Synthesis and Characterization of Nanoparticles produced in Flames

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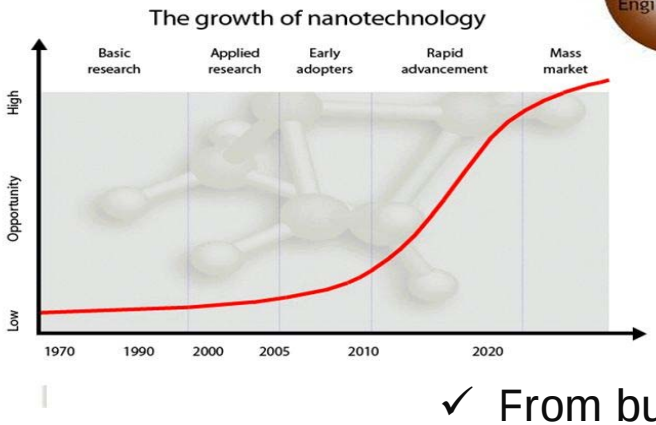
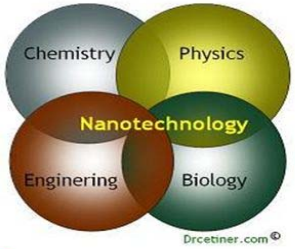
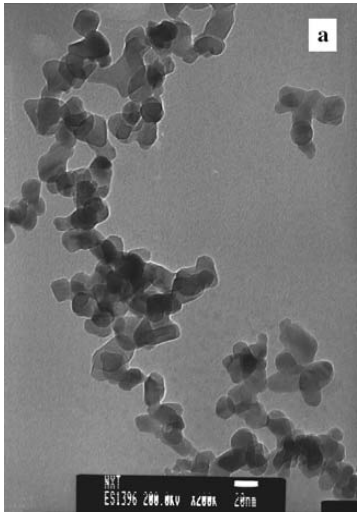
Oxide Nanoparticles

Particle aggregates with dimension in the range 2 nm – 200 nm



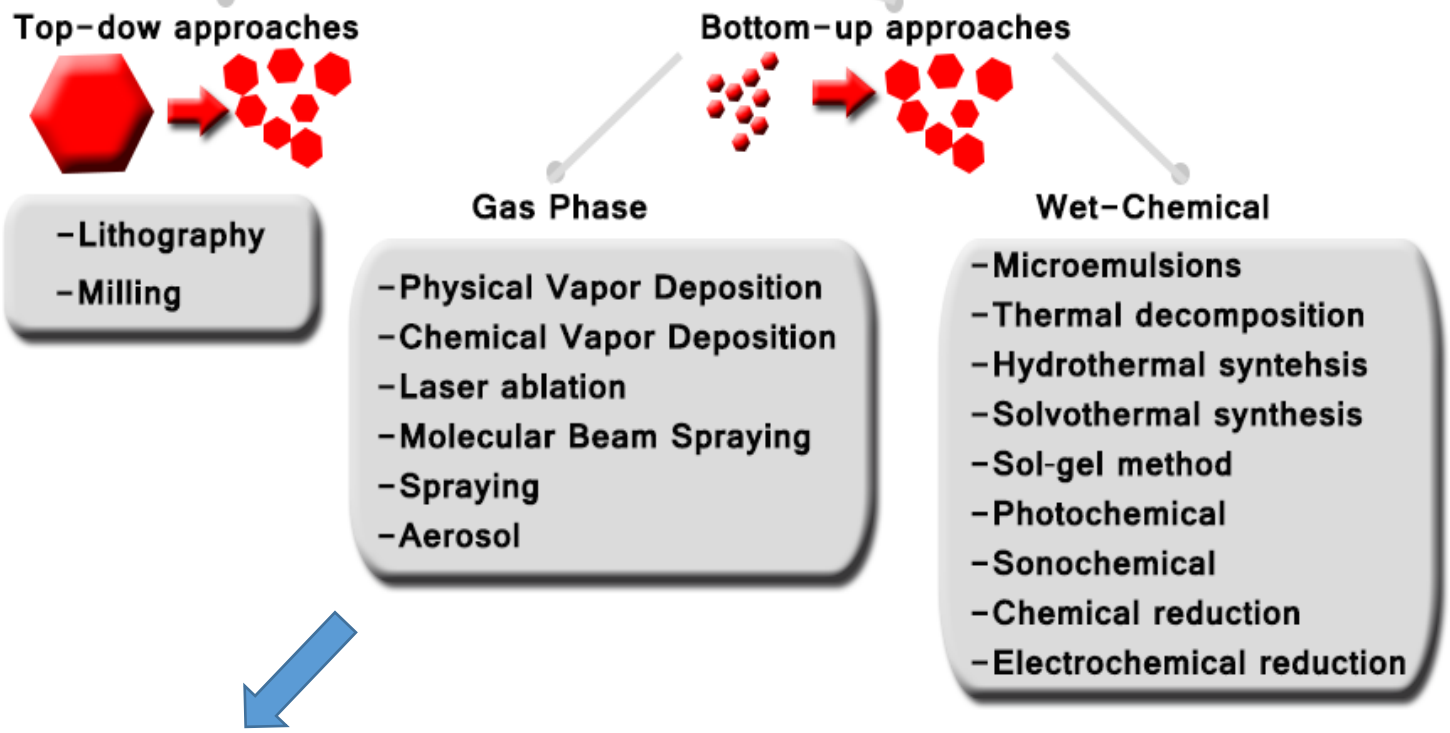




- ✓ From bulk to nanoscale, increase of surface area/volume
- ✓ Properties depend on composition, dimension, shape.

- ✓ Catalysis
- ✓ Photocatalysis
- ✓ Gas –sensing
- ✓ White pigment in paints, plastics and cement
- ✓ Electronics

Production techniques of nanomaterials

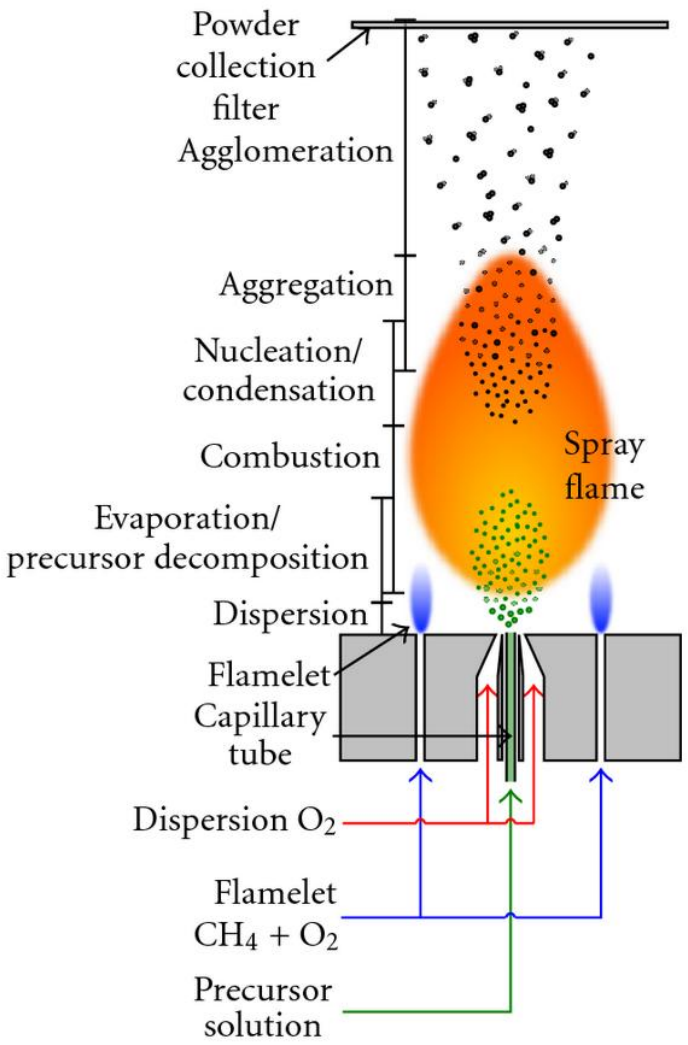


Flame Synthesis

By changing the flame/reactor conditions (fuel, oxidizer, temperature field,..)

it is possible to control **Dimension, Morphology and Phase** of the nanoparticles.

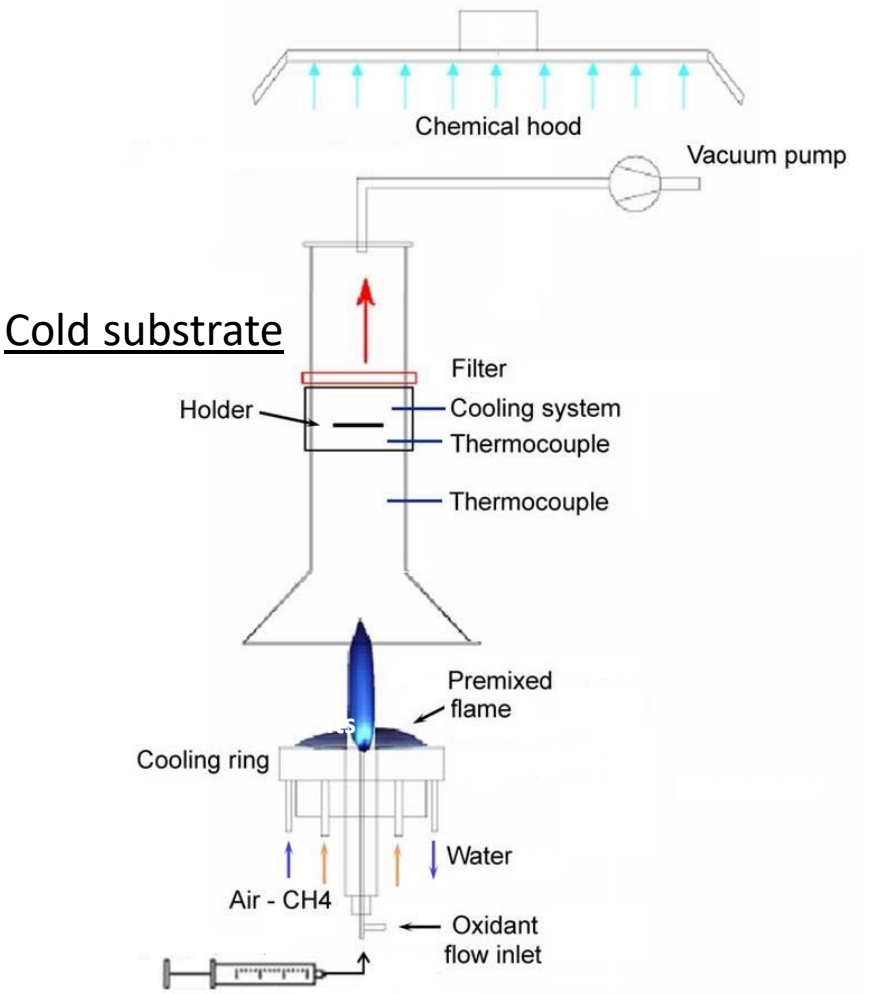
Why to use Flame Spray Pyrolysis?



- Powerful method to produce nanoparticles oxides with different properties
- Production of single- / multi-component nanoparticles (playing with precursors)
- Massive production
- Nanoparticles collected in powder (on a glass fiber filter) or deposited on a cold substrate (via thermophoresis)

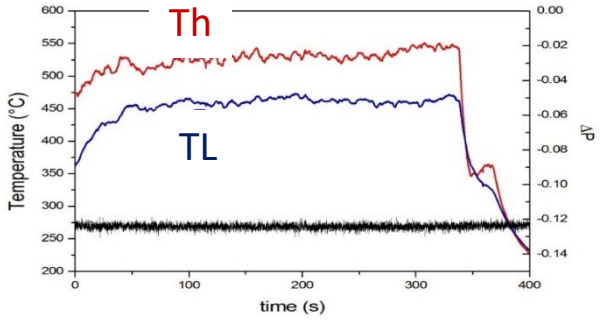
L. G. Bettini, M. V. Diamanti, M. Sansotera, M.P. Pedeferri, W. Navarrini, P. Milani, Journal of Nanoparticle Research 2016

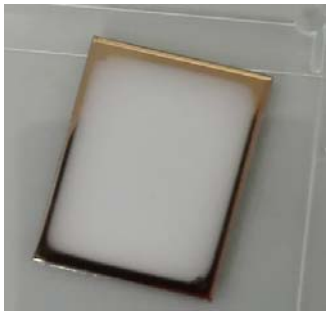
Flame Spray Pyrolysis - Experimental Set-up



On-line Synthesis monitoring

- Temperature by thermocouples
- Flow rate by pressure drop

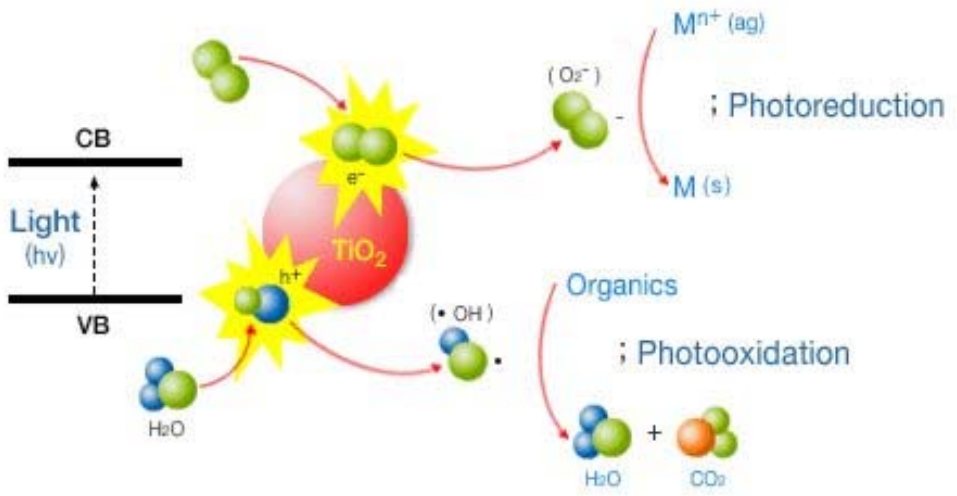




+ annealing @ 400°C

Glass fiber filter
for powder collection

Example: TiO_2 for Photocatalysis



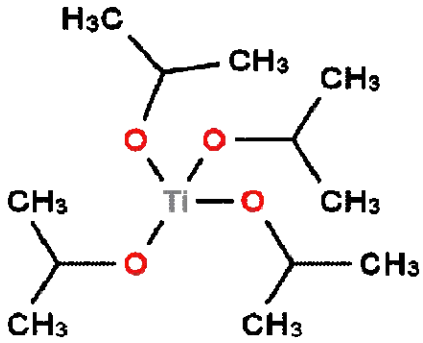
TiO_2 photoactivity depends on:

- ✓ Crystalline phase (Anatase, Rutile or mixed)
- ✓ Nanoparticle size and surface area
- ✓ Impurities or defects
- ✓ Crystallographic exposure surface

→ *Process control and material synthesis parameters*

TiO_2 Synthesis for Photocatalysis

TiO_2 (pure/with defects), + dopants



- Precursor: TTIP
- TTIP (0.5mol) in Ethanol - 4 ml/min flow rate
- 5 l/min O_2

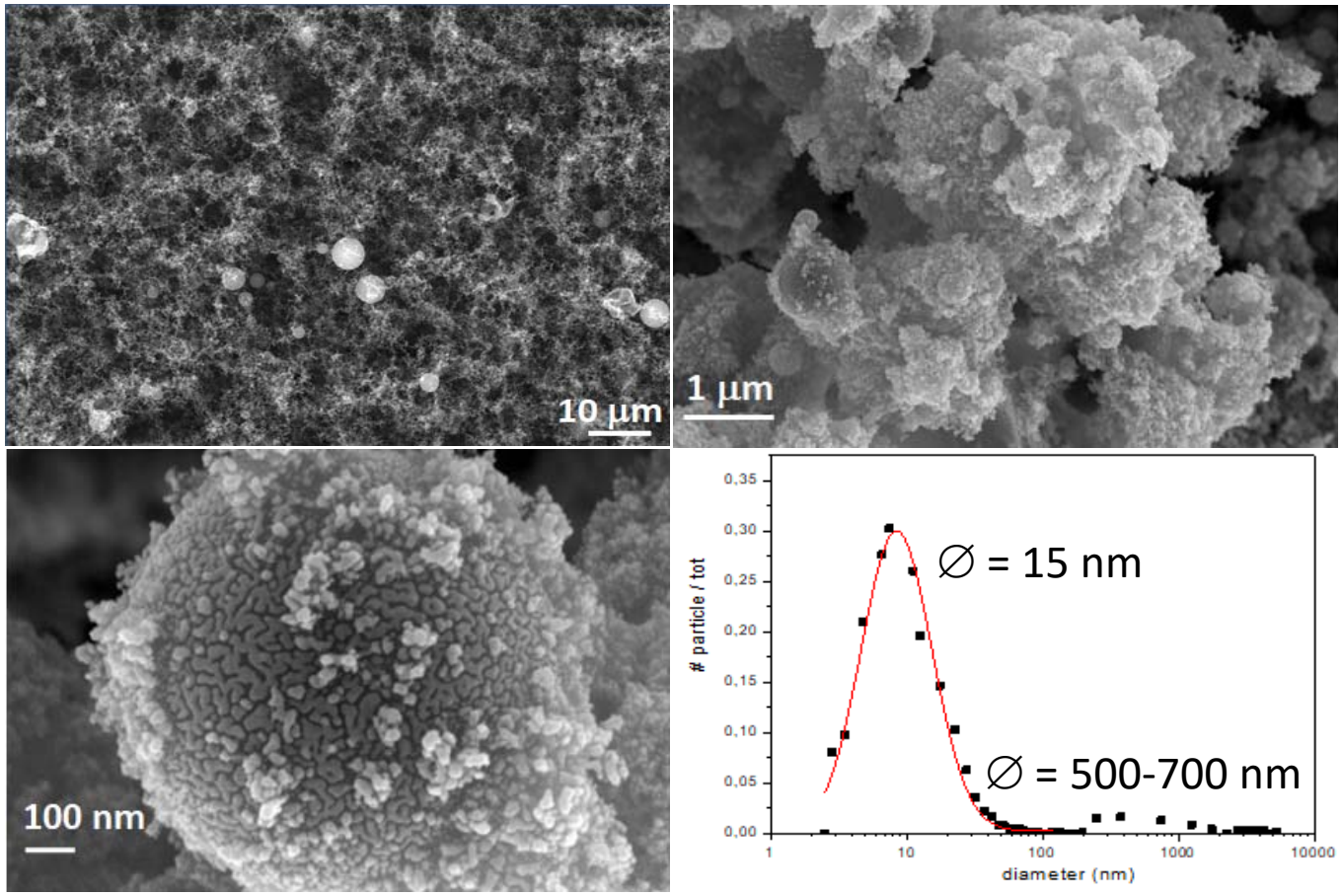
- **Anatase** and **Rutile**
- **A** more thermodinamically stable for size **< 11 nm**
- **R** more stable for size **> 35 nm**

Characterization

- FT-IR spectroscopy
- XRD analysis
- SEM /STEM analysis
- UV-vis spectroscopy
- Fluorescence spectroscopy for phase monitoring
- In-situ size monitoring (LII)

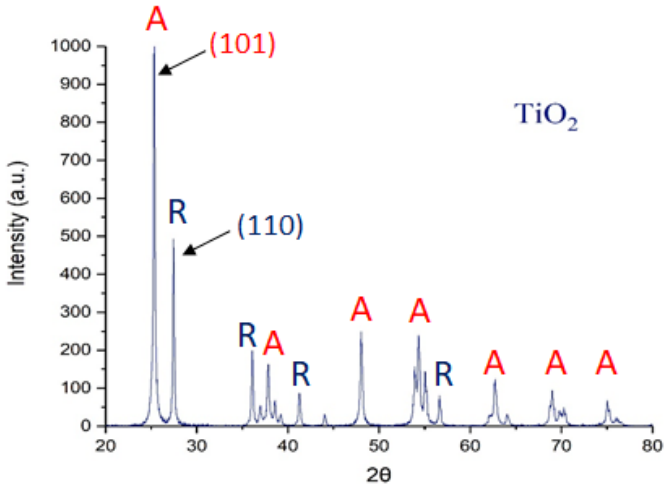
C. Bellomunno, F. Cignoli, S. De Iuliis, S. Maffi, G. Zizak (2005) Spectrochimica Acta

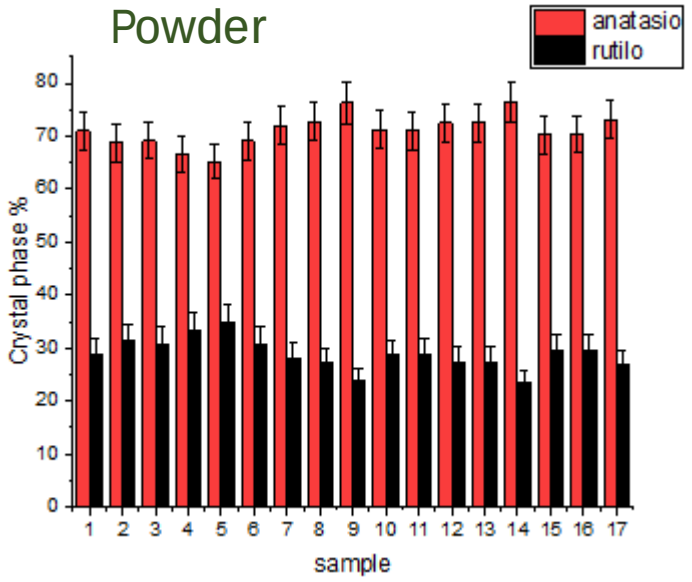
Morphological Characterization

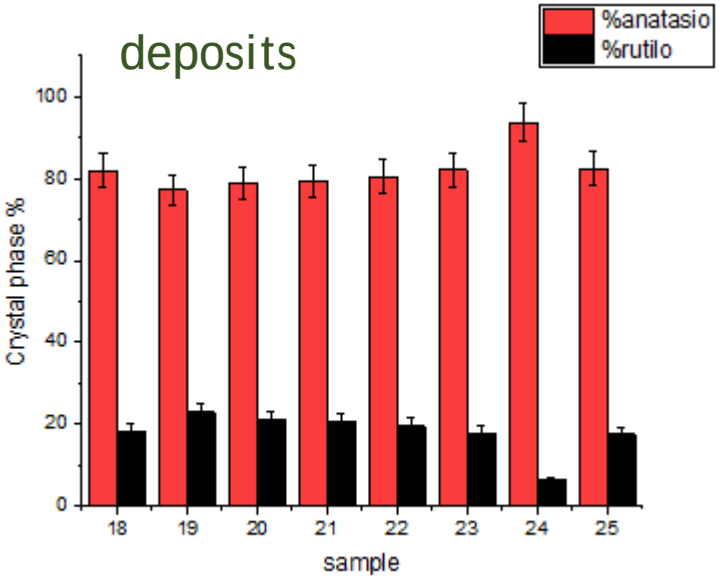


- High nanoparticle surface area
- Aggregates of particles with spherical shape

Structural Analysis: XRD



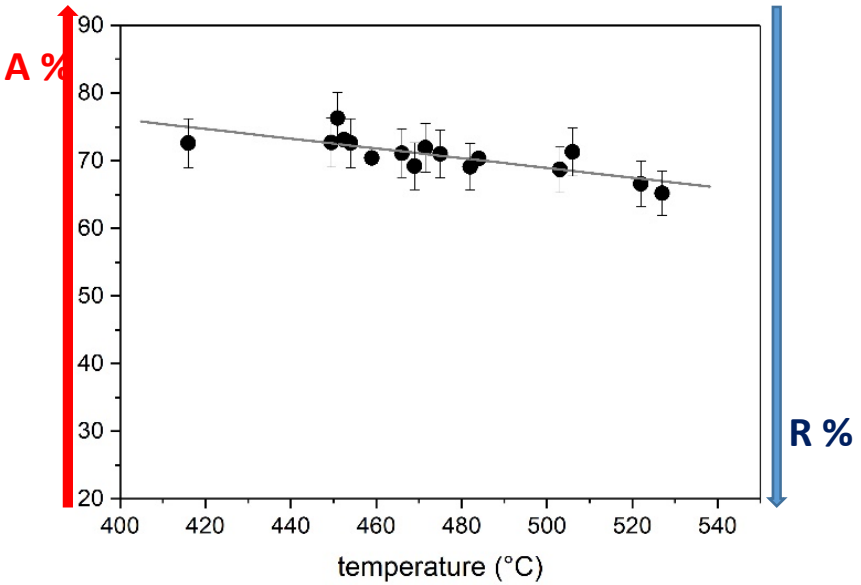


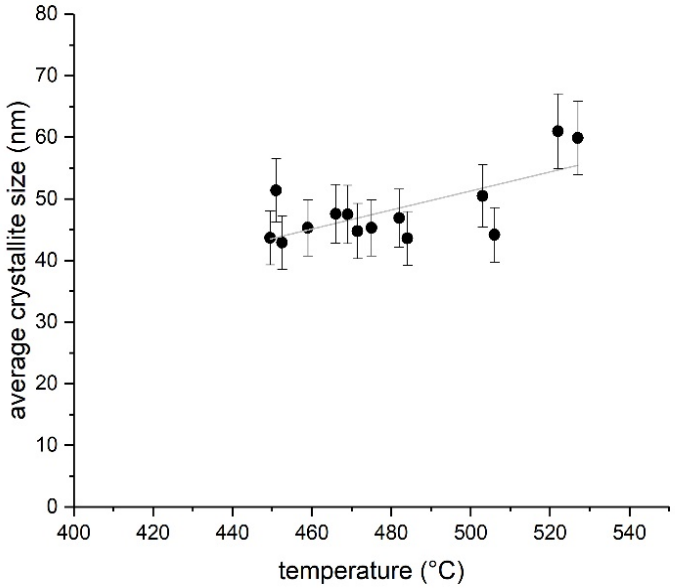


- ✓ Anatase (A) and Rutile (R)
- ✓ Phase composition:
  - Powder: A ~ 70 %, R ~ 30 %
  - Deposits: A ~ 80 %, R ~ 20 %

A/R higher for cold deposit

Crystallite size, Phase, Temperature



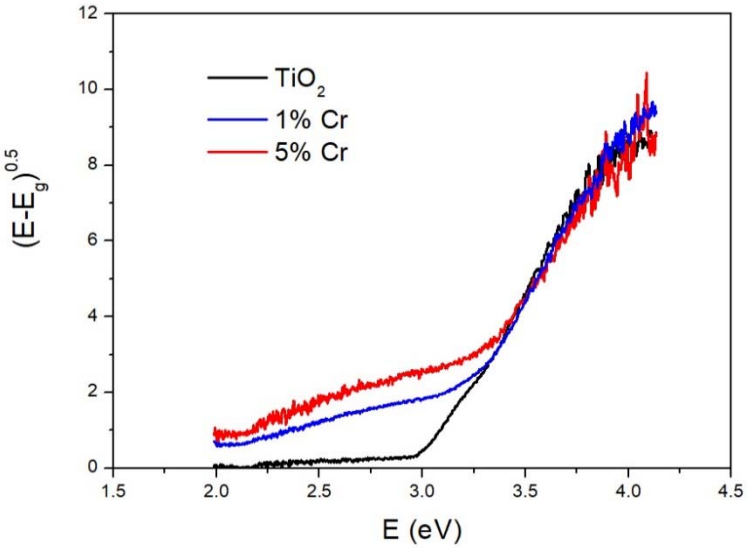


✓ **A** decreases with temperature

✓ Crystallite increases with temperature (40 nm a ~ 60 nm)

A→ R with increasing dimension

UV-vis for Energy band gap



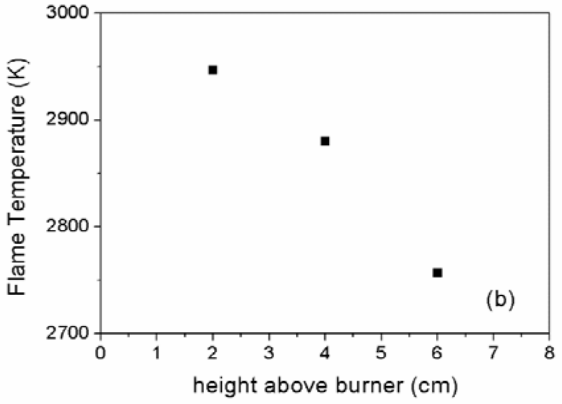
Example:

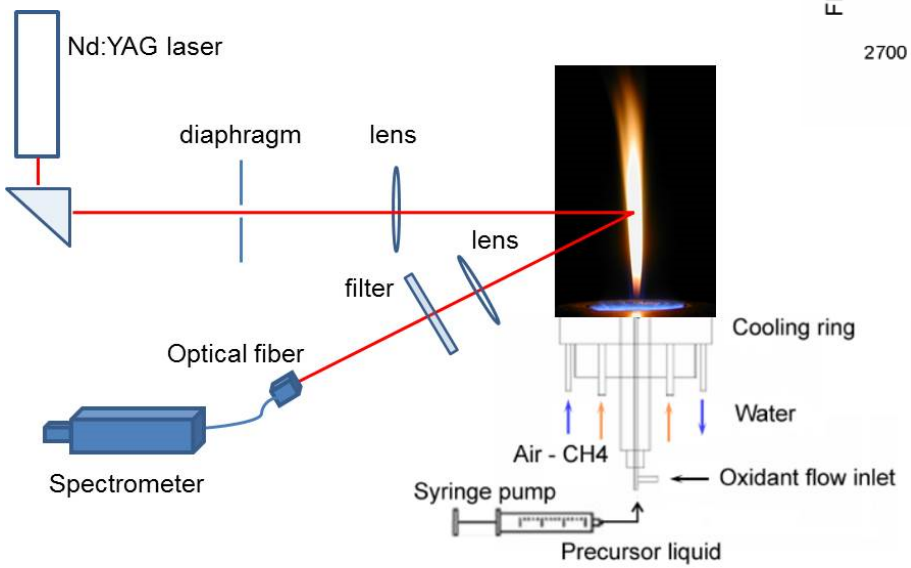
- TiO_2
- TiO_2 (0.5M) + 1%(5%) Cr

- Sample preparation: Nanoparticles collection on a quartz plate via fast flame insertion (thermophoresis)
- Band gap evaluation from Ex-situ reflectance measurements

In-situ Synthesis monitoring

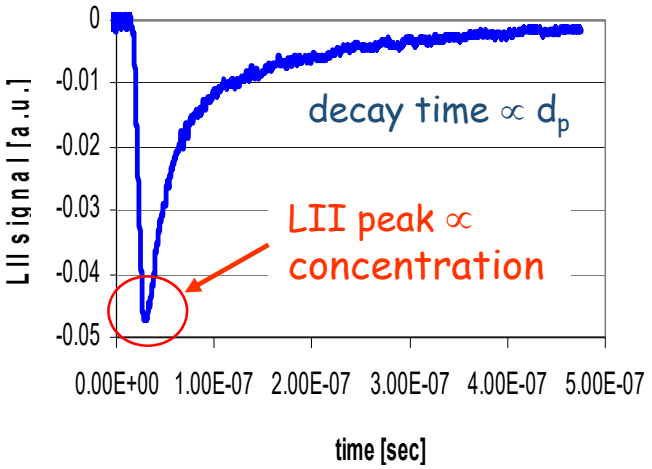
- Light emission to measure flame temperature





- Laser-Induced Incandescence for nanoparticle size

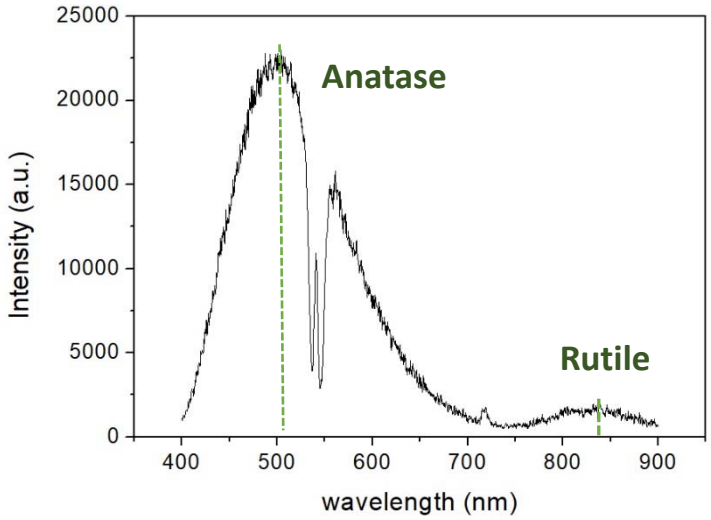
UV (266 nm) pulsed laser



Fluorescence Spectroscopy

- Nanoparticle phase analysis
- Preliminary ex-situ measurements for calibration purpose
- GOAL: on-line nanoparticle phase monitoring

Excitation wavelength: **355 nm**
TiO_2 deposit on stainless still



Acknowledgments

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Thank you for your attention!