An original tuneable plasma process for the synthesis of tailored nanoparticles

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Namur Institute of Structured Matter

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Outline

The process

A versatile plasma process

Some plasma diagnostic

A first approach to study the degradation kinetic of organometallic

The different type of nanoparticles

Tailor-made nanoparticles

Conclusions & perspectives

1. The process

A versatile plasma process

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The process

An easy plasma process

Plasma degradation of a solid organometallic precursor (M-acac) mixed with powder substrate, in a inert or reactive atmosphere

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- ICP-RF plasma 13.56 MHz

The process

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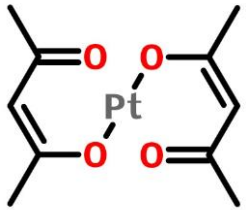
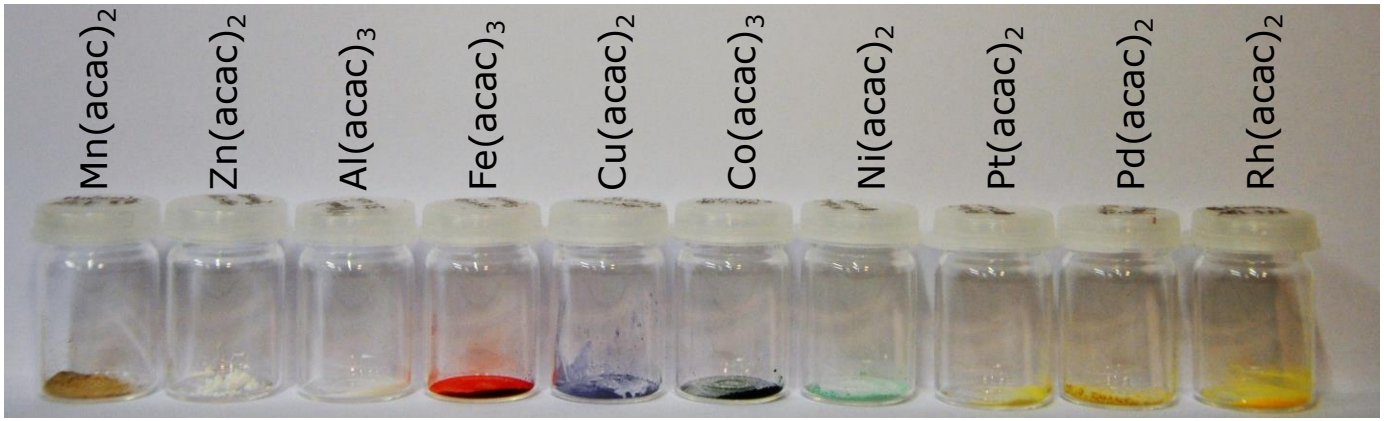
Plasma degradation of a solid organometallic precursor (M-acac) mixed with powder substrate, in a inert or reactive atmosphere

- Metal acetylacetonate





The process

An easy plasma process

Plasma degradation of a solid organometallic precursor (M-acac) mixed with powder substrate in a inert or reactive atmosphere

Graphene nanoplatelets
Carbon black
Nanotubes
Carbon xerogel
TiO_2

The process

An easy plasma process

Plasma degradation of a solid organometallic precursor (M-acac) mixed with powder substrate, in a inert or reactive atmosphere

Argon
Oxygen
Nitrogen
Ammonia

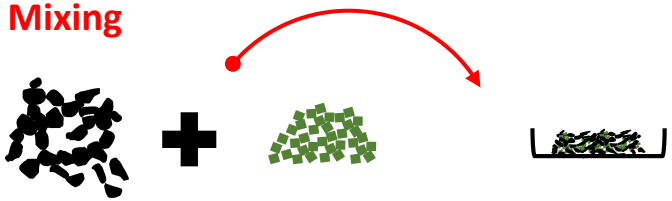
The process - methodology

Methodology

The process - methodology

Methodology

I. Mixing



Powdery material

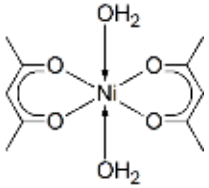
Solid OM precuror

Raw materials:

Mesoporous carbon xerogel

Graphene, xerogel, $TiO_{2, ...}$

M(acac) powder precursor (crystalline)



The process - methodology

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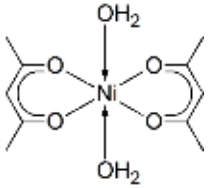
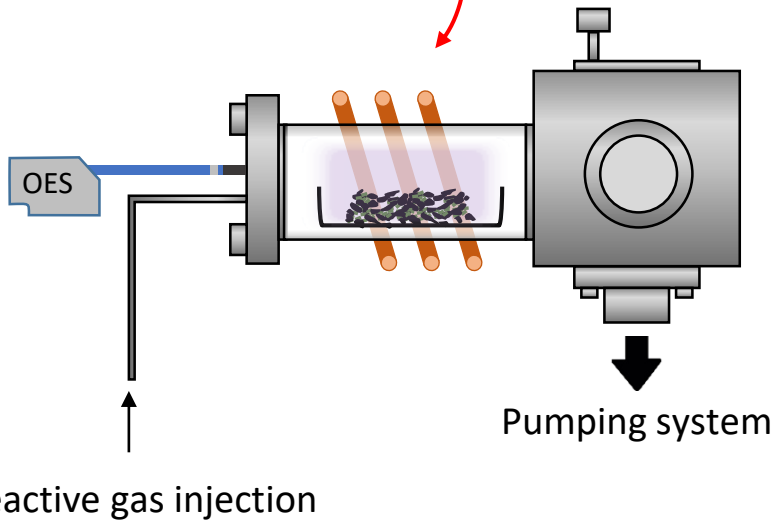


I. Mixing

II. Plasma treatment



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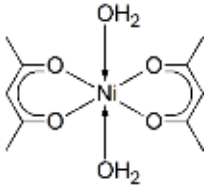

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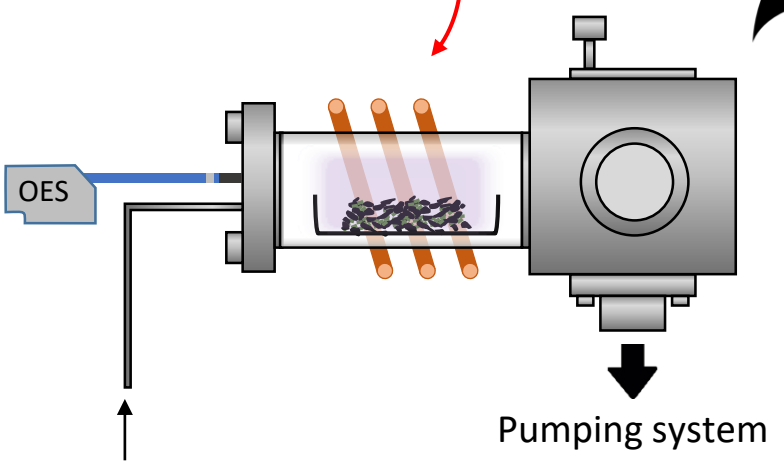
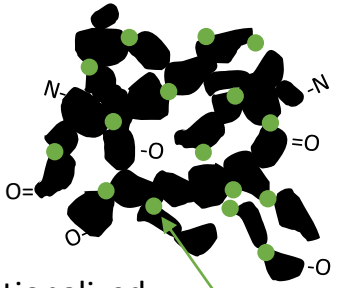
II. Plasma treatment



Reactive gas injection

Pumping system

III. Nanoparticles formation



Functionalized carbon xerogel

Nanoparticles

The process - methodology

Methodology

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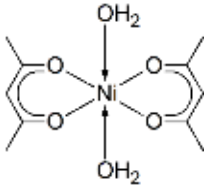

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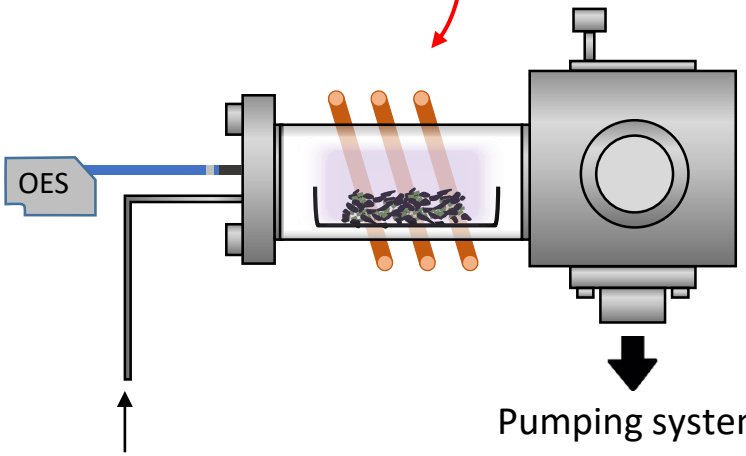

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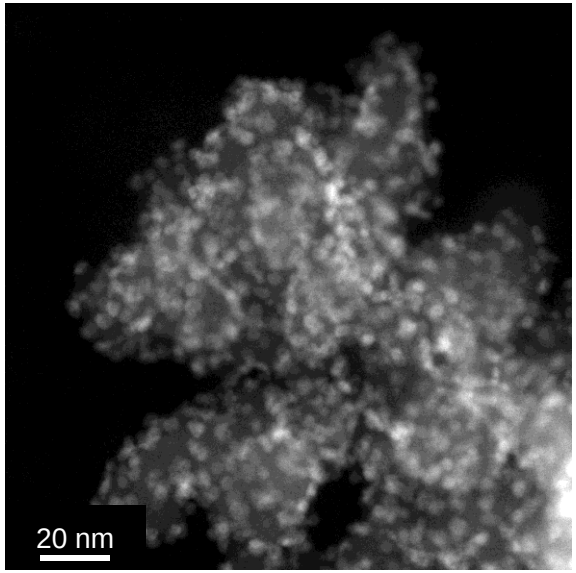
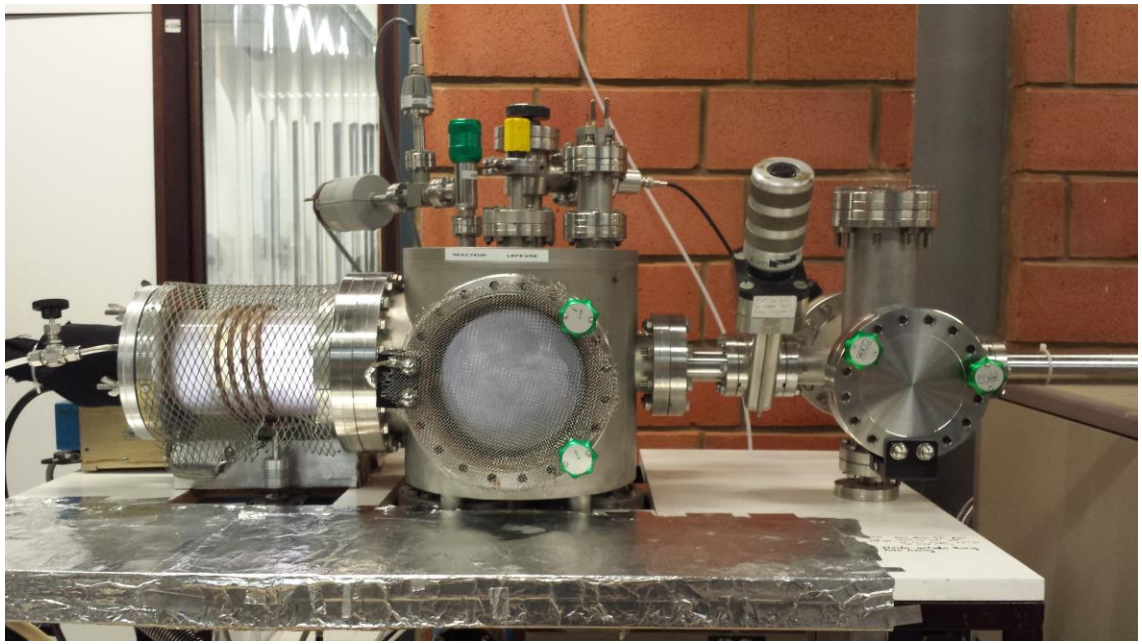


The process – Pros & Cons



Variable parameters
Pressure
Power/time
Gas (Ar/O_2, N_2 or NH_3)

Pros
Dry & cheap* process
Short (≈1h), low temperature
Upscalable
Substrate functionalization
Control of the chemistry, size and crystallinity
Plasma diagnostic

Cons
Difficult to adjust the metal loading
What happens to organic part?
Poor control of the morphology

** 0.5-2€/g for Fe, Cu, Zn, or $Mn(acac)_2$ compared to Fe sputtering target ≈20-30€*

2. Some plasma diagnostic

A first approach to study the degradation kinetic of organometallic

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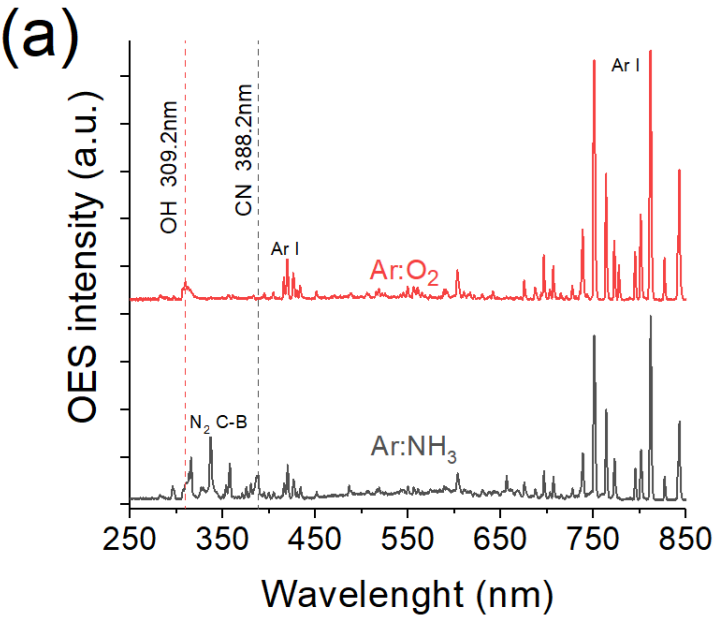
Plasma diagnostic – degradation kinetic of organometallic

Plasma treatment of Ni(acac) in different atmosphere



Plasma emission at the beginning of the treatment

HAYE et al., ACS Appl. Nano Mater. 1, 1, 265-273 (2018)

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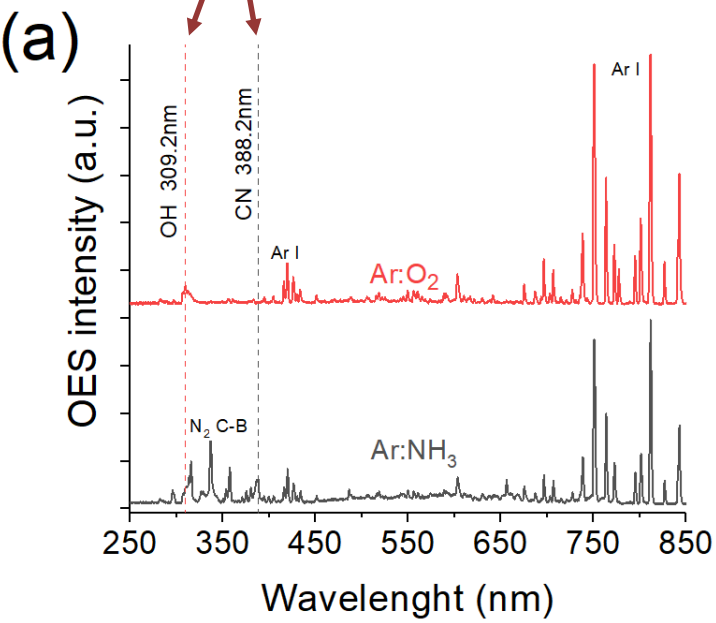
Organic lines: rise and fall evolution with time (**OH** or **CN**)

Ar lines: constant intensity with time



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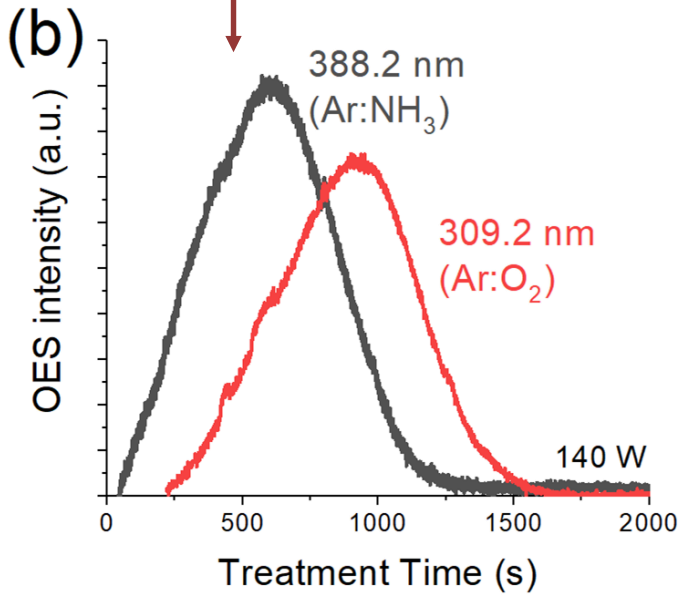

Evolution of OH lines for Ar:O_2 and CN lines for Ar:NH_3 treatment with time

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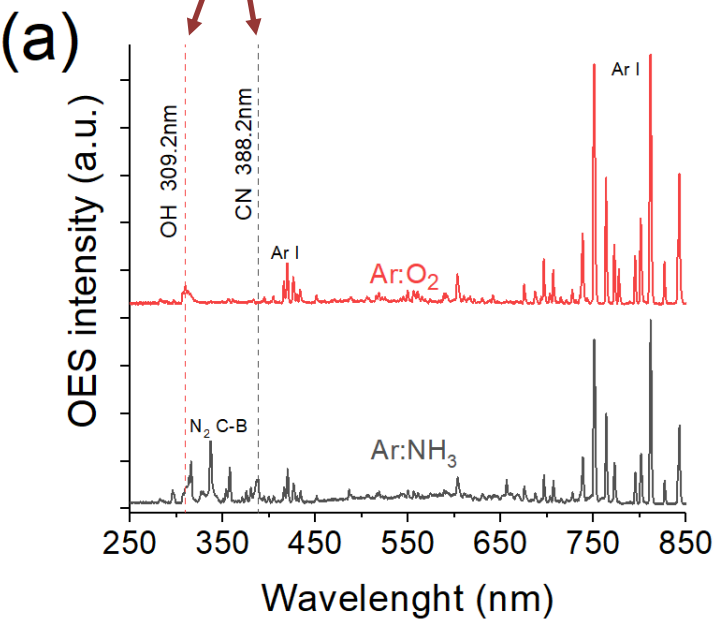
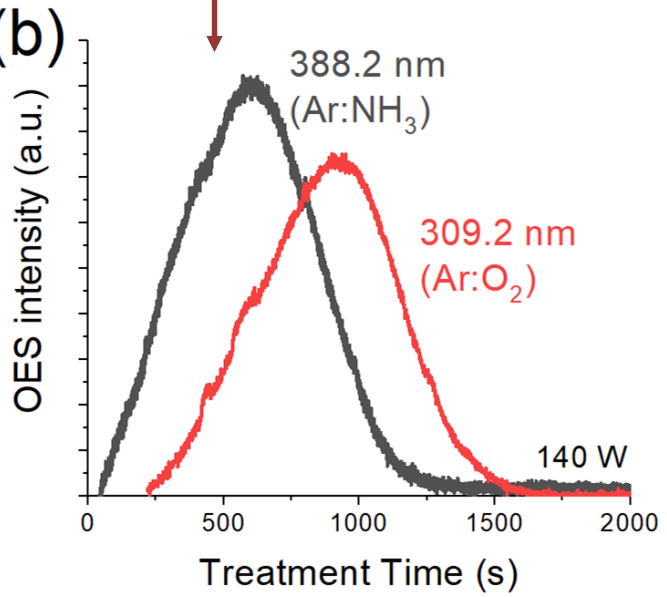


Plasma emission at the beginning of the treatment

In-situ monitoring of the precursor degradation



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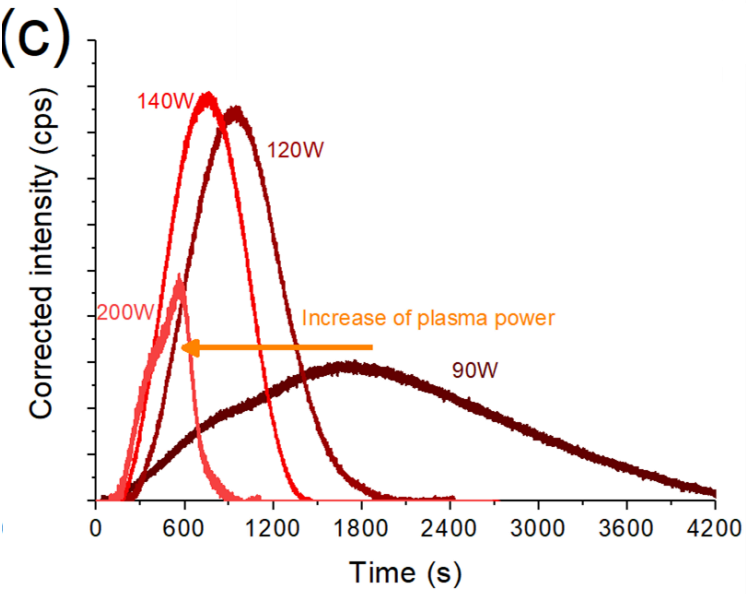

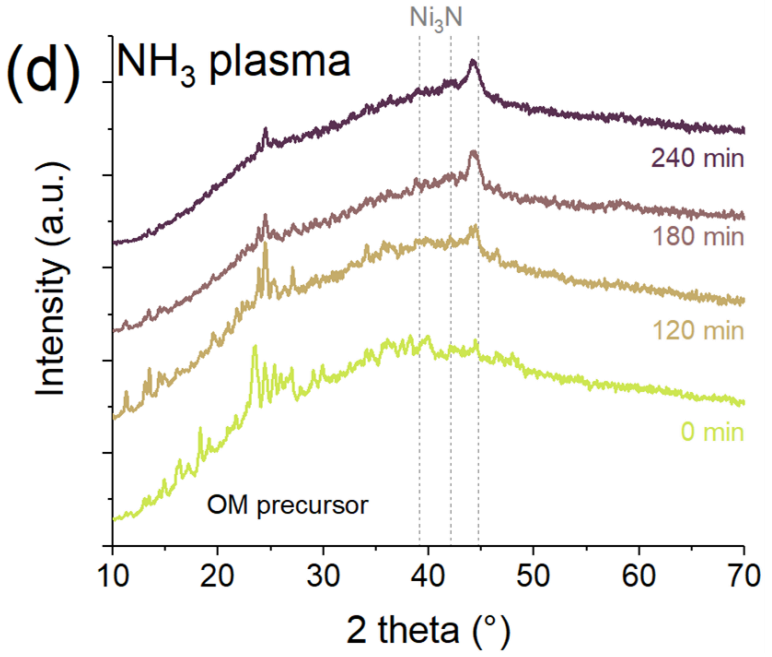

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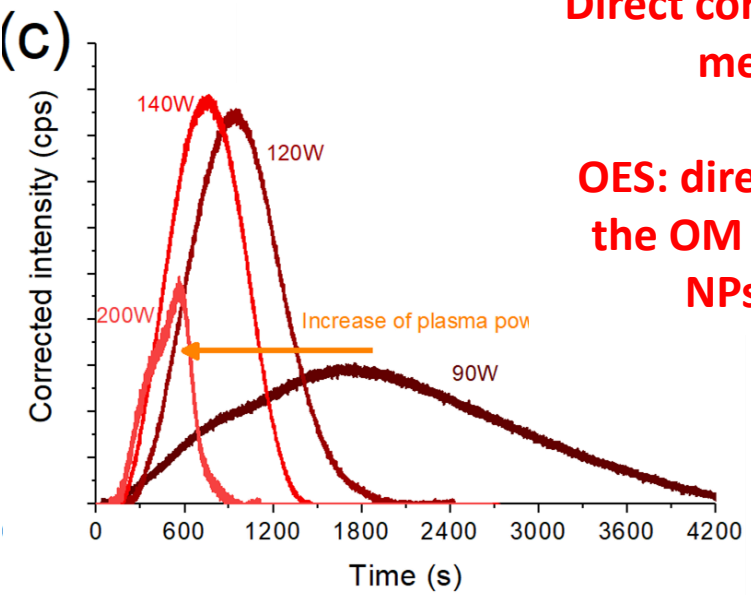
Plasma diagnostic – degradation kinetic of organometallic

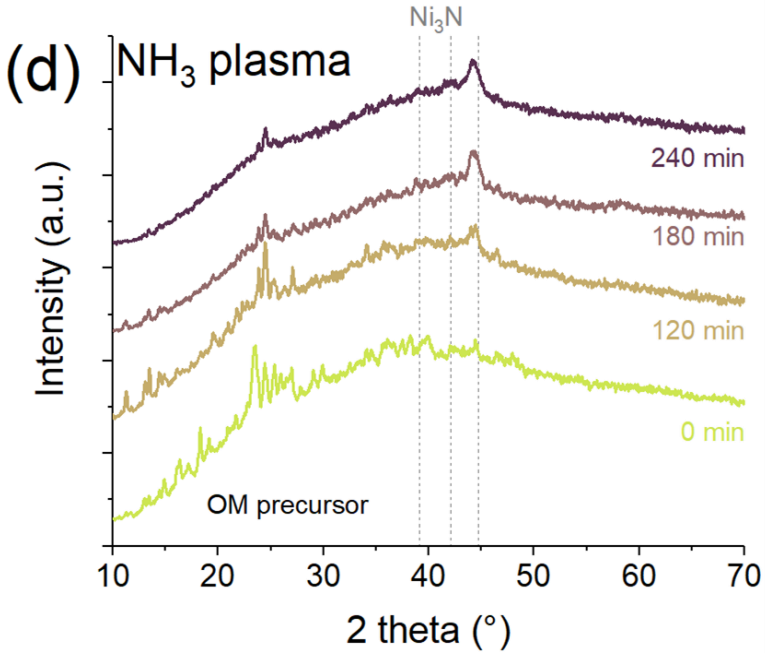
Plasma treatment of Ni(acac) in different atmosphere

Shift of the evolution with the plasma power

Direct correlation with XRD measurement

OES: direct observation of the OM degradation and NPs generation





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3. The different nanoparticles

Tailor made nanoparticles

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The different nanoparticles – some examples

Fe_3N nanoparticles on carbon xerogel

$Fe(acac)_2$ + XG in

$Ar:NH_3$

60min, 200W

Variation of the pressure

6 to 45 mTorr (0.8 to 6 Pa)

HAYE et al., ACS Appl. Nano Mater. 2, 4, 2484-2493 (2019)

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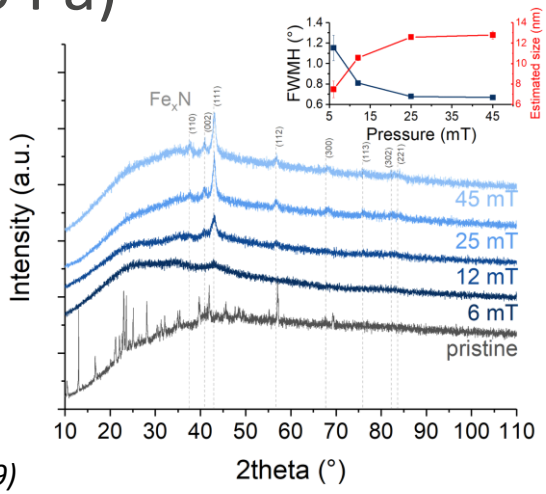

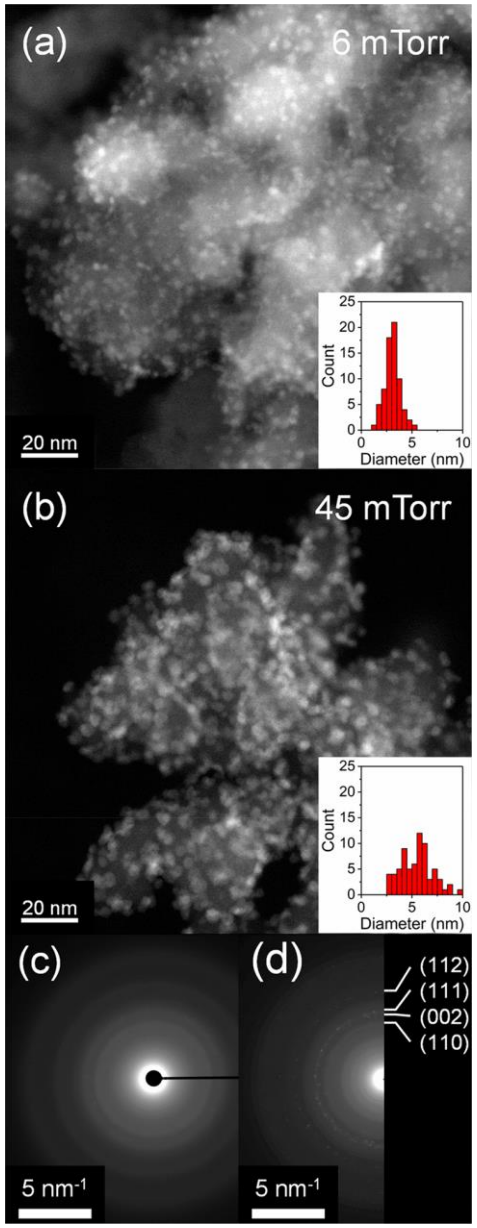

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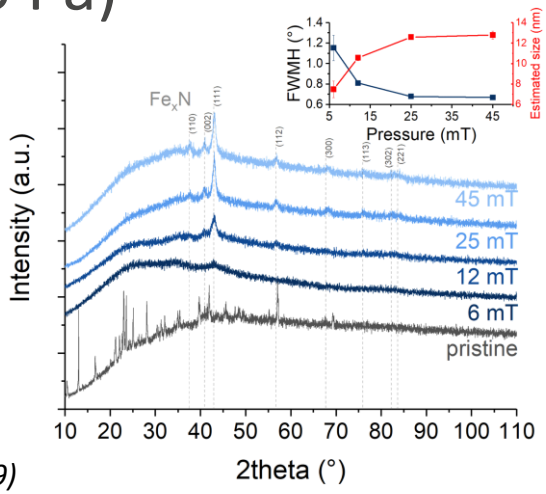
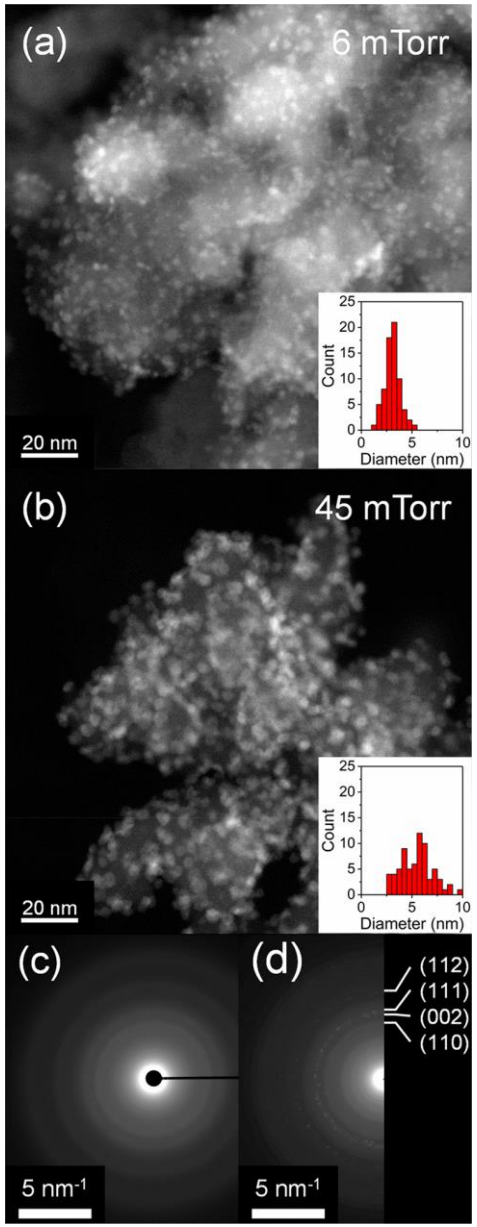
Change of crystallinity





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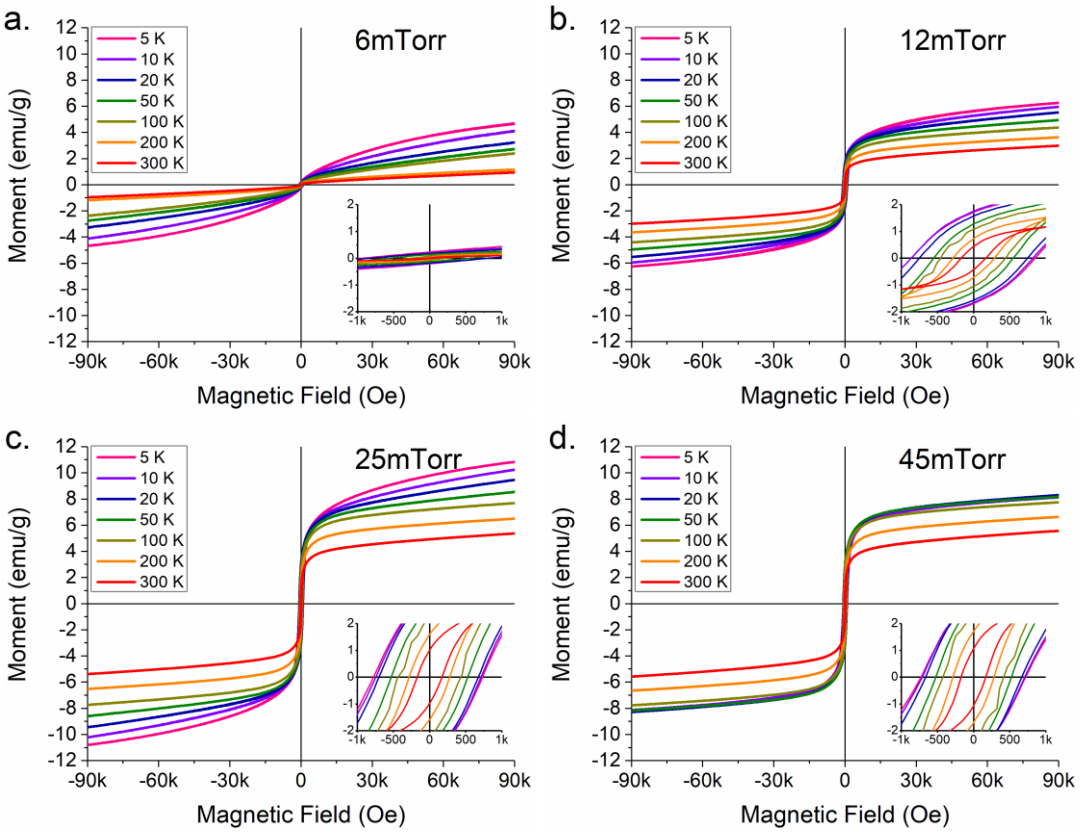
Magnetic nanoparticle



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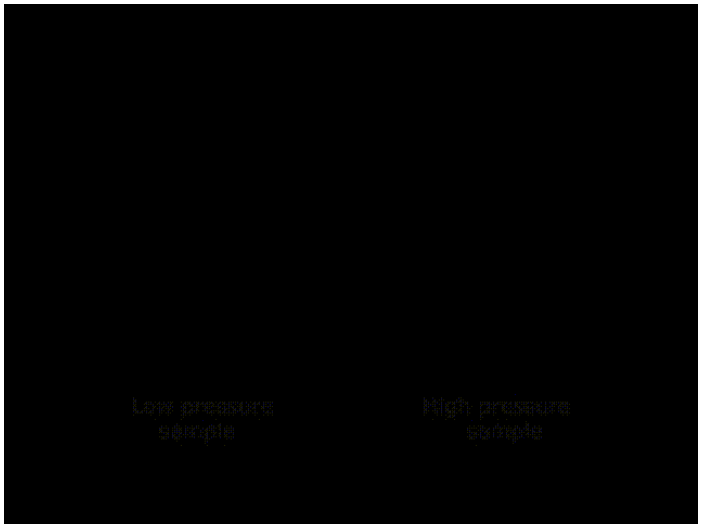
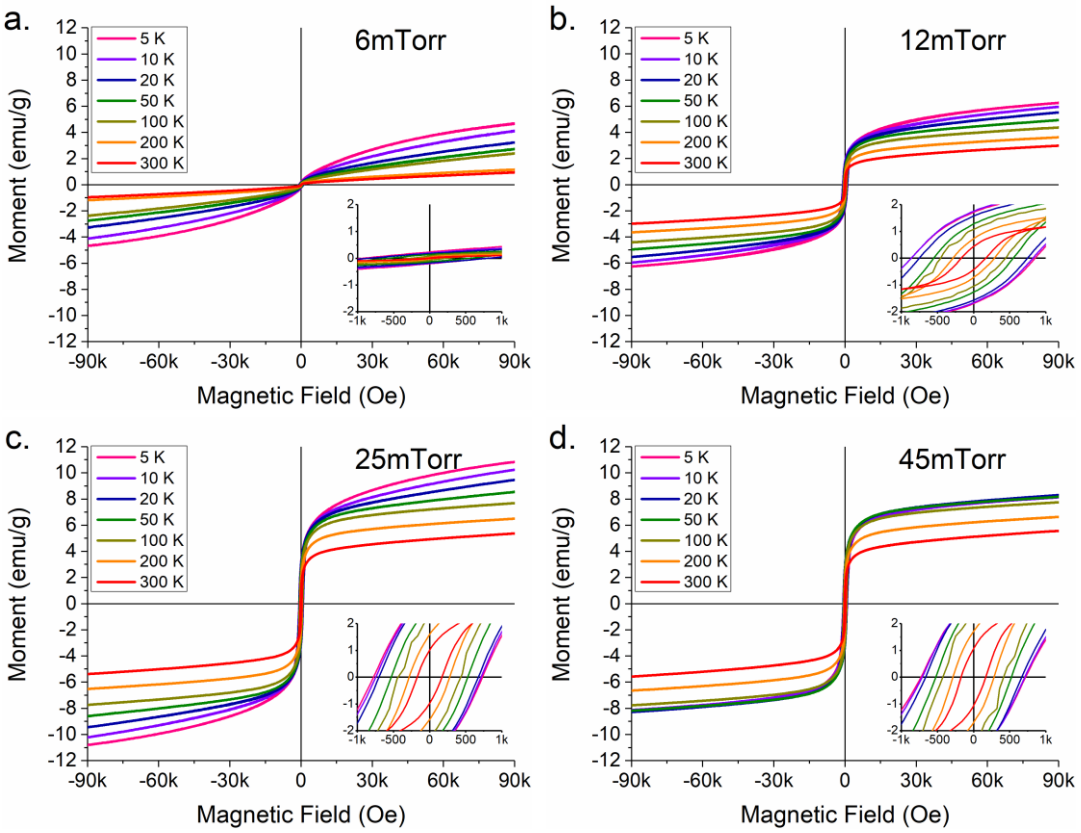
Magnetic nanoparticle

Control of the crystallinity
→ control of the magnetism





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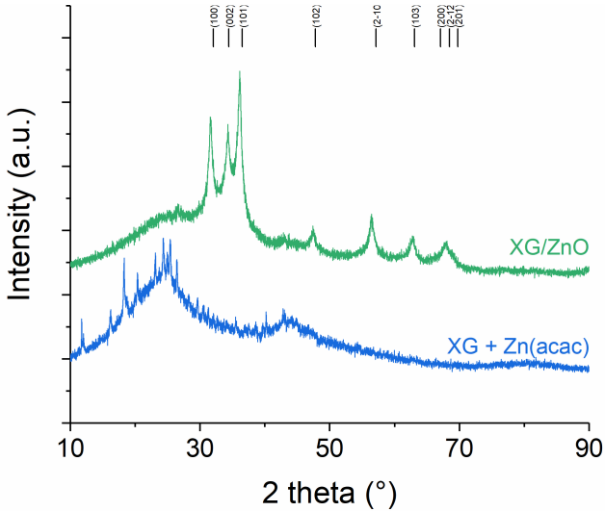
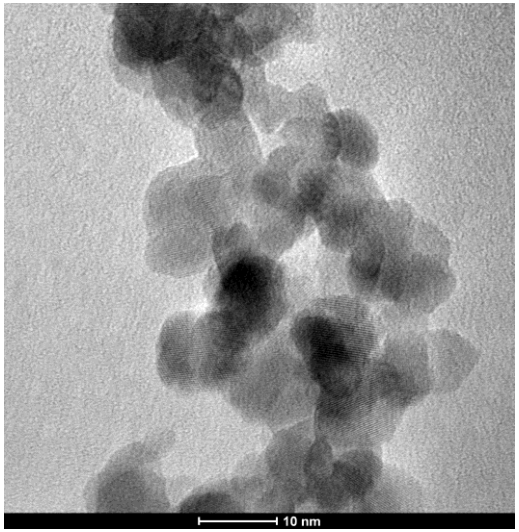


Chains of ZnO nanoparticles
ZnO NPs anchored to XG



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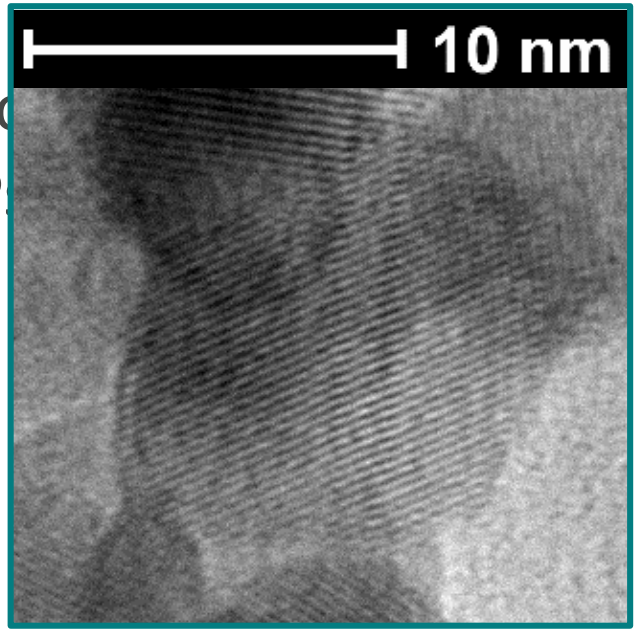

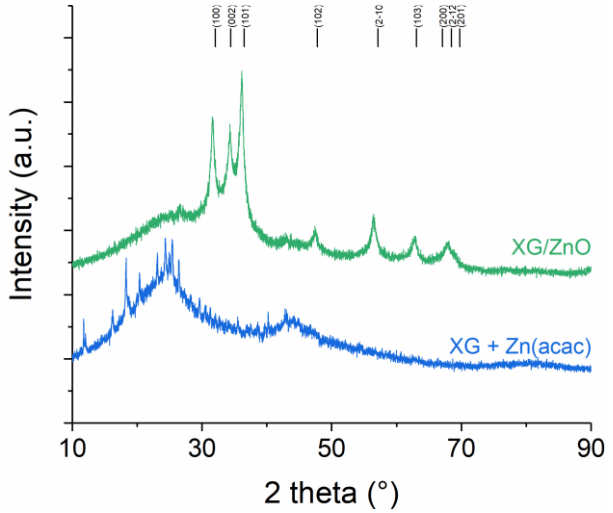

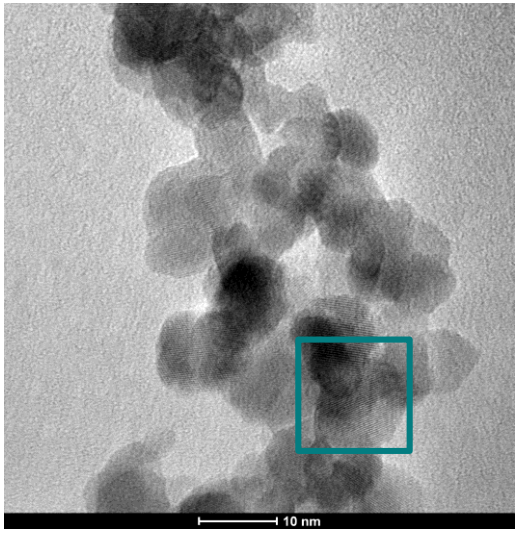

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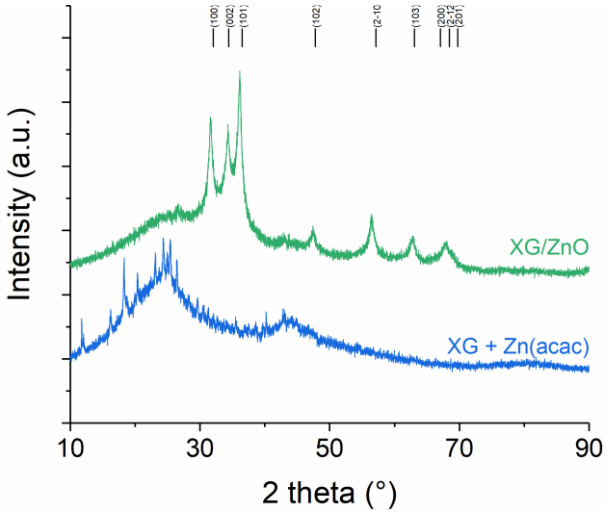

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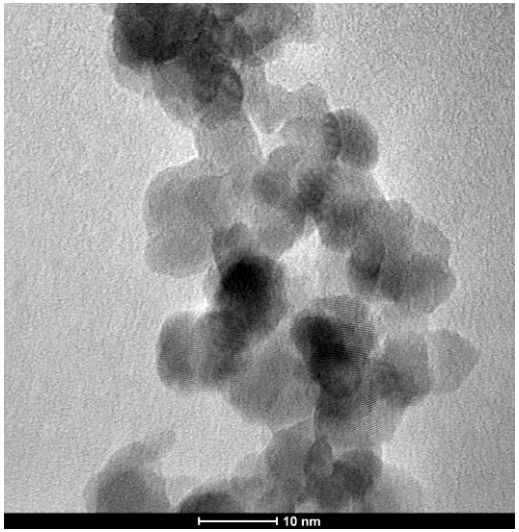


DUAL EFFECT: ZnO nanoparticles synthesis
+
carbon functionalization
Comparison with pure C_{XG} treated in O_2 or NH_3

HAYE et al., submitted in Journal of Colloid and Interface Science

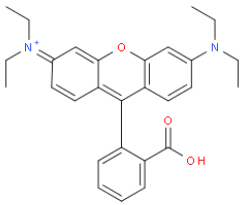
The different nanoparticles – some examples

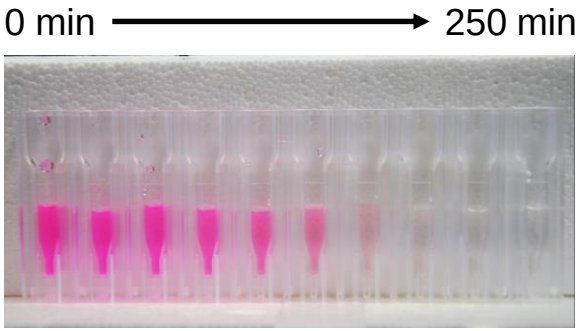
ZnO nanoparticles on carbon xerogel

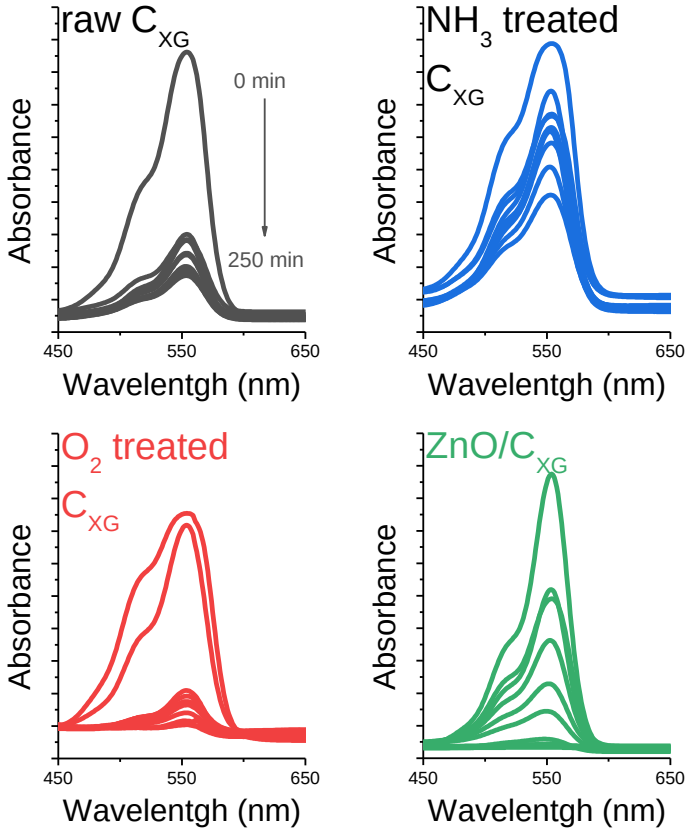
Photocatalytic properties

visible light irradiation (Ne lamps)

Rhodamine B degradation







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4. Going beyond

bimetallic

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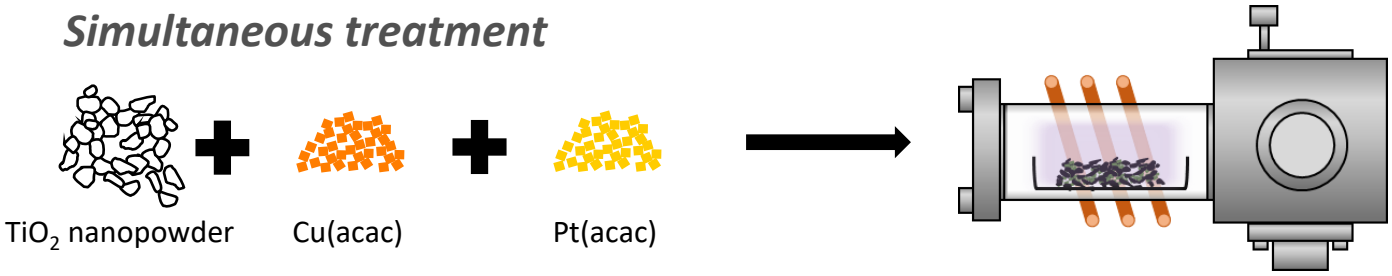
The different nanoparticles – some examples

PtCu bimetallic NPs on TiO_2 – simultaneous method



CHAVEE et al., manuscript in preparation

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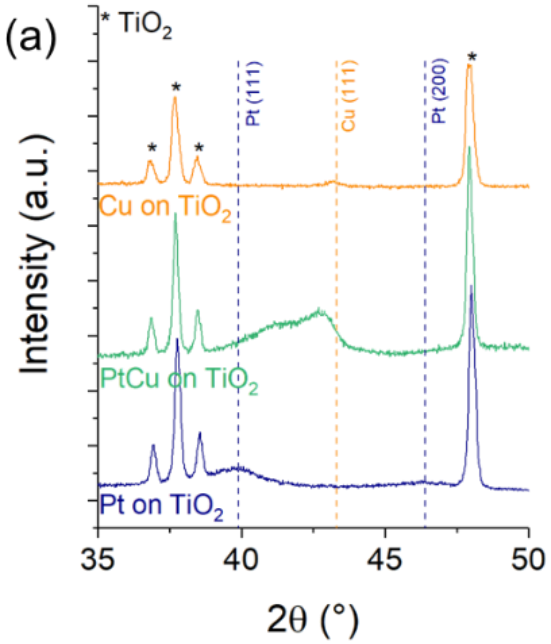

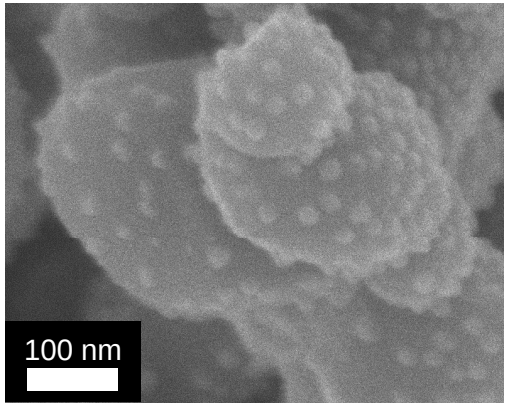

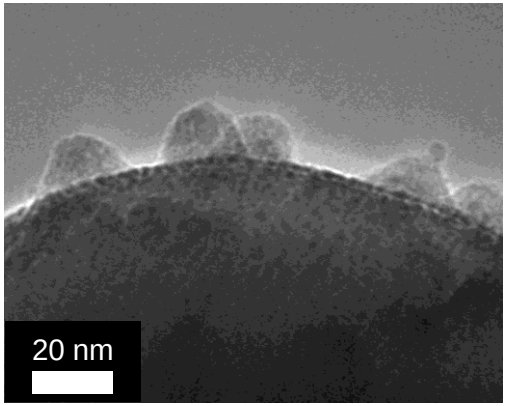

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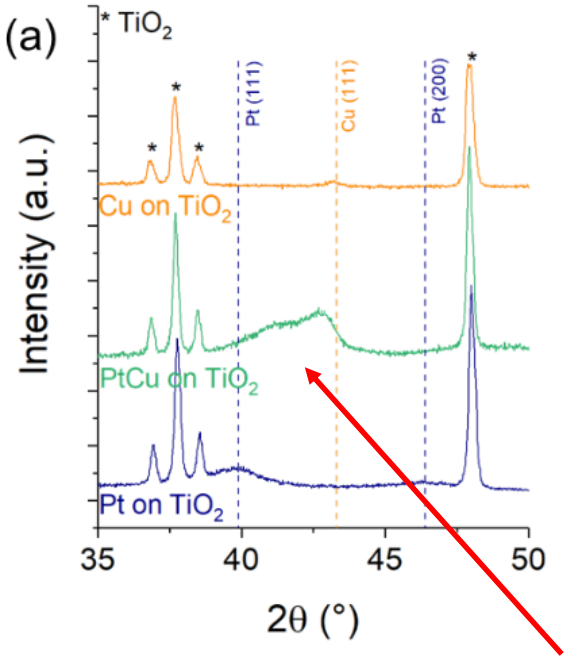
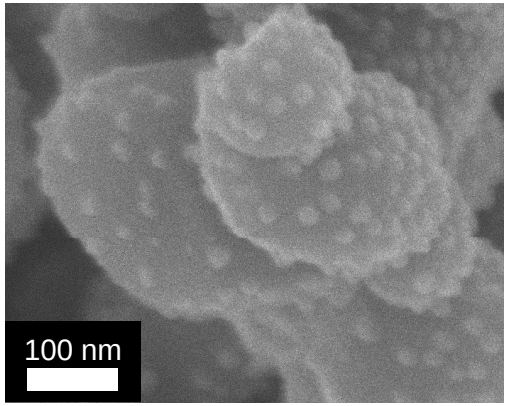
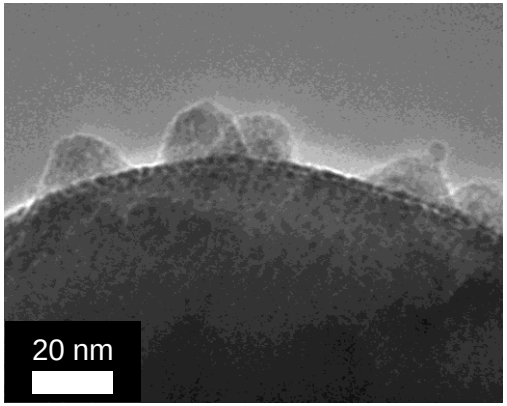
The different nanoparticles – some examples

PtCu bimetallic NPs on TiO_2 – simultaneous method







PtCu on TiO_2

Diffration peak between Cu (111) and Pt (111)
PtCu alloy formation

CHAVEE et al., manuscript in preparation

Conclusions & Perspectives

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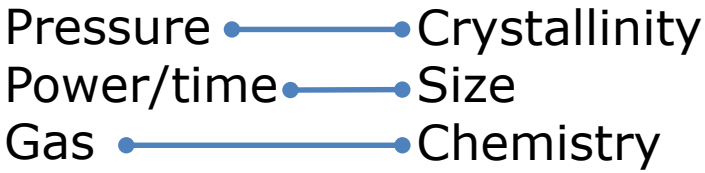
Conclusion

A versatile process to synthesis functional nanoparticles
(Pt, Pd, Rh, Cu, Fe_3N, Ni_3N, NiO, ZnO, Cu_xO_y, Al_2O_3, CoO, MnO_x, PtNi, PtCu)

The sky is the limit

Take home message:

Possibility to control the size,
the crystallinity and the chemistry of the NPs



Thank you for your attention
Question?

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