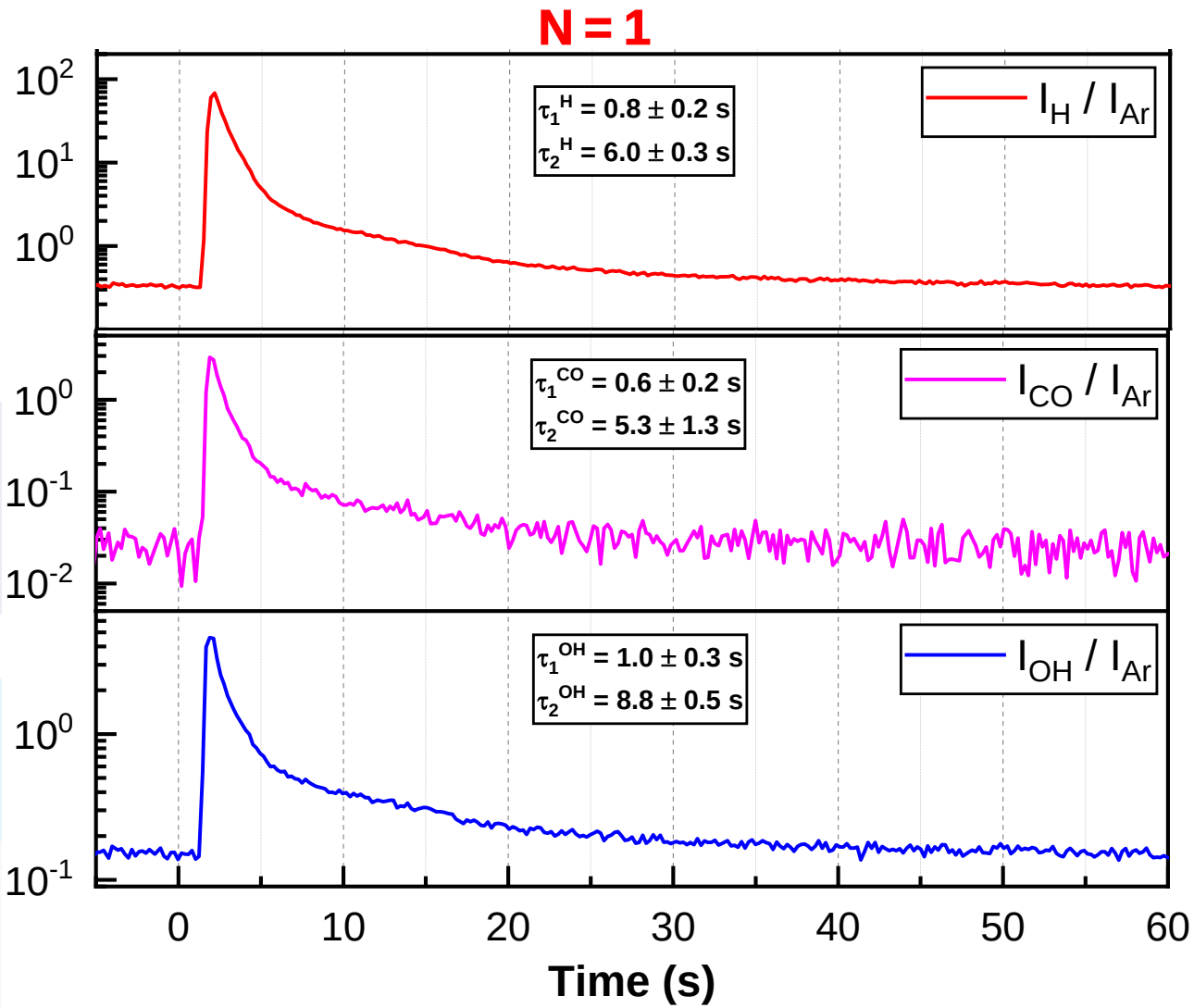
- same variations of I_{Ar} and I_O/I_{Ar} with and without NPs in the solvent

NPs remain inside the droplets and do not interact with the plasma

- strong decrease of I_{Ar} upon solvent injection related to the pressure increase and the associated decrease in n_e and T_e

- I_O/I_{Ar} representative of O atom density :
  - initial decrease => oxydation reactions
  - fast increase attributed to a change in O recombination at the wall due to surface reactions (such as H_2O adsorption)
  - then slow decay corresponding to the progressive elimination of by-products and « cleaning » of the walls

Time resolved optical emission spectroscopy (N=1)

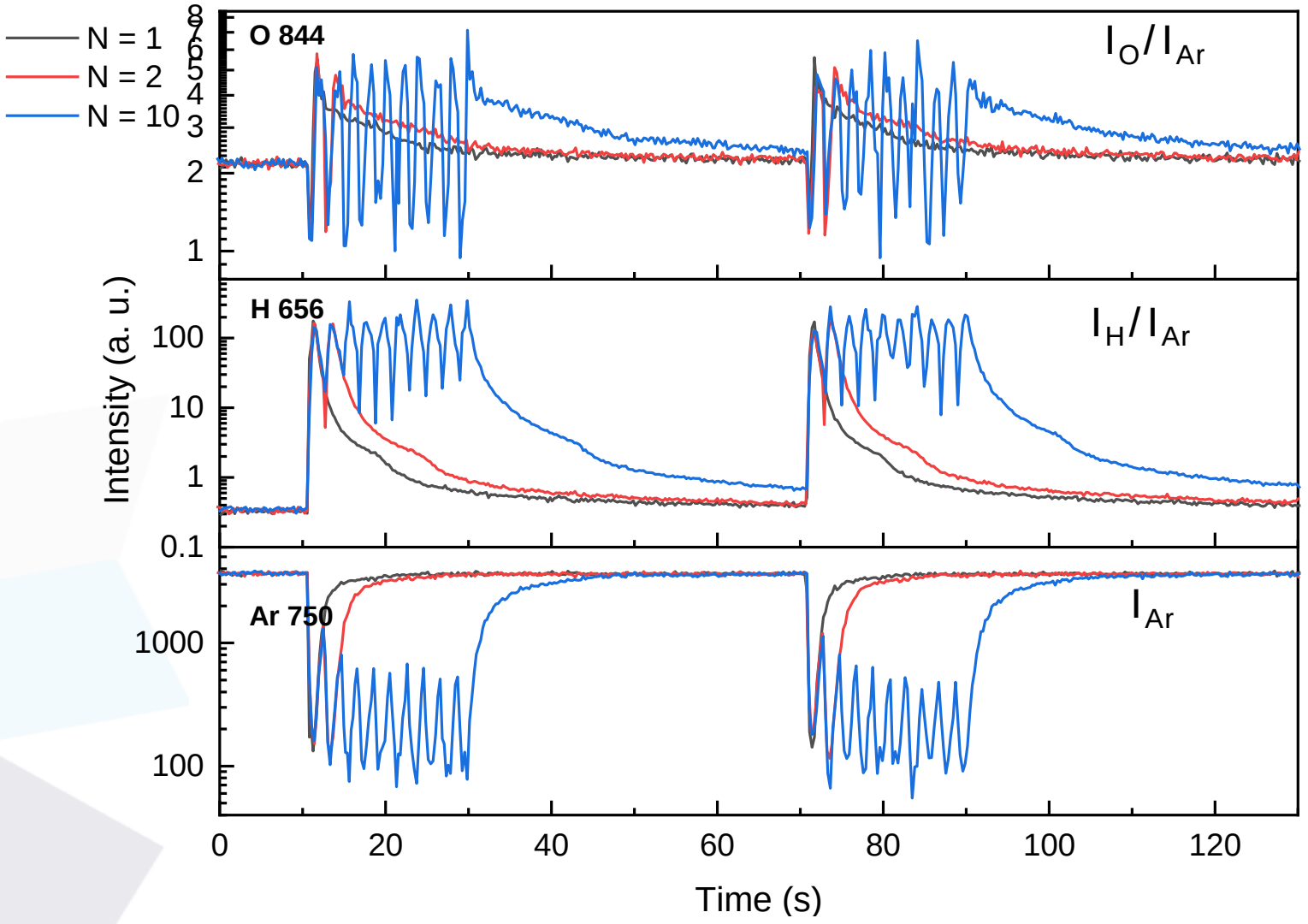


- Very similar time variations for I_H/I_{Ar}, I_{CO}/I_{Ar} and I_{OH}/I_{Ar} : CO, CO_2 H_2 and H_2O formed upon oxidation of the solvent in the oxygen plasma
- Strong increase within the first second after the solvent injection followed by a decrease with two time scales (1 and 5-8 s), similar to the pressure decrease

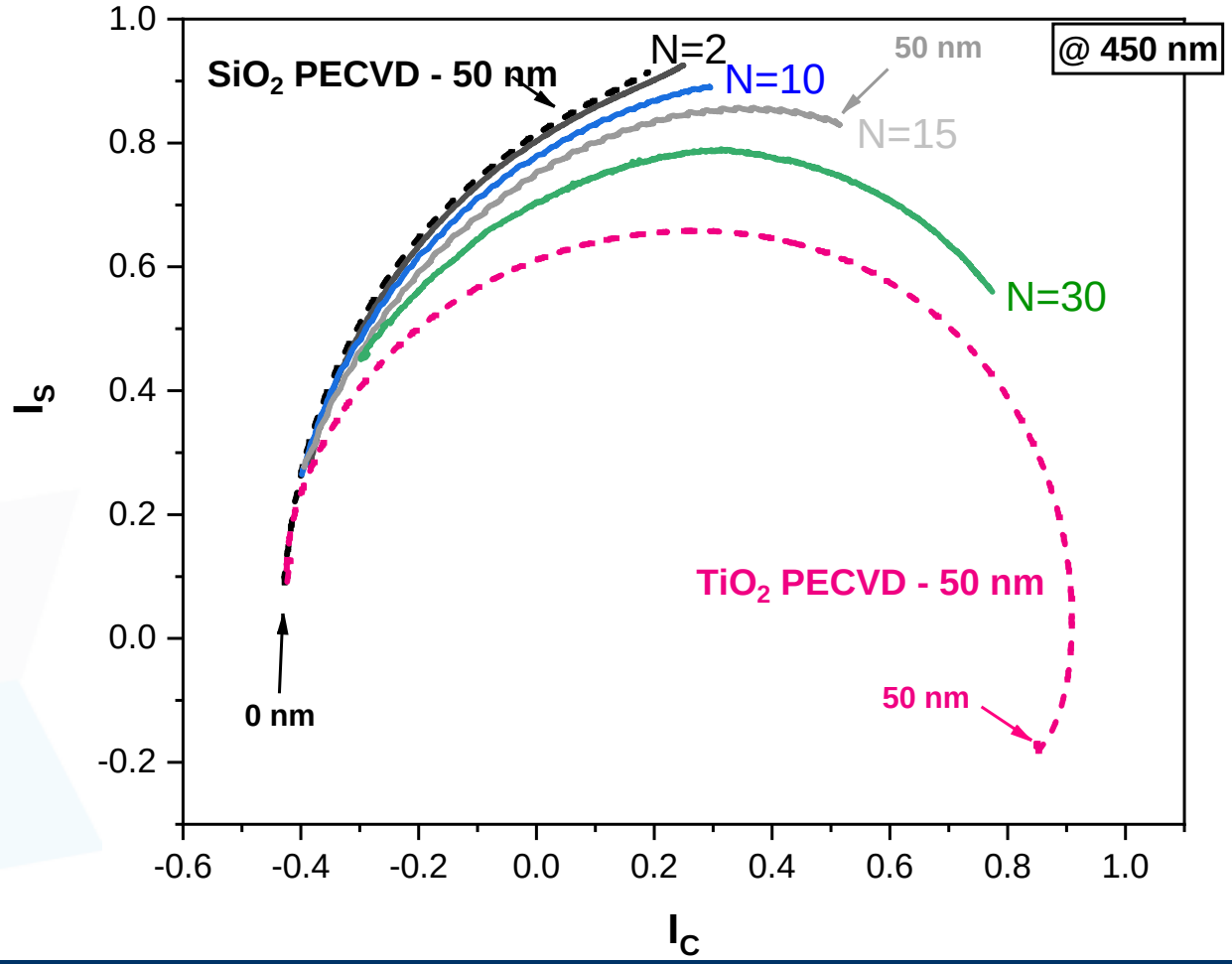
Chouteau et al. *J. Phys. D: Appl. Phys.*, 2022, 55, 505303

Time resolved optical emission spectroscopy (N=10)

N=10

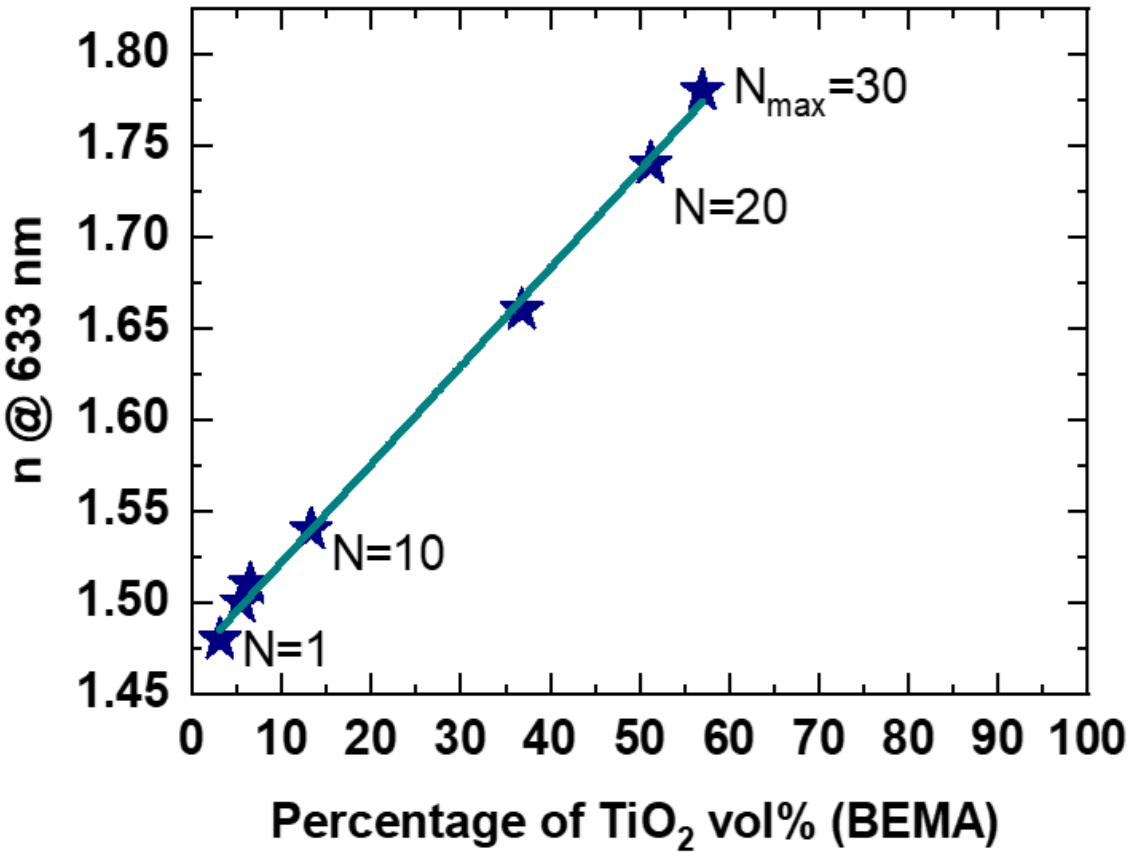


TiO_2-SiO_2 NCs thin films - *in situ* ellipsometric trajectories



TiO_2–SiO_2 NCs trajectory plots between the pure SiO_2 matrix and the PECVD TiO_2 film ones.

Volumic fraction of TiO_2 NPs in the TiO_2-SiO_2 NC thin films



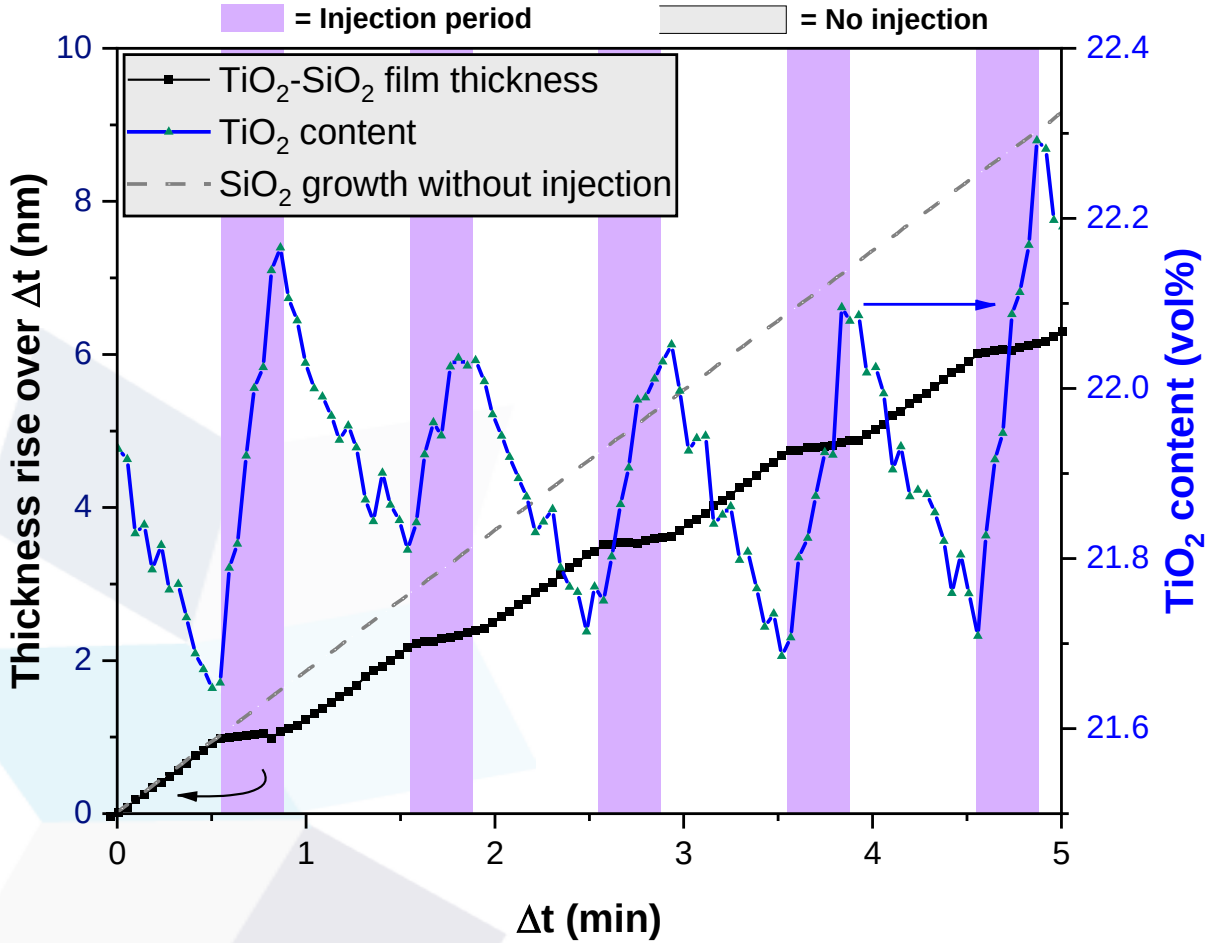
Spectroscopic Ellipsometry :

NC film modeled using Bruggeman effective medium approximation (BEMA) model with TiO_2 NPs (Tauc-Lorentz dispersion law) in a SiO_2 matrix

Large range of TiO_2 NPs volume content in the SiO_2 matrix obtained by varying the solution injection conditions : N = 1 to 30 → 1 to 58 % fraction of TiO_2 NPs

Deposition kinetics of the NC thin film (ellipsometry)

N = 10



during the injection periods:

- decrease of the SiO_2 matrix deposition rate (due to n_e and T_e decrease)
- increase in the TiO_2 NP volume fraction

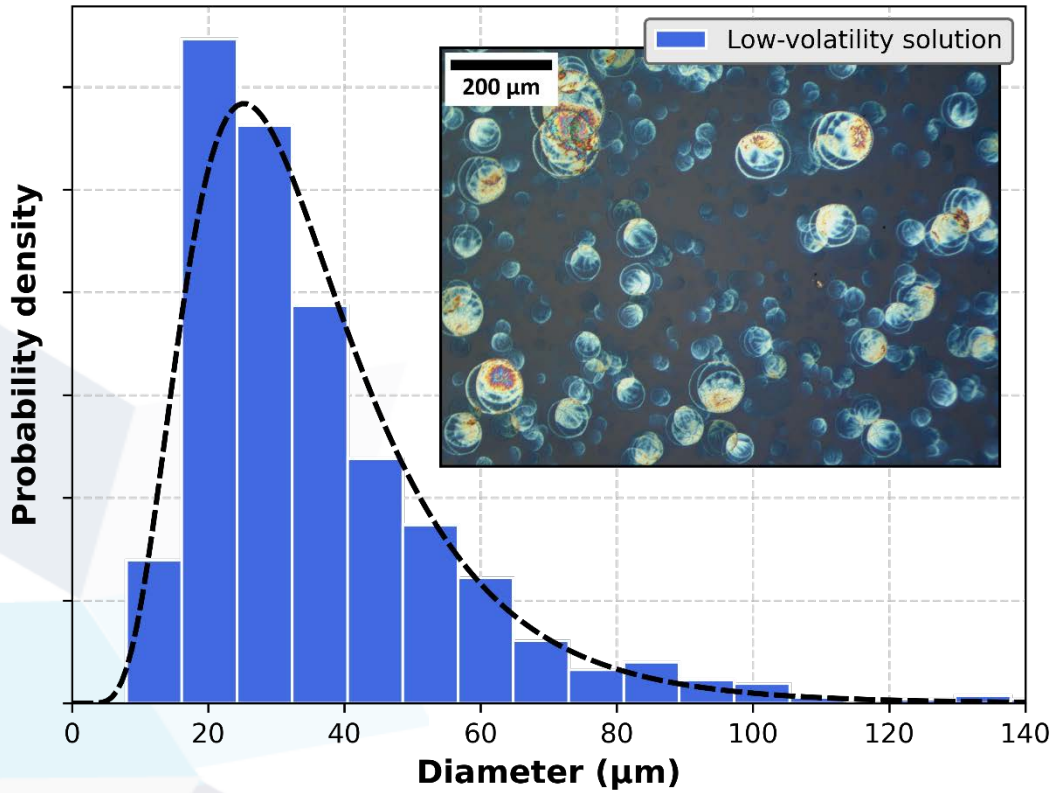
DLI-ON: mainly deposition of TiO_2 NPs and inhibition of SiO_2

DLI-OFF: deposition of the silica matrix

Chouteau et al. *J. Phys. D: Appl. Phys.*, 2022, 55, 505303

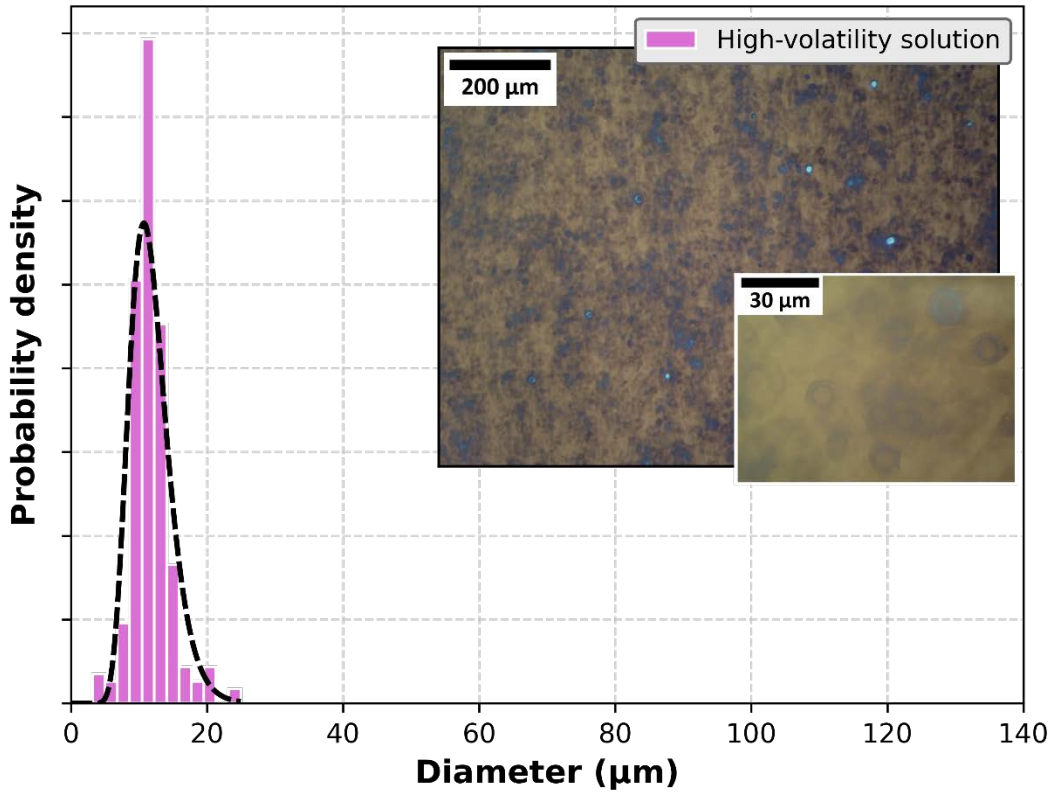
Optimisation of TiO_2/SiO_2 NC deposition

PC/PG/MeOH : 20:55:25 (vol %)



Mean diameter: **36 µm**
Standard deviation: **10 µm**

PC/PG/MeOH : 11:9:80 (vol %)



Mean diameter: **12 µm**
Standard deviation: **3 µm**

Evaporation of a droplet in the plasma

- Particle balance:

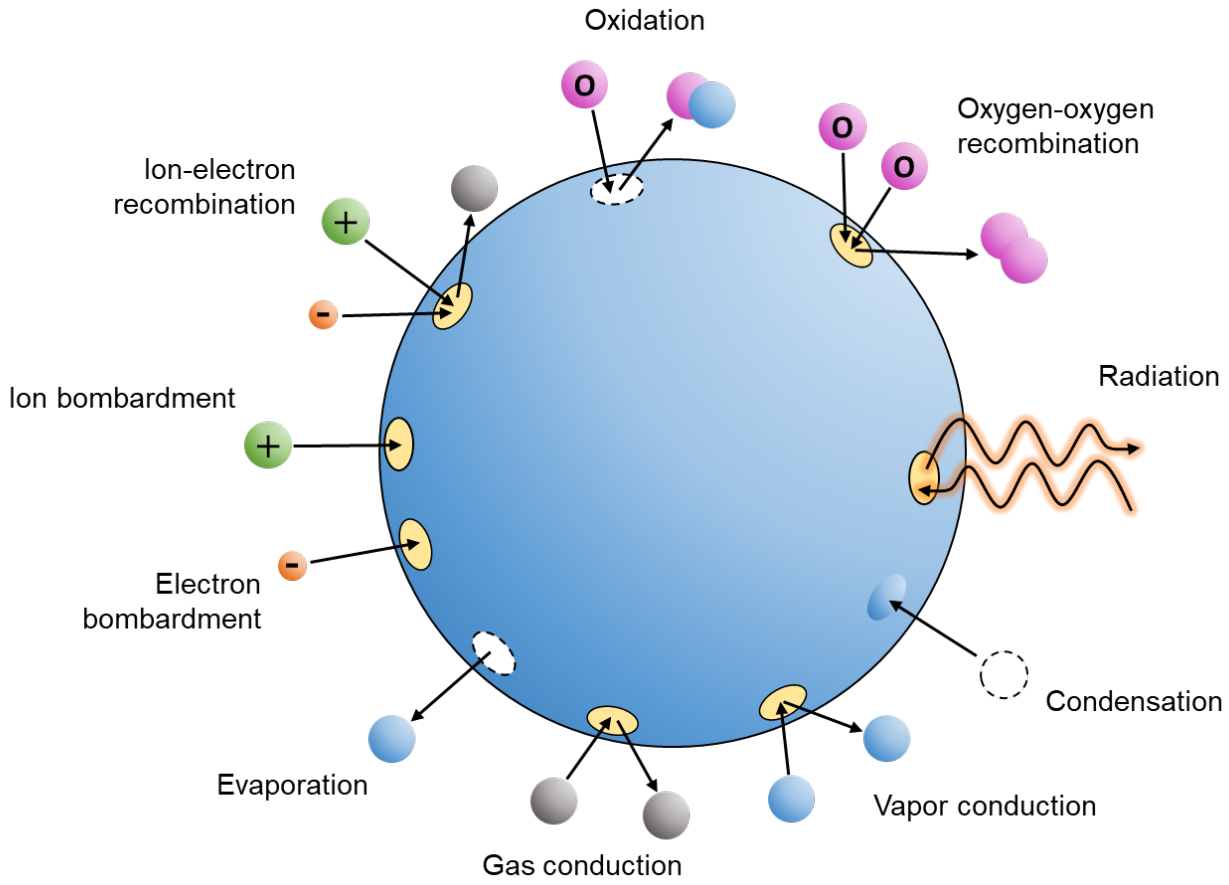
$$\frac{dr_d}{dt} = -\sum_{\substack{i=MeOH,\\ PC,PG}} x_i \frac{M_i}{\rho_i} \frac{\alpha_{e,i} P_{sat,i}}{\sqrt{2\pi M_i R T_d}}$$

where:

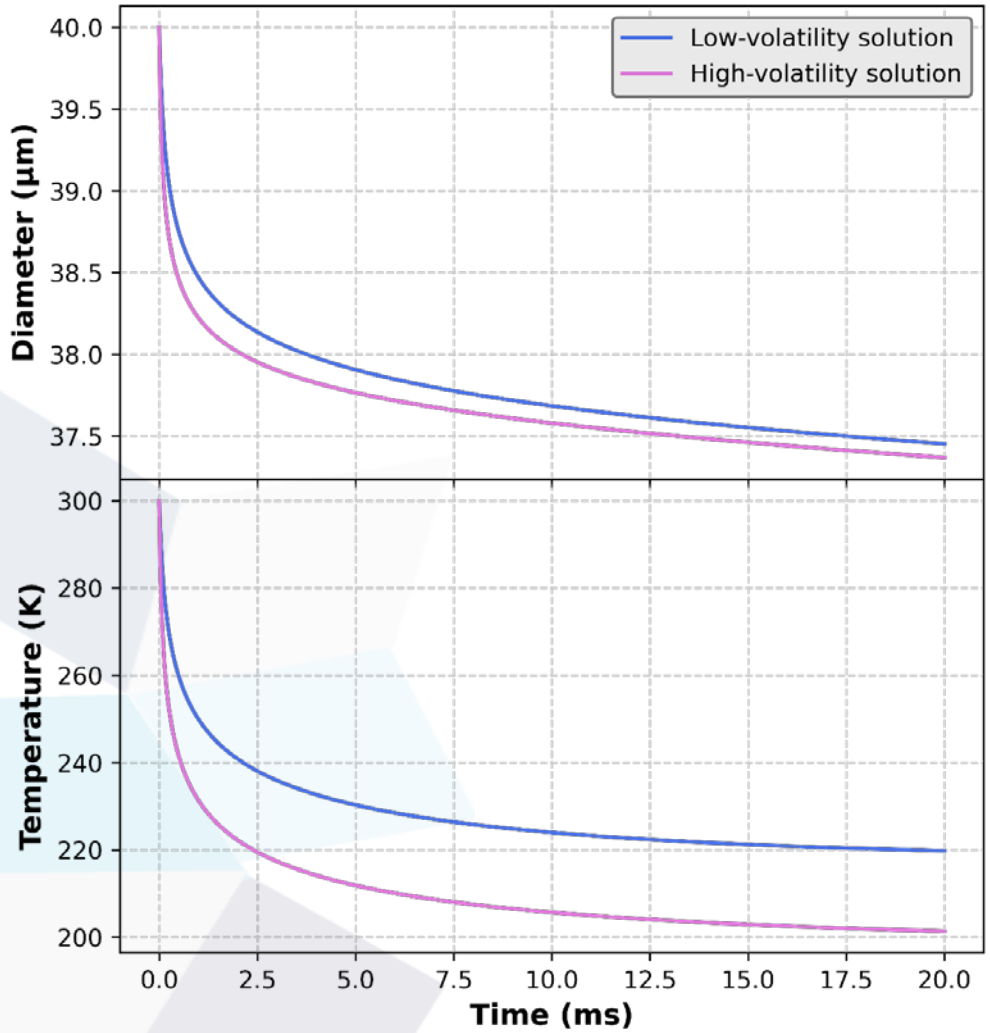
$$P_{sat} = P_{ref} \exp\left(\frac{H_{vap}}{R}\left(\frac{1}{T_{ref}} - \frac{1}{T_d}\right)\right)$$

- Energy balance:

$$\frac{dT_d}{dt} = \frac{A_d}{m_d C_p}(J_{in} - J_{out})$$



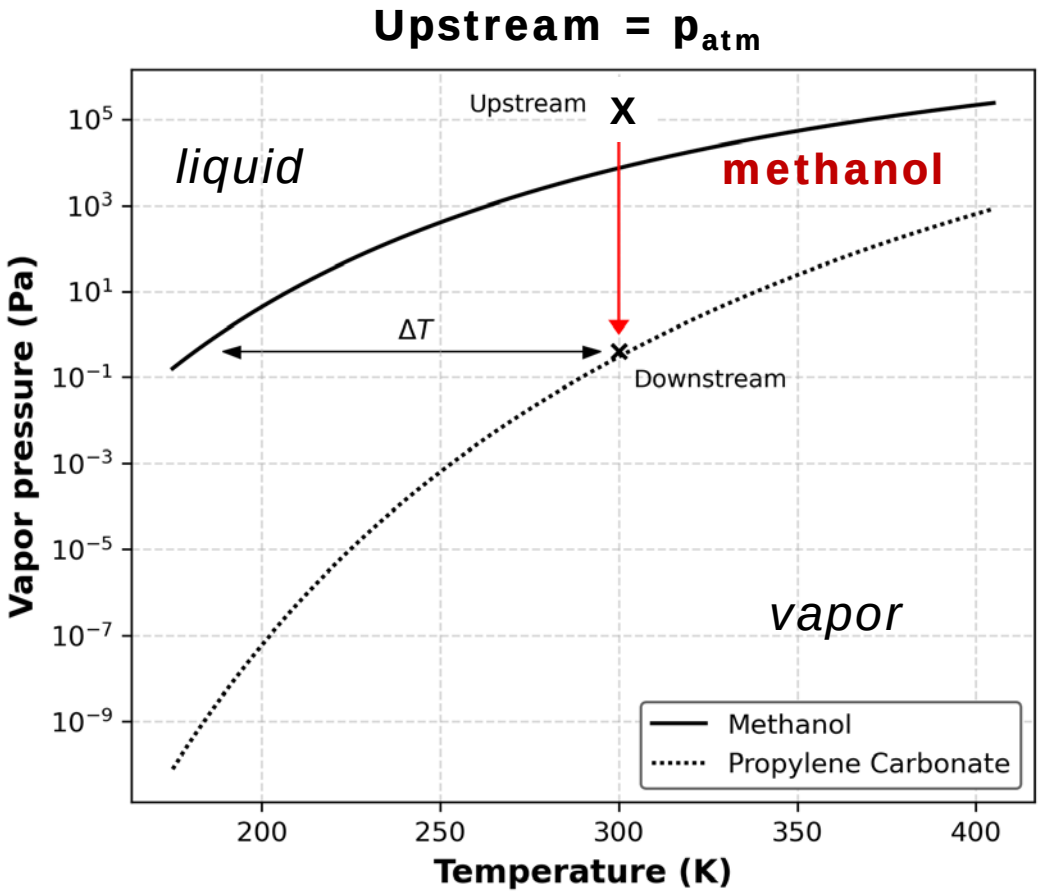
Evaporation of a droplet in the plasma



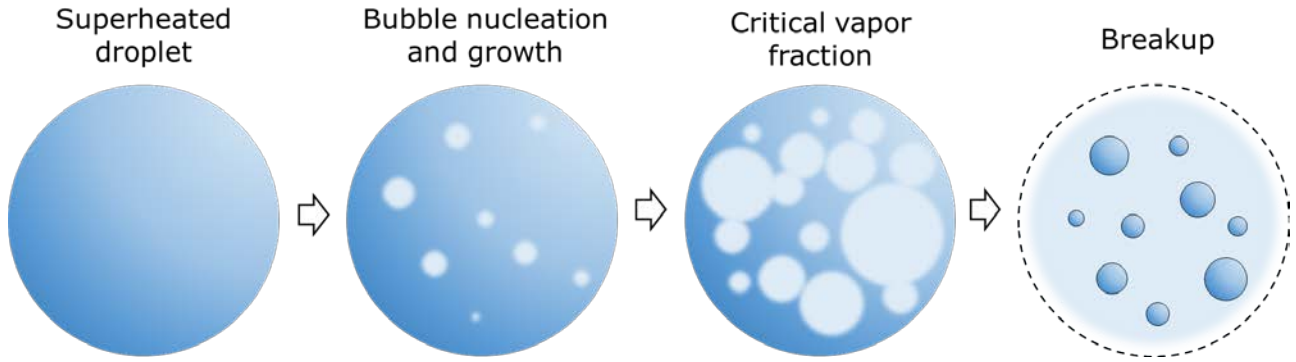
- Simulated time 20 ms to match with the droplets' travel time from injector to sample.
- Evaporation is self-limited: rapid cooling due to solvent evaporation prevents further evaporation.
- No significant difference between low- and high-volatility solution

Evaporation alone cannot account for the experimental observations

Flash Boiling



Solvents	Propylene carbonate	Propylene glycol	Methanol
Boiling point	242 °C	188 °C	65 °C
Vapor pressure at 25°C	4 Pa	11 Pa	12300 Pa

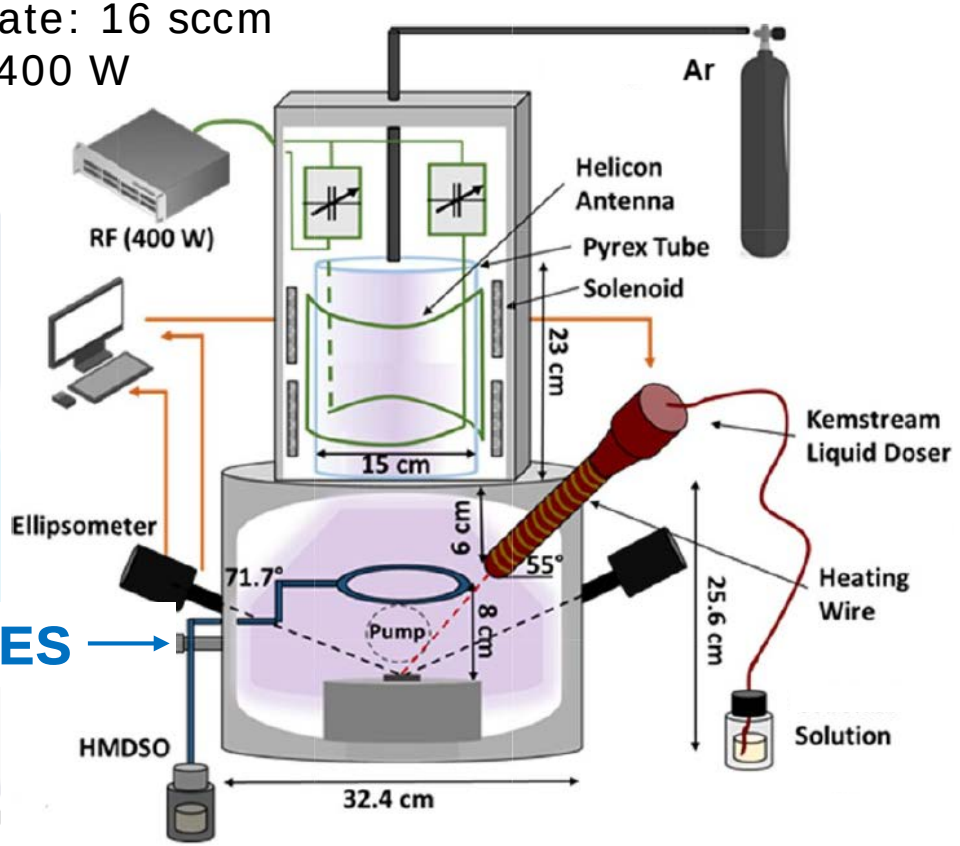


Methanol added to PC/PG ➔ Flash Boiling ➔ Reduction in the droplet size ➔ better dispersion of TiO_2 NP in the SiO_2 matrix

n_{meta}, T_e, $n_e(t)$ in Ar + pulsed solvent injections (N=1)

- study of the plasma time evolution upon pulsed solvent injection
- what is the influence of the solvent nature ?

- Pressure: 3 mTorr
- Ar flowrate: 16 sccm
- Power: 400 W

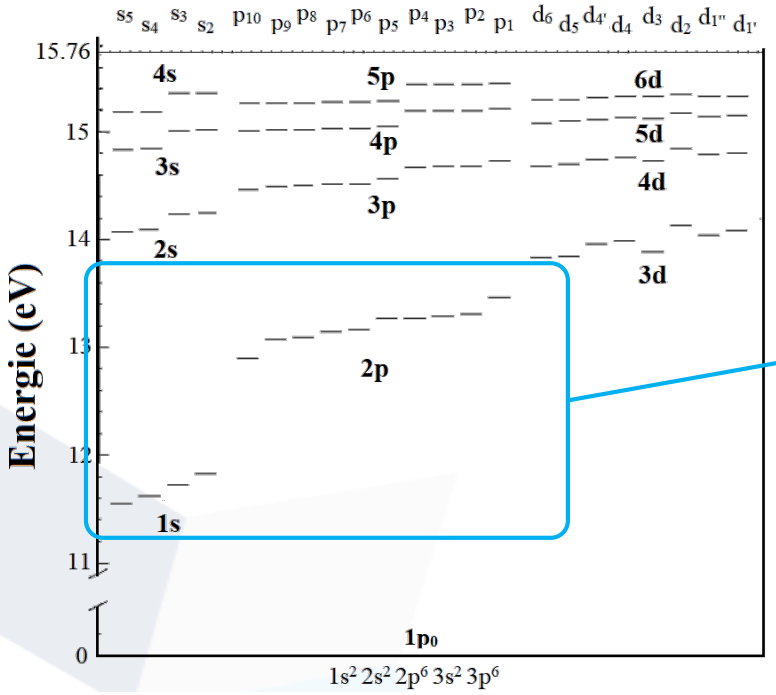


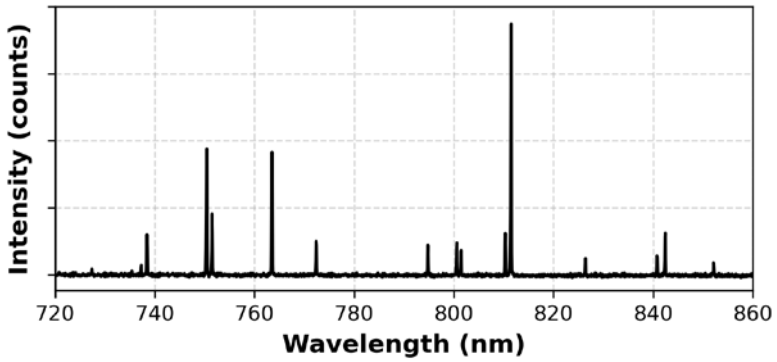
TROES →

Methanol – MeOH – CH_4O
Propylene Carbonate – PC – $C_4H_6O_3$

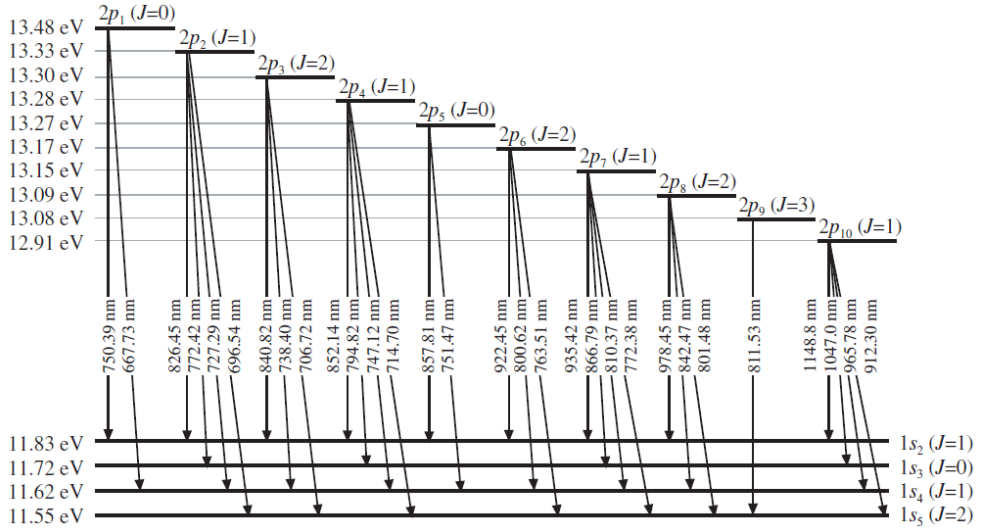
THERMOPHYSICAL PROPERTIES		
Solvent	MeOH	PC
Vapor pressure at 300 K	~19 kPa	~10 Pa
Boiling temperature at 1 atm	338 K	515 K
	⇩	⇩
	Volatile	Non-volatile

Methods – Optical Emission Spectroscopy





Boffard *et al.*, Plasma Sources Sci. Technol. **19** 065001 (2010)



Measured intensity for a $2p_i$ to $1s_j$ transition:

$$I(\lambda_{ij}) = n_{2p_i} A_{ij} \theta_{ij}$$

θ_{ij} → Radiation trapping coefficient (aka "Escape factor")

A_{ij} → Einstein coefficient for the transition

n_{2p_i} → Number density of emitting level $2p_i$

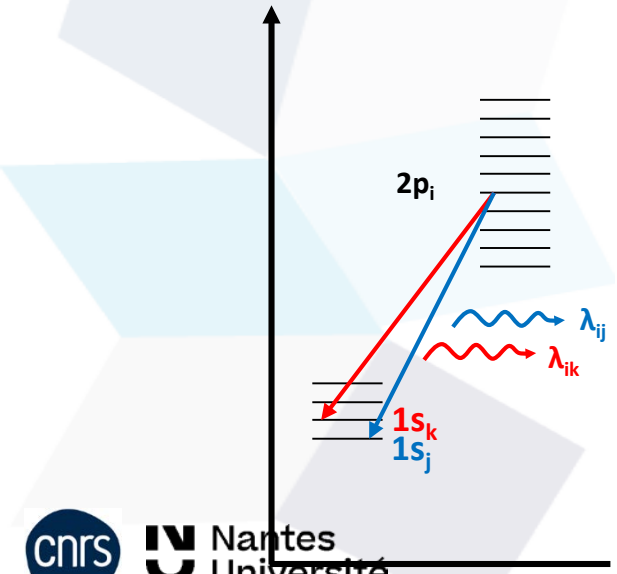
Methods – Determination of Ar metastable density n_{1s}

- **radiation trapping used to evaluate the number density of metastable Ar (n1s) using the ratio of lines emitted by the same 2p level**

- **main assumptions:**
  - no significant contribution of Ar resonant levels ($1s_2$ and $1s_4$) to radiation trapping $\theta_{1s_r} \approx 1$
  - plasma homogeneous along the line of sight $\theta \neq f(x)$

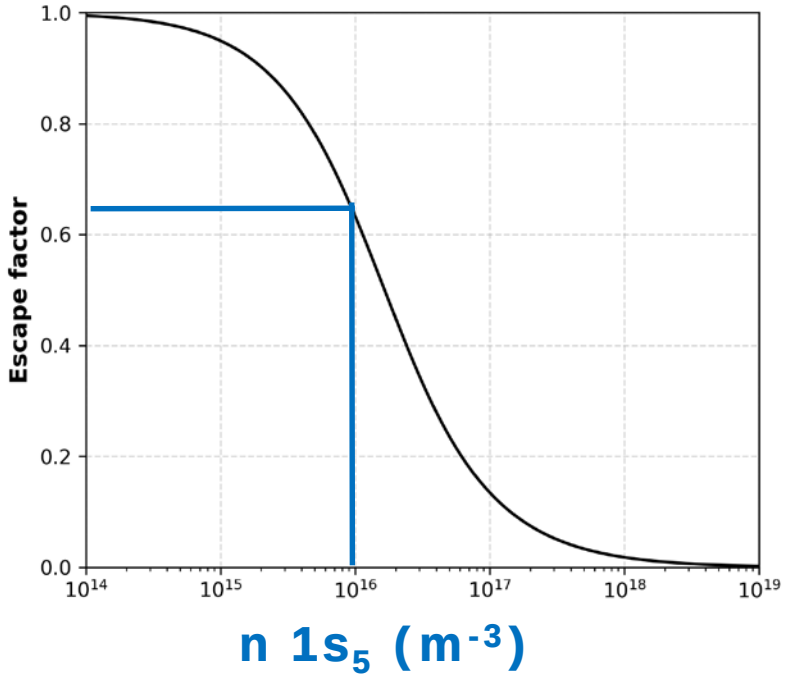
$$\frac{I(\lambda_{ik})}{I(\lambda_{ij})} = \frac{\cancel{n_{2p_i}} A_{ik}\,\theta_{ik}}{\cancel{n_{2p_i}} A_{ij}\,\theta_{ij}} \Rightarrow \frac{\theta_{ij}}{\theta_{ik}} = \frac{A_{ik}\,I(\lambda_{ij})}{A_{ij}\,I(\lambda_{ik})} \Rightarrow \boxed{\theta_{ij,exp} \approx \frac{A_{ik}\,I(\lambda_{ij})}{A_{ij}\,I(\lambda_{ik})}}$$

(assuming level $1s_k$ is resonant)



To be compared with:

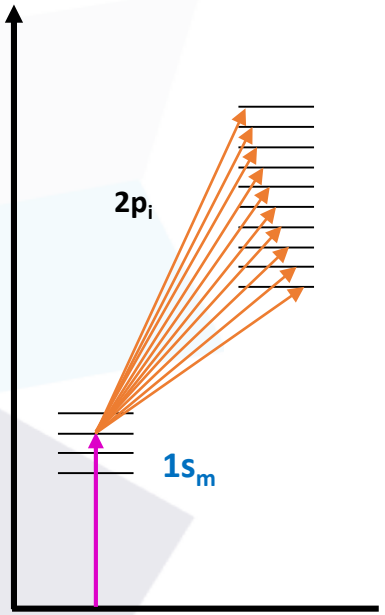
$$\theta_{ij,theo} = \frac{I_T}{I_0} = \frac{\int_{-\infty}^{\infty} I_{\lambda_0} e^{-k_\lambda(n_{1s_j})L} d\lambda}{k_\lambda(n_{1s_j})L} = \boxed{f(n_{1sj})}$$



Methods – Determination of T_e

- **T_e deduced from a simplified $1s_m$ (metastable Ar atoms) particle balance**

- **Main assumptions:**
  - **Quasi-stationnarity: $\frac{dn_{1s_m}}{dt} = 0$**
  - **$1s_m$ levels mostly created by electron impact on ground-state Ar atoms**
  - **$1s_m$ levels mostly lost by electron-impact re-excitation to 2p levels**



$$k_{Gr\rightarrow 1s_m} \cancel{n_e} n_{Ar} = \sum_i k_{1s_m\rightarrow 2p_i} \cancel{n_e} n_{1s_m}$$

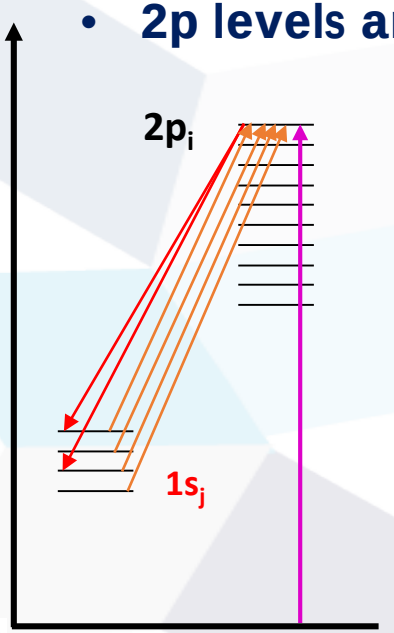
$$\Rightarrow \quad \boxed{n_{1s_m} = \frac{k_{Gr\rightarrow 1s_m}}{\sum_i k_{1s_m\rightarrow 2p_i}} n_{Ar} = f(T_e)}$$

$$k_{j\rightarrow i} = \sqrt{\frac{2}{m_e}} \int_0^\infty \sigma_{j\rightarrow i} f_e(T_e) E^{1/2} dE$$

Methods – Determination of n_e

- Time variations of n_e estimated from the line intensity of a fully-escaping radiative transition and a simplified 2p levels particle balance

- Main assumptions:
  - Quasi-stationnarity: $\frac{dn_{2p}}{dt} = 0$
  - 2p levels are mostly created by electron impact on ground-state Ar atoms and 1s levels
  - 2p levels are mostly lost by spontaneous photon emission through 2p-to-1s transitions



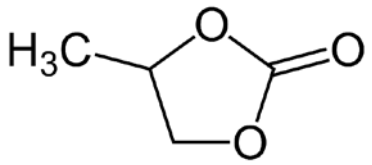
$$k_{Gr\to 2p}\, n_e\, n_{Ar} + \sum_j k_{1s_j\to 2p}\, n_e\, n_{1s_j} = \sum_j A_{ij}\theta_{ij} n_{2p_i}$$

$$\Rightarrow \quad n_{2p_i} = \frac{k_{Gr\to 2p} n_{Ar} + \sum_j k_{1s_j\to 2p} n_{1s_j}}{\sum_j A_{ij}\theta_{ij}}\, n_e$$

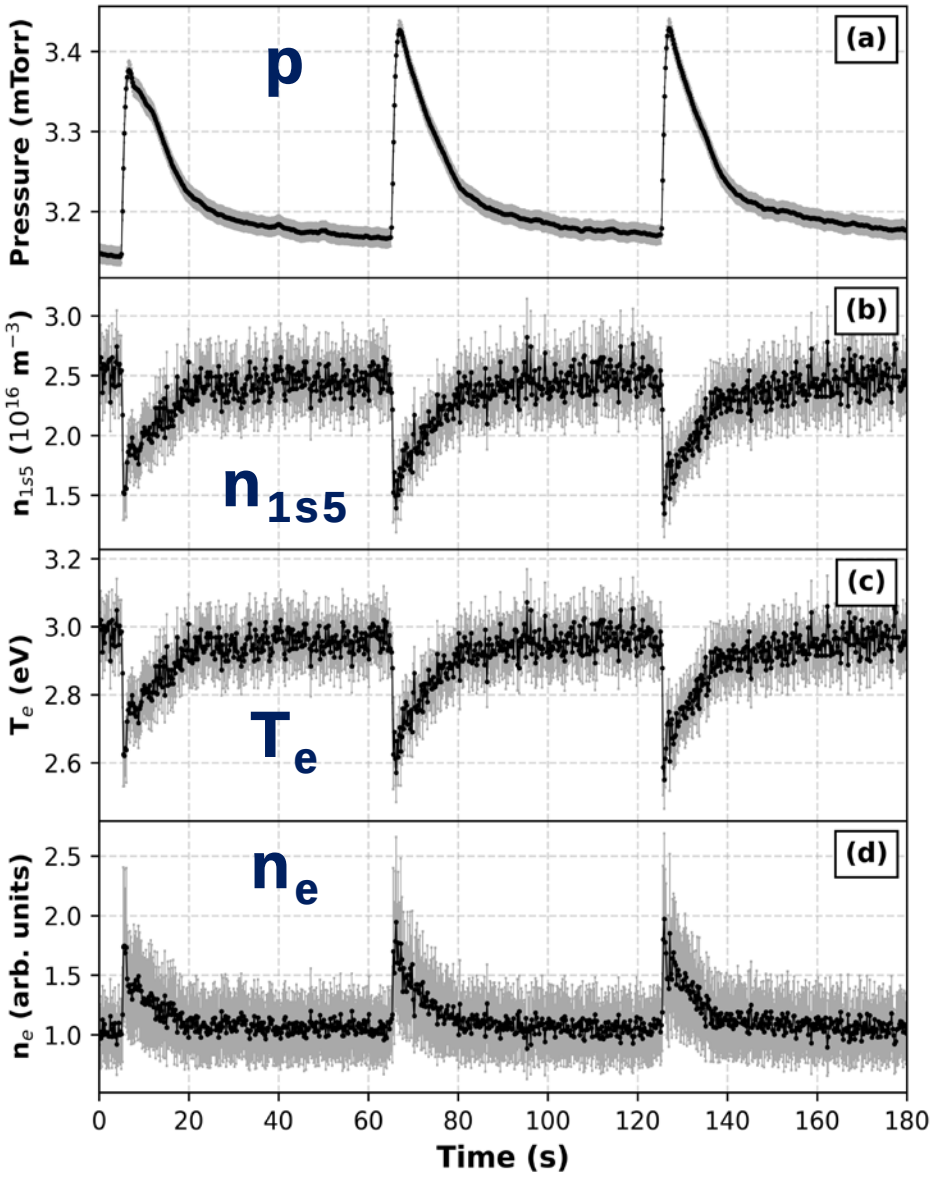
$$I_\lambda = n_{2p_i}\, A_{ij}\, \theta_{ij} \quad \Rightarrow \quad \frac{I_\lambda}{k_{Gr\to 2p} n_{Ar} + \sum_j k_{1s_j\to 2p} n_{1s_j}} \propto n_e$$

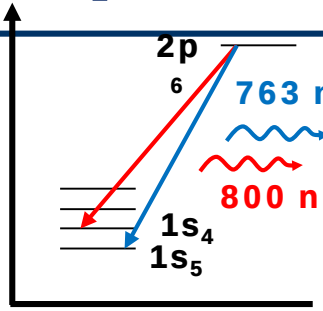
Results – PC added to the argon plasma

Propylene Carbonate



Low Vapor pressure at 300 K = 10 Pa





n_{1s5} measured through radiation trapping:

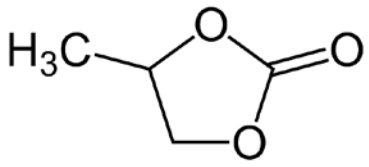
$$\theta_{763} \approx \frac{A_{800}\, I(763\ nm)}{A_{763}\, I(800\ nm)} = f(n_{1s_5})$$

- quick drop of Ar $1s_5$ metastable density upon liquid injection, then slow increase back up to its nominal value
- T_e decrease attributed to the creation of new vapor species (solvent, CH_x…) with lower E_{ion}
- n_e increase also attributed to the facilitated ionization

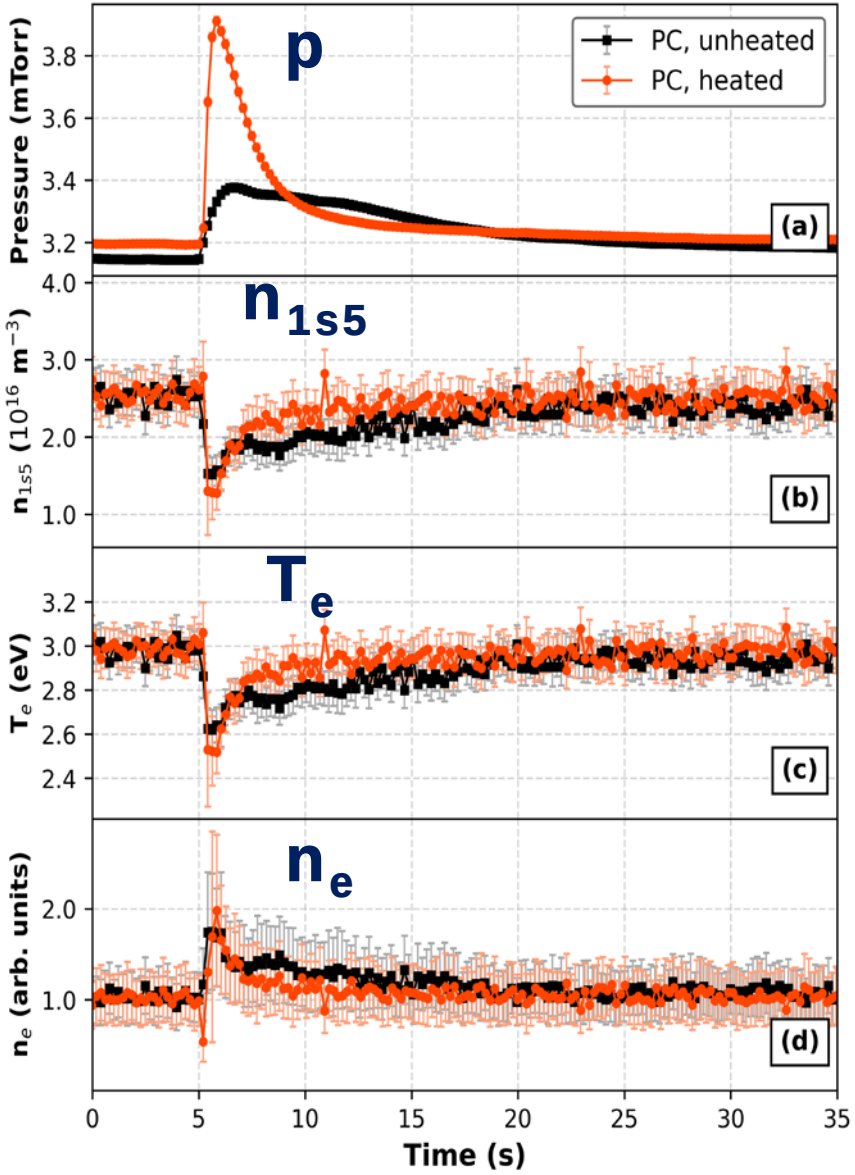
S. Chouteau et al, *PSST* **33,** 075016 (2024)

Results – heated and unheated PC added to the Ar plasma

Propylene Carbonate



Strong increase of PC vapor pressure when heated (423 K)

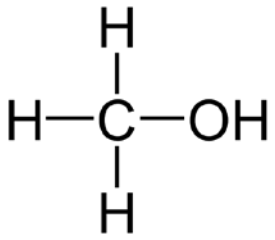


- **similar trends for p, n_{1S5}, T_e, n_e (t) but with more pronounced and faster kinetics when PC is heated (especially for p(t))**
- **effects attributed to the increased volatility of PC**

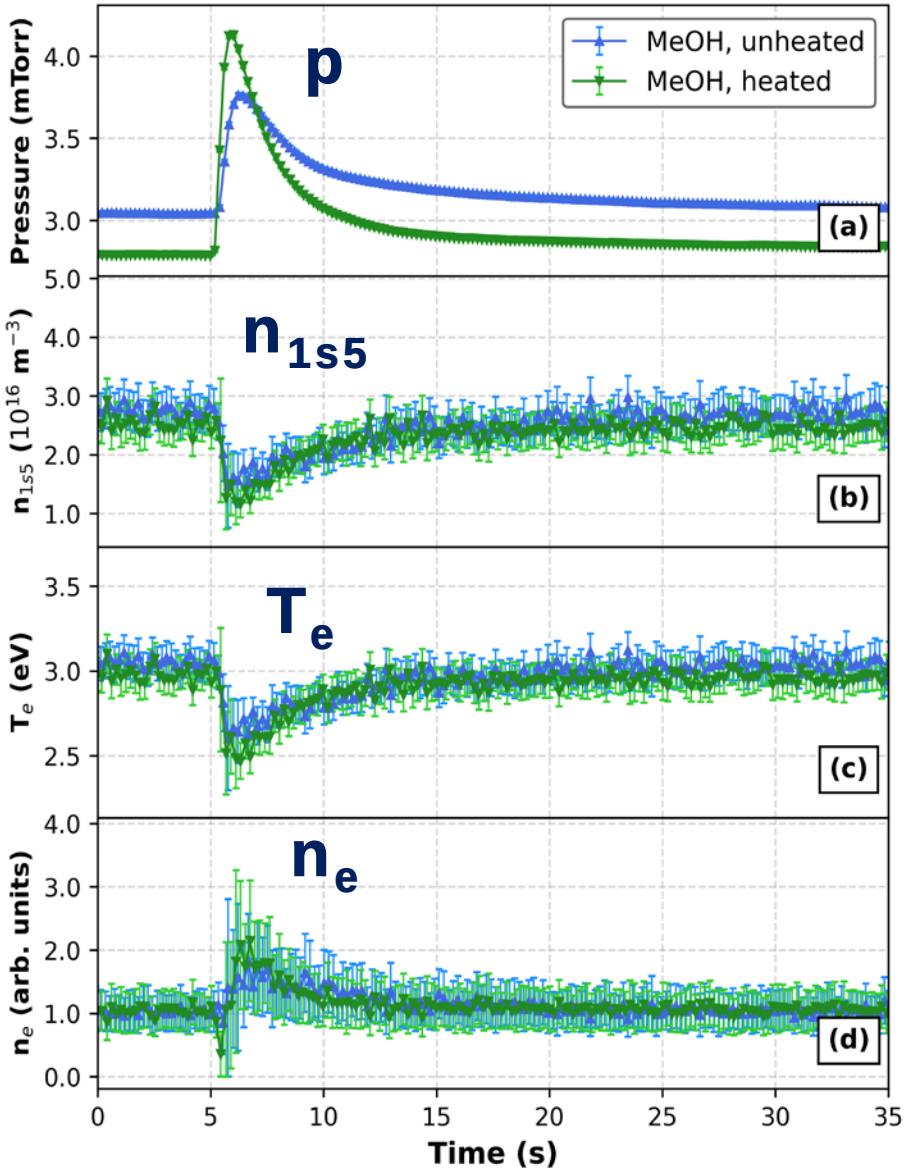
S. Chouteau et al, *PSST* **33,** 075016 (2024)

Results – Methanol added to the argon plasma

Methanol



High vapor pressure at 300 K = 19 kPa



- similar results for n_{1S5}, T_e, n_e (t) as in the case of PC injection
- only a small effect of the heating due to the already high volatility of methanol at 300 K

S. Chouteau et al, *PSST* **33,** 075016 (2024)

Ar/solvent plasma : impact of solvent droplets

- Measurements and simple modelling of n_{1s}, T_e and n_e carried out as a first step towards the spectroscopic analysis of a low-pressure misty plasma
- Liquid injection apparently triggered facilitated ionization, leading to a drop in electron temperature and an increase in electron density
- MCR model developed by S. Chouteau to determine the time variations of N_{meta}, n_e and T_e from the plasma emission spectrum

Outline

- Introduction/context : nanocomposite films deposition by injecting a colloidal solution in a PECVD plasma (misty plasma, aerosol assisted plasma deposition)

- Study of TiO_2-SiO_2 nanocomposite films deposition
  - Time resolved OES and *in situ spectroscopic* ellipsometry
  - Evaporation of solution droplets in the plasma
  - n_{meta}, T_e, $n_e(t)$ in Ar/ solvent plasmas

- Optimisation of ZnO-SiO_2 nanocomposite films deposition by TROES

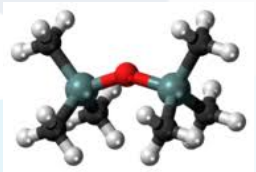
- Conclusion / Perspectives

$ZnO-SiO_2$ NC films deposition

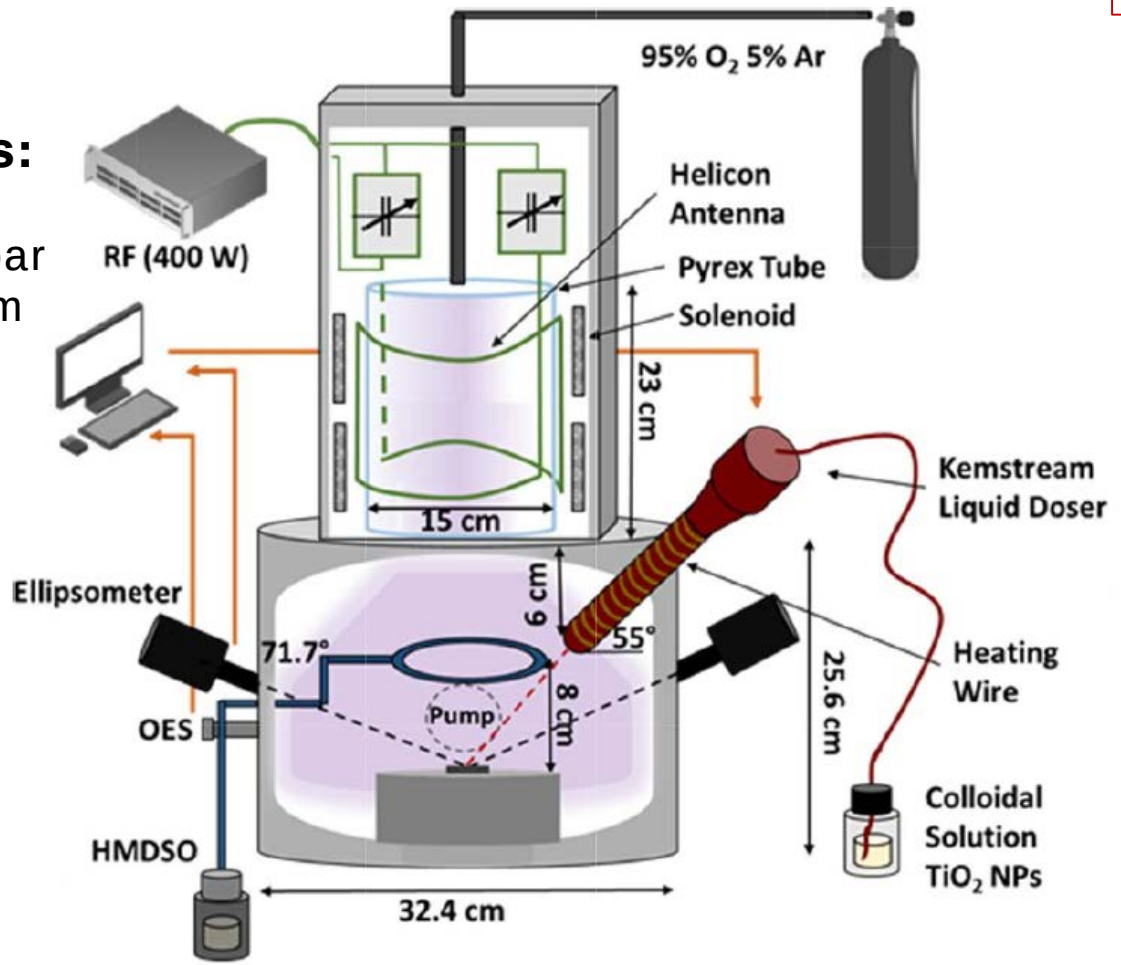
PhD Julien Chevet (2021-2025)

Plasma conditions:

- Pressure: 4 10^{-3} mbar
- O_2 flowrate: 24 sccm
- Power: 400 W



- **HMDSO** flowrate : 0.11 sccm



ZnO NP obtained by hydrolysis of zinc dicyclohexyl

Nanoparticle solution:

- **ZnO NPs** (5 nm in diameter) C=0.25 mol/L
- oleic acid C=0.05 mol/L
- dodecylamine C=0.25 mol/L
- in **pentane**

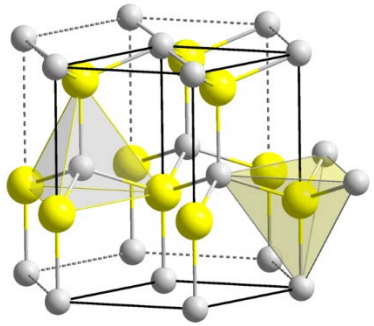
LCC

M.L. Khan et al., *Adv. Funct. Mater*, 2005, 458

LuMINA ANR-21-CE08-0011

ZnO and ZnO NPs properties

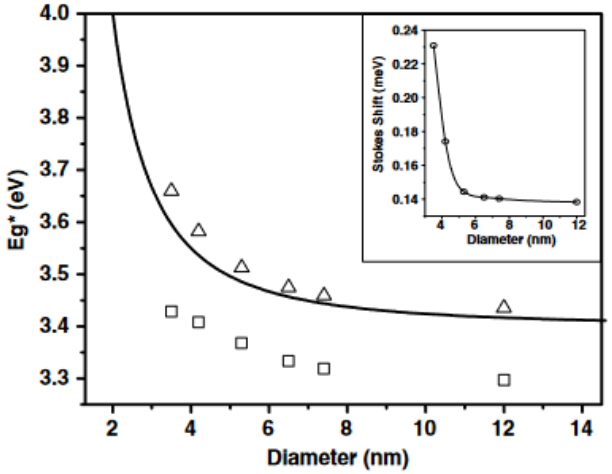
Bulk ZnO:
direct gap = 3,37 eV



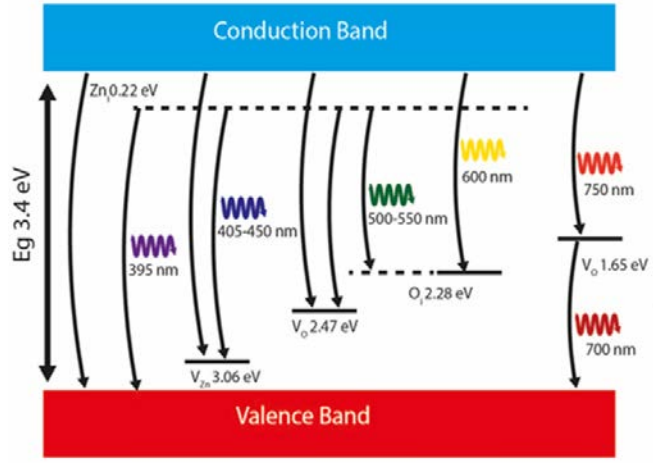
ZnO wurtzite

ZnO powder

Gap and PL spectra of ZnO NPs

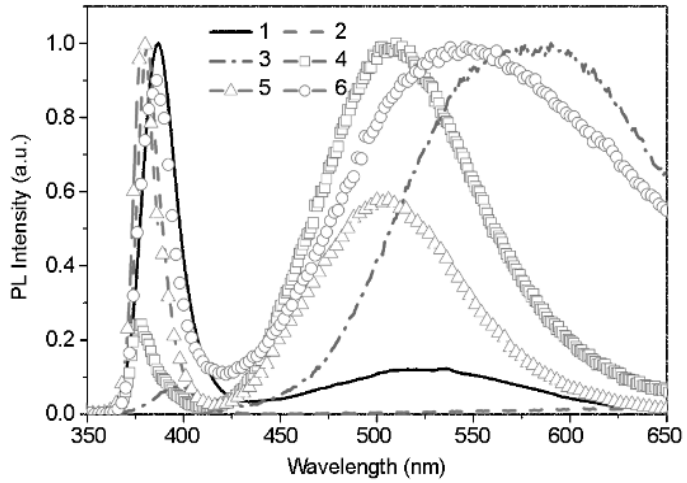


Kuo-Feng Linet et al, Chem Phys Lett. 2005



Deep level emissions

Galdámez-Martinez et al., Nanomaterials 2020, 10, 857



A.B. Djurisic et al, Small 2 (2006) 944-961

- excitonic emission (UV)
- strong luminescence in the visible range

TiO_2 anatase NPs colloidal solution:

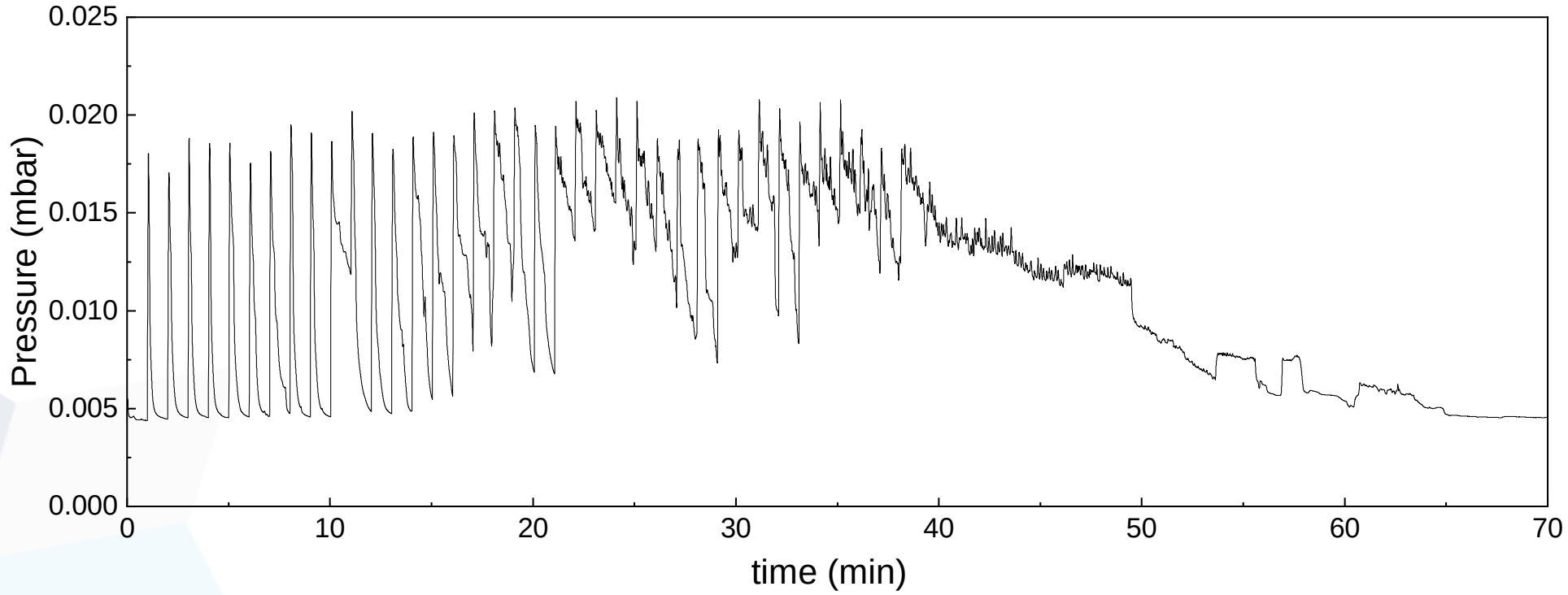
Solvents	Propylene carbonate PC	Propylene glycol PG	Methanol
Boiling point	242 °C	188 °C	65 °C
Vapor pressure at 25°C	4 Pa	11 Pa	12300 Pa
Volatility	-	-	+ + +

ZnO NPs solution:

	Ligand: Oleic Acid (OA)	Ligand: Dodecylamine (DDA)	solvent: Pentane	solvent: HMDSO	solvent: heptane
Boiling point	360 °C	259 °C	36 °C	101 °C	98°C
Vapor pressure	131 Pa at 176°C	8400 Pa at 170 °C	53300 Pa at 18,5°C	2000 Pa at 25°C	6090 Pa at 25°C
Volatility	-	-	+ + +	+	+

Time variation of p: HMDSO/pentane 80:20 + OA + DDA

N = 2

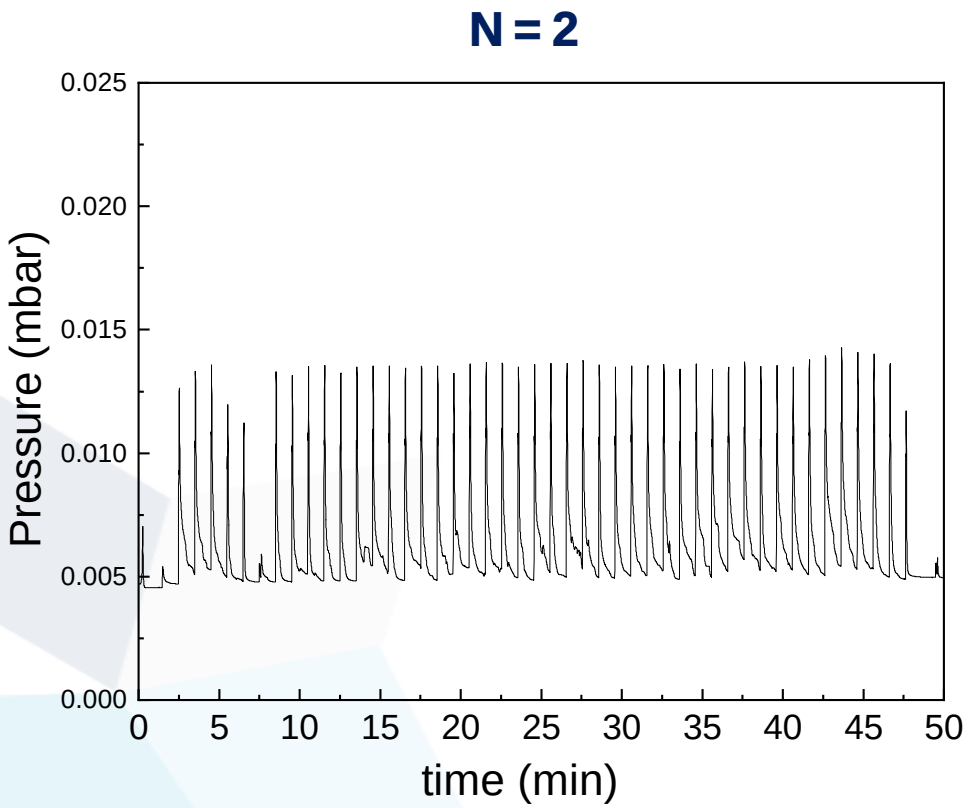


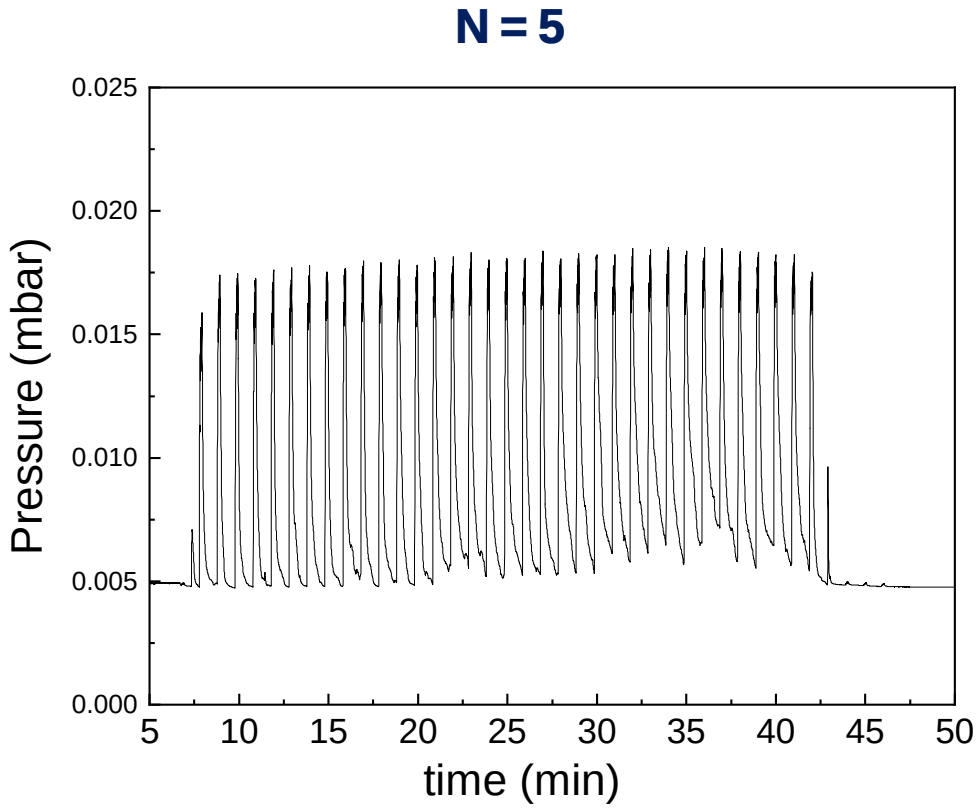
liquid injection for t > 15 min

➔ non regular injections and pressure increase

➔ HMDSO/pentane abandoned and replaced by heptane

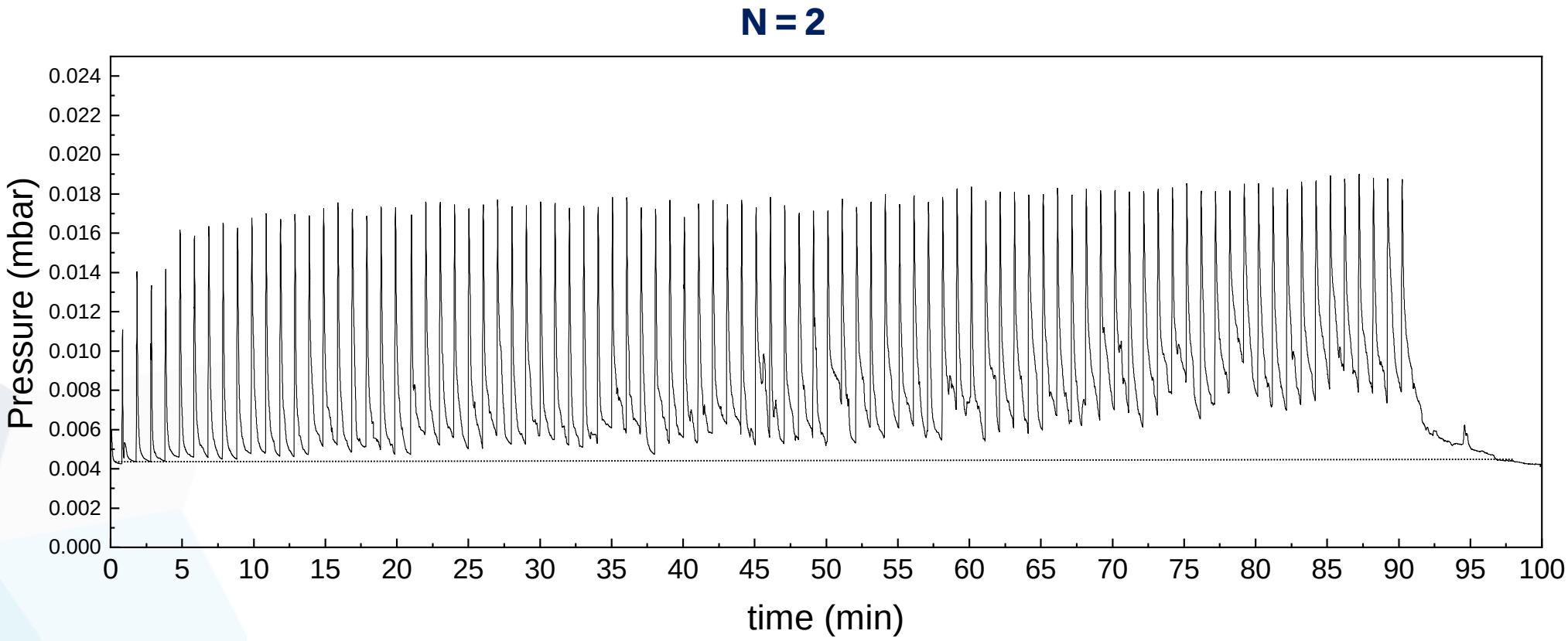
Time variation of pressure : heptane + OA + DDA





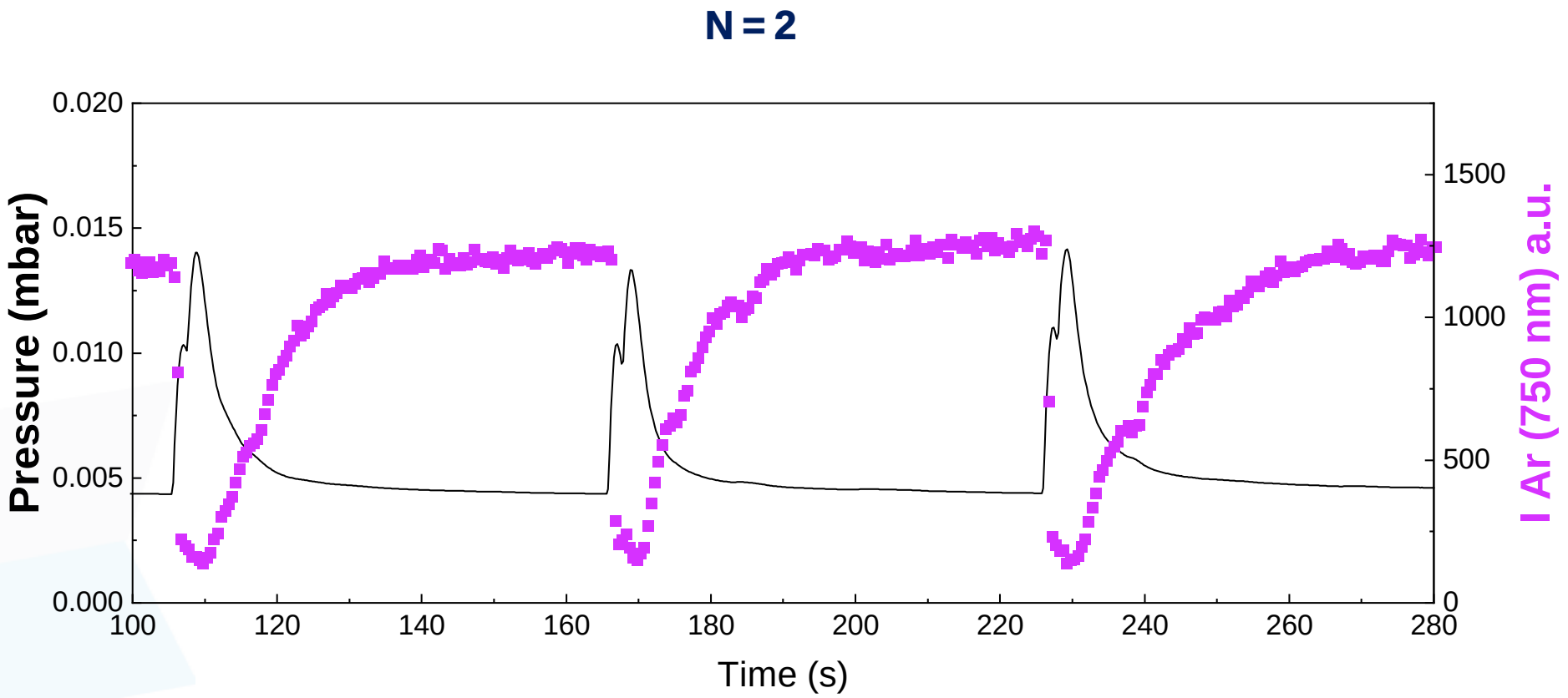
- **Good reproducibility of the liquid injections during 1 h for N = 2**
- **some drift of the pressure observed for t > 30 min for N = 5**

Time variation of pressure : heptane + ZnO NPs +OA + DDA



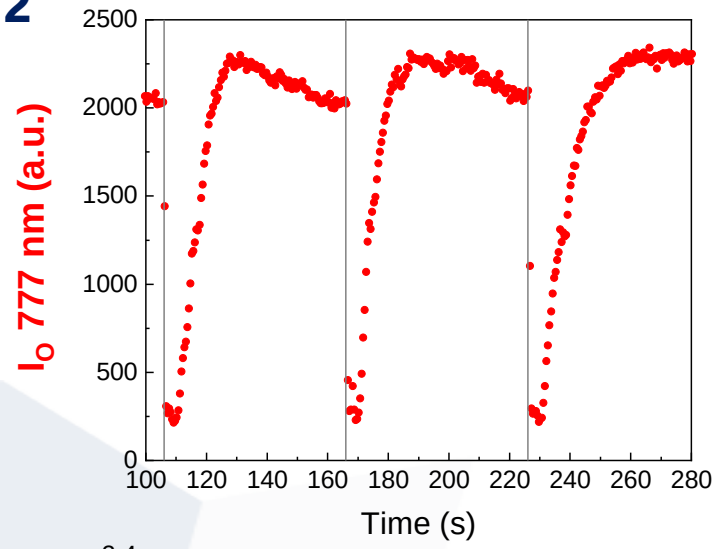
- **stable process during 1 h**
- **small increase of the base pressure for t > 1h**

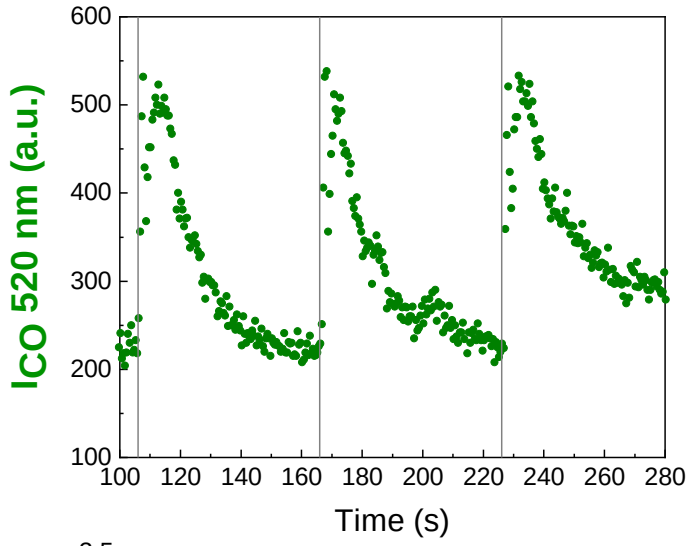
TROES: heptane + ZnO NPs +OA + DDA

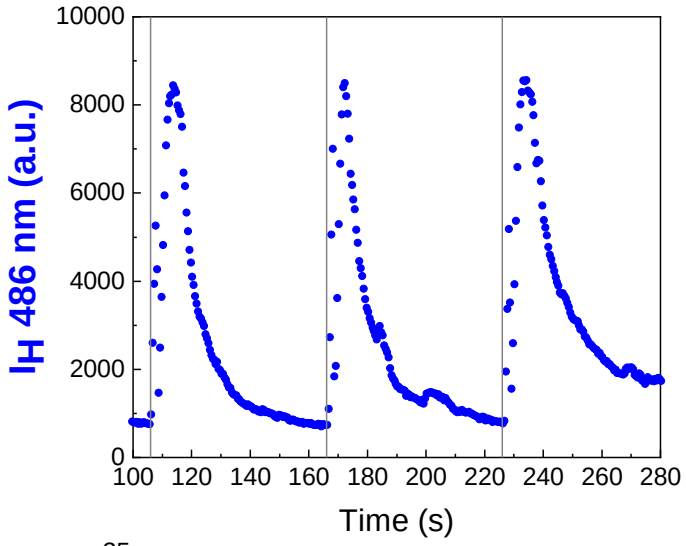


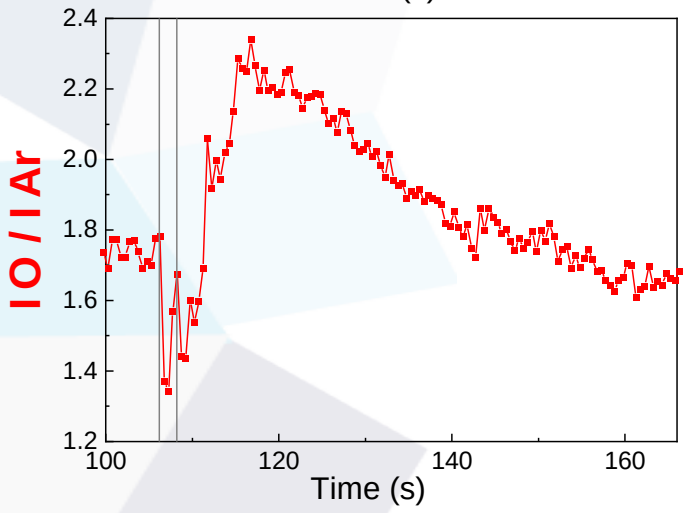
TROES measurements

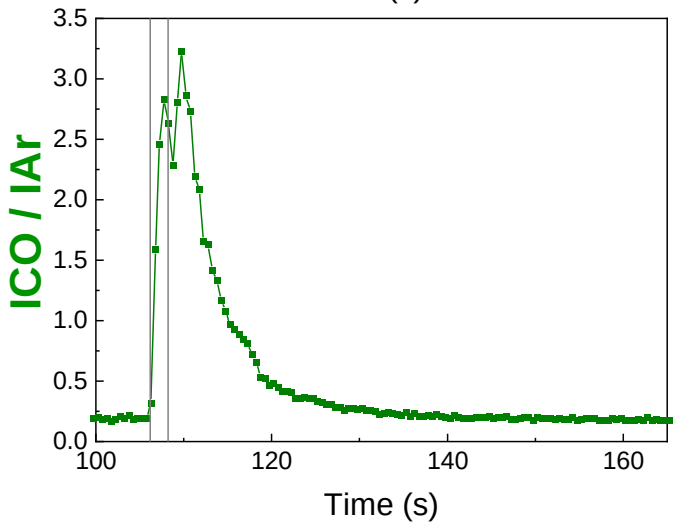
N = 2

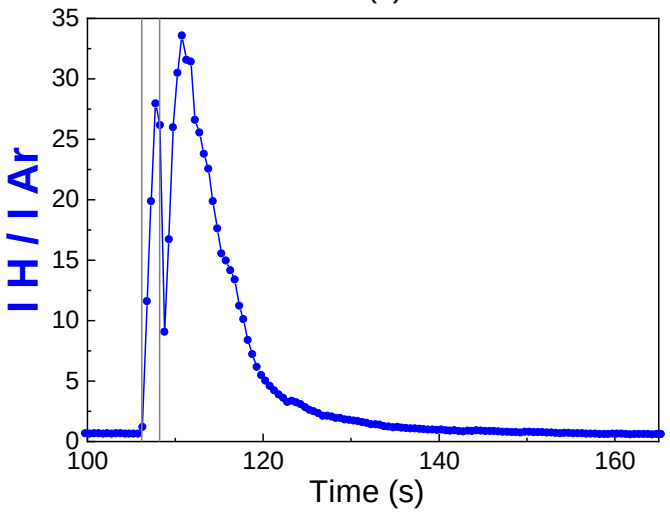












NC Film Analysis

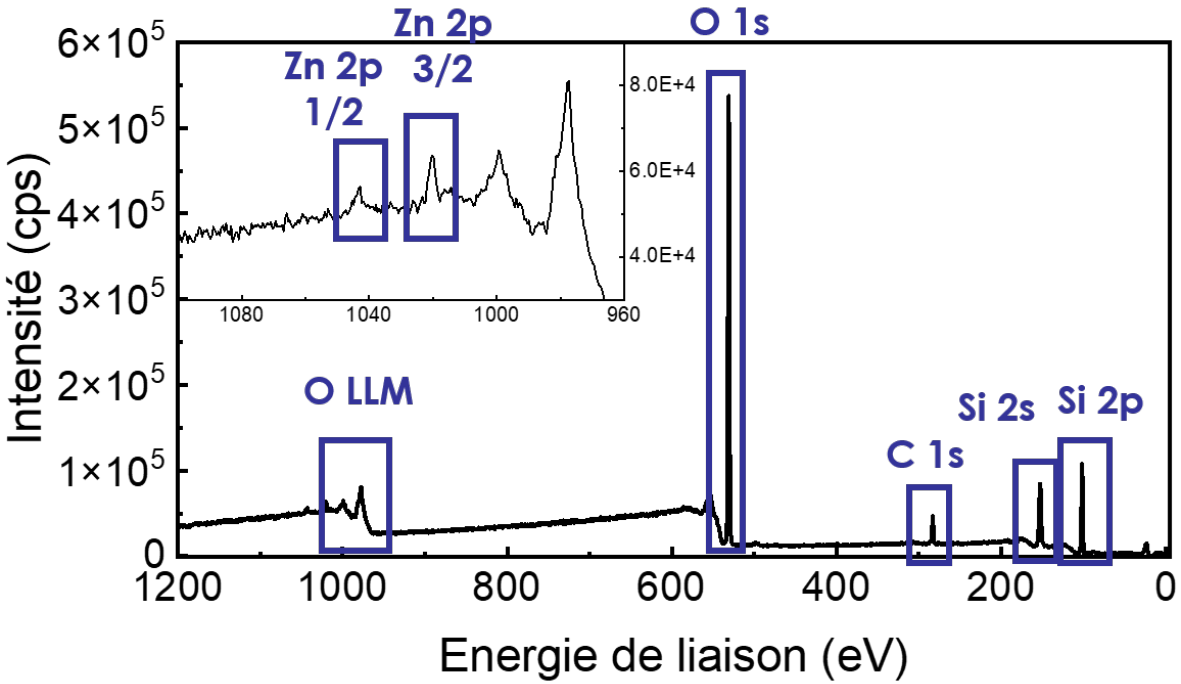
N = 2, d = 200 nm

XPS spectra :

=> SiO_2 matrix and ZnO NP

ITO substrate
Kratos Nova
Source Al Kα 1486,6 eV
Pass energy 80 eV

Energy resolution 0.95 eV
Step scan 0.5 eV
Slot 700 x 300 µm²



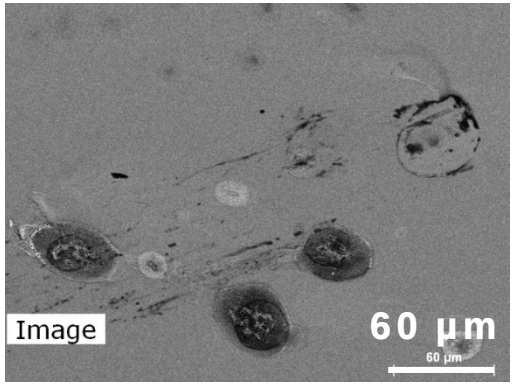
Spectroscopic ellipsometry (*in situ*):

=> ellipsometric data very well fitted using only the SiO_2 matrix (due to the low fraction of ZnO NPs in the NC film)

=> determination on the film thickness (d)

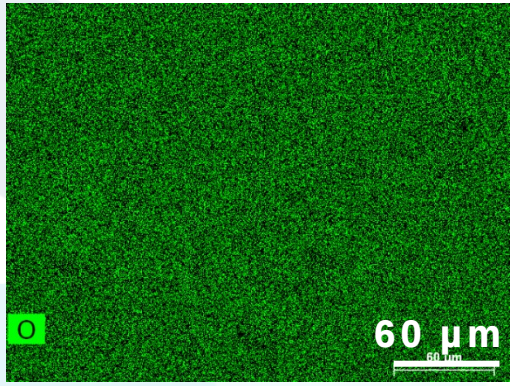
NC film: SEM image and EDX mapping

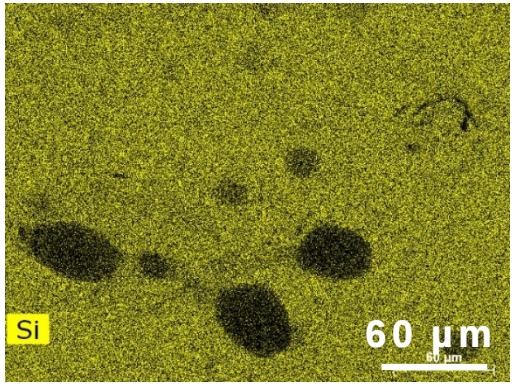
N = 2

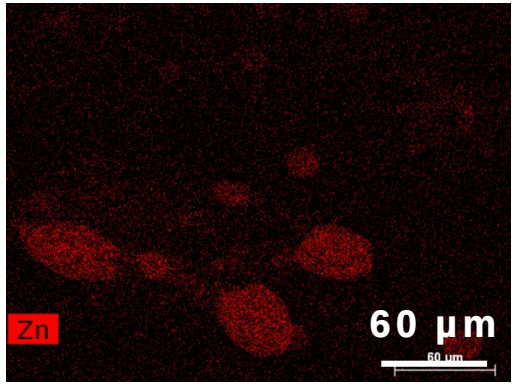


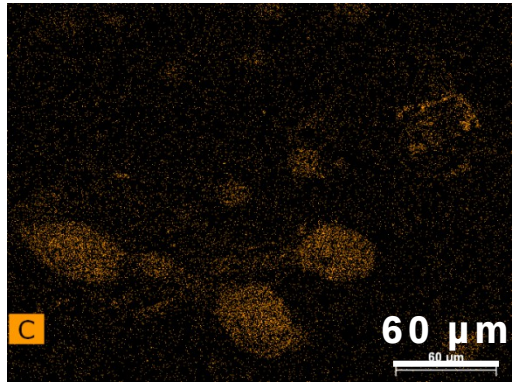
SEM image in secondary electrons

x 400





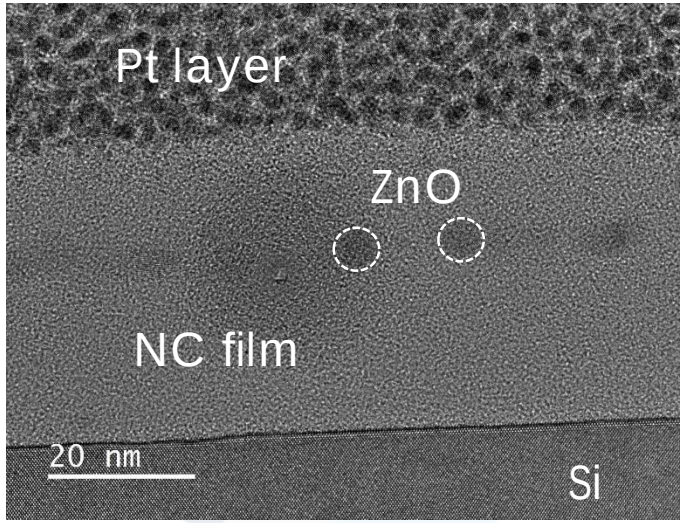




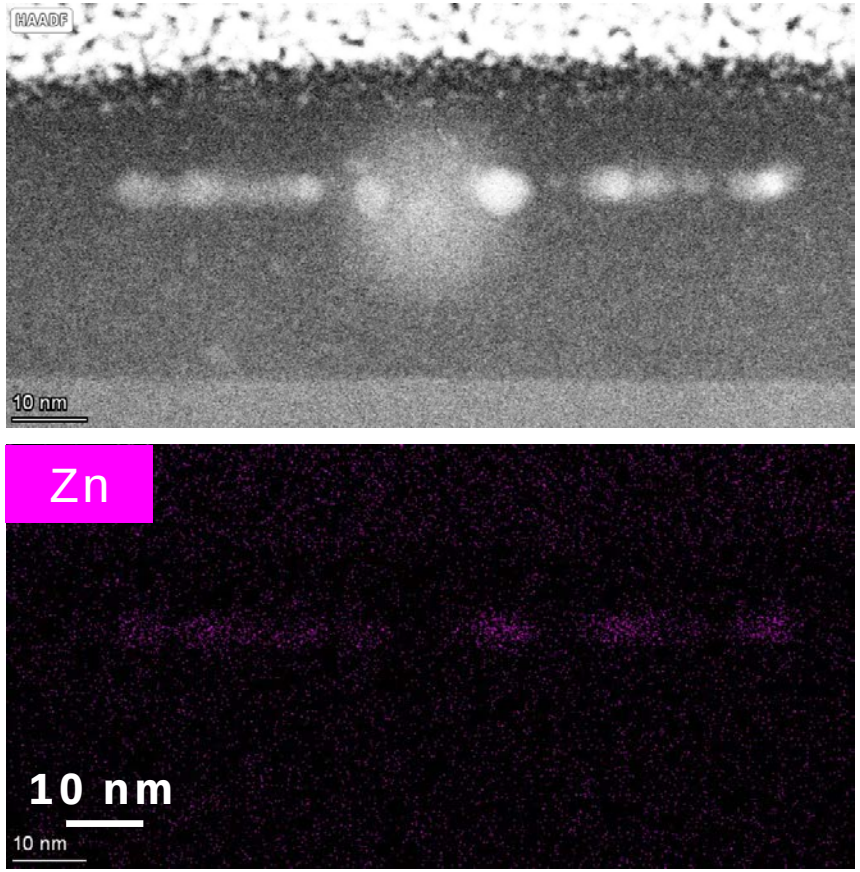
Drop imprints observed on the top of the NC film

NC film: TEM analysis of a FIB cross section

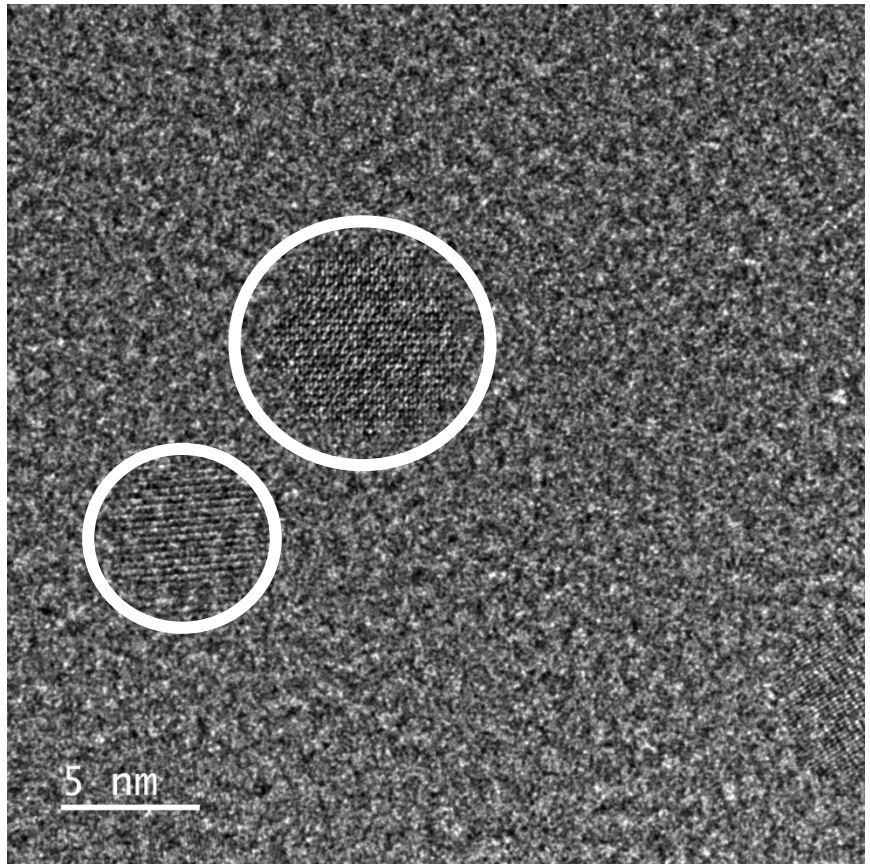
N = 4



x
230 000



HAADF and EDX mapping
x 690 000

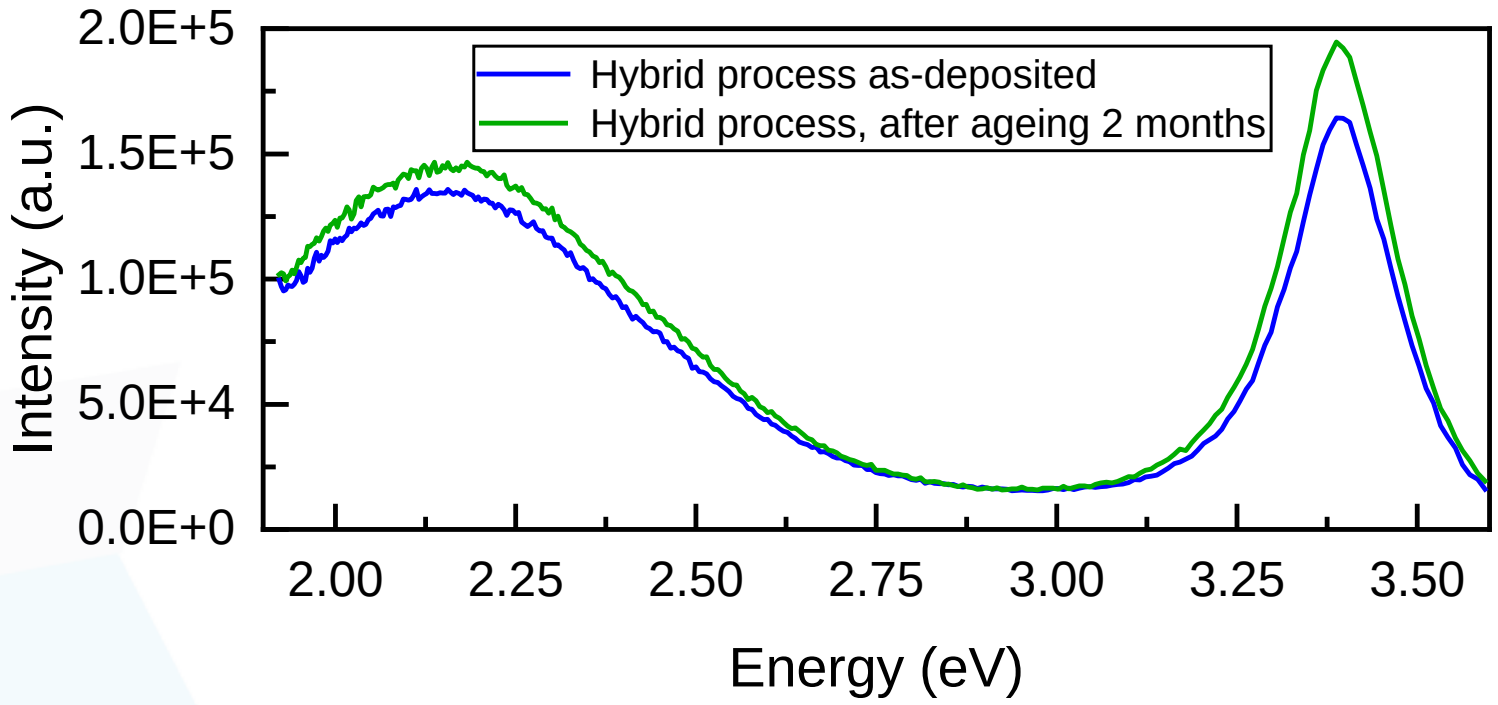


TEM image x 1100000

NC film Photoluminescence

N = 4

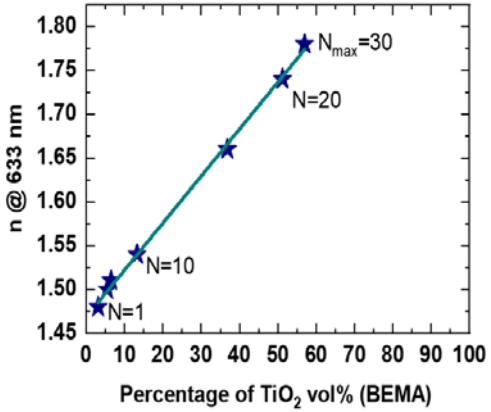
Excitation at 330 nm



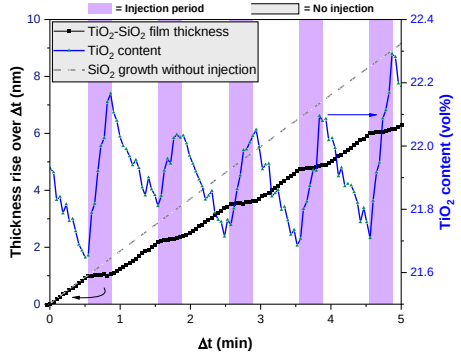
- **Photoluminescence of ZnO NP (excitonic and visible emissions)**
- **ZnO NP efficiently protected by the SiO_2 matrix (PL conserved upon ageing)**

Conclusion

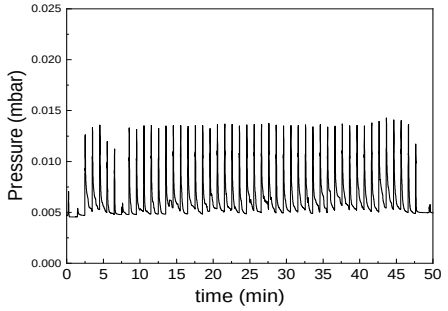
- A wide range of TiO_2 NPs fraction in a SiO_2 matrix (1 to 58%) obtained by varying the number of solution injections in a sequence with a relatively good dispersion of the NPS in the matrix



- Ellipsometry => insight into the NC film deposition kinetics: during the injection periods : decrease of the SiO_2 matrix deposition rate (due to n_e and T_e decrease)



- Plasma time resolved diagnostics of the plasma (pressure, TROES...)
  → better understanding of the plasma/droplet interactions
  → optimisation of the NC deposition process



Conclusion : misty plasmas / aerosol assisted plasma deposition

- Large range of NC films available…..

  But some mandatory conditions….
  - adapted colloidal solution : stable, ecofriendly solvents with vapor pressure compatible with the pulsed injection in the plasma
  - compatibility between the PECVD gas mixture used for the matrix deposition and the solvent

- some examples :
  →ZnO NPs in a SiO_2 matrix, Au NP in SiO_2 matrix…..
  → NP in carbon matrix, using Ar plasma and the solvent as precursors for the matrix deposition

- in progress : Fe_20_3 NPs in TiO_2 matrix

Perspectives : plasma diagnostics with a better time (and spatial) resolution

Thank you to all the contributors
Thank you for your attention