



# Characterization of the precipitation of Oxide Dispersion Strengthened (ODS) ferritic steels reinforced by Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> pyrochlore phase

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# Characterization of the precipitation of Oxide Dispersion Strengthened (ODS) ferritic steels reinforced by $\text{Y}_2\text{Ti}_2\text{O}_7$ pyrochlore phase

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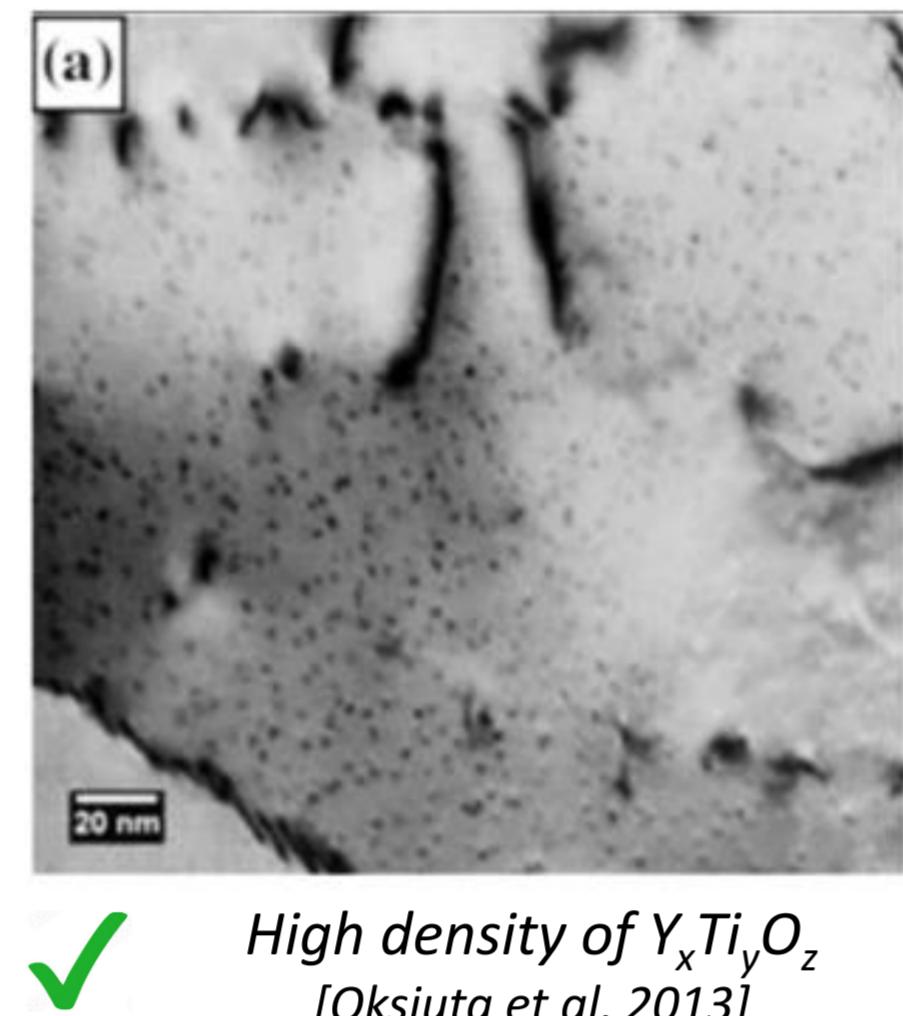
Institut des Sciences Chimiques de Rennes  
UMR CNRS 6226

PhD thesis started in Oct. 2019

## ODS ferritic steels as SFR cladding materials

- Sodium-cooled Fast Reactors (SFR) : natural resources optimization, enhanced safety, efficiency, waste reduction...

Cladding tube solicitations :  $\approx 650^\circ\text{C}$ ,  $> 150$  dpa, up to 100 MPa, 30,000 h  
⇒ new materials for fuel cladding.



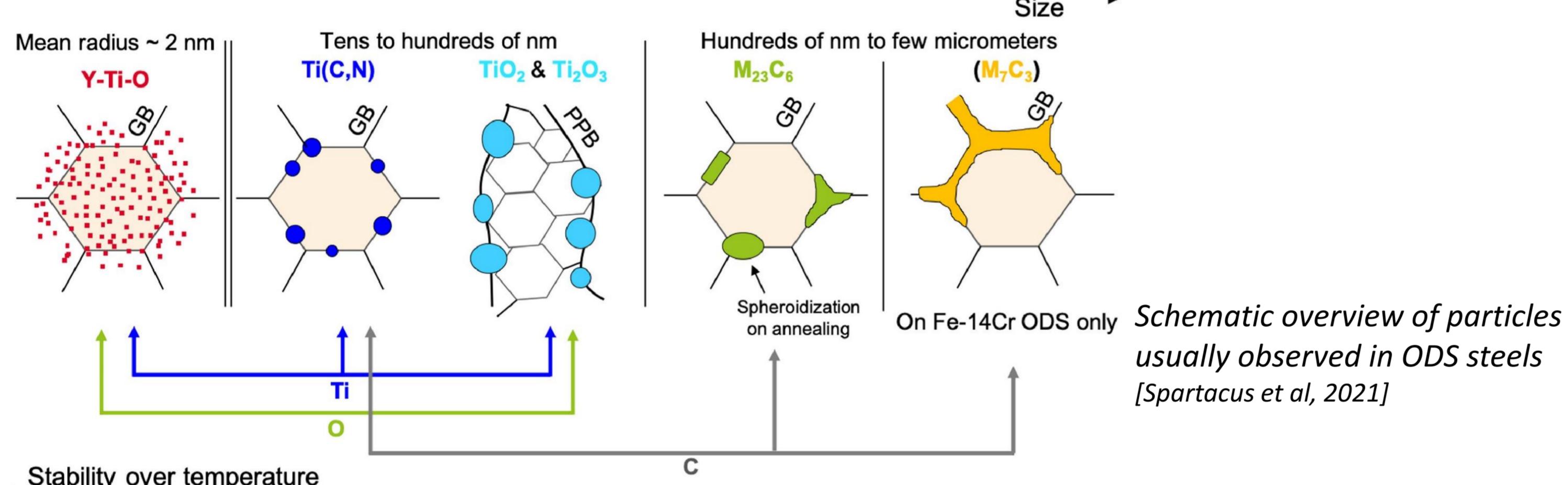
- ODS ferritic steels : high creep lifetime and stability under irradiation

- Nanometric precipitation :

- $\text{Y}_x\text{Ti}_y\text{O}_z$  stable up to  $1200^\circ\text{C}$
- Dense ( $> 10^{23}/\text{m}^3$ )
- Homogeneous (no segregation)

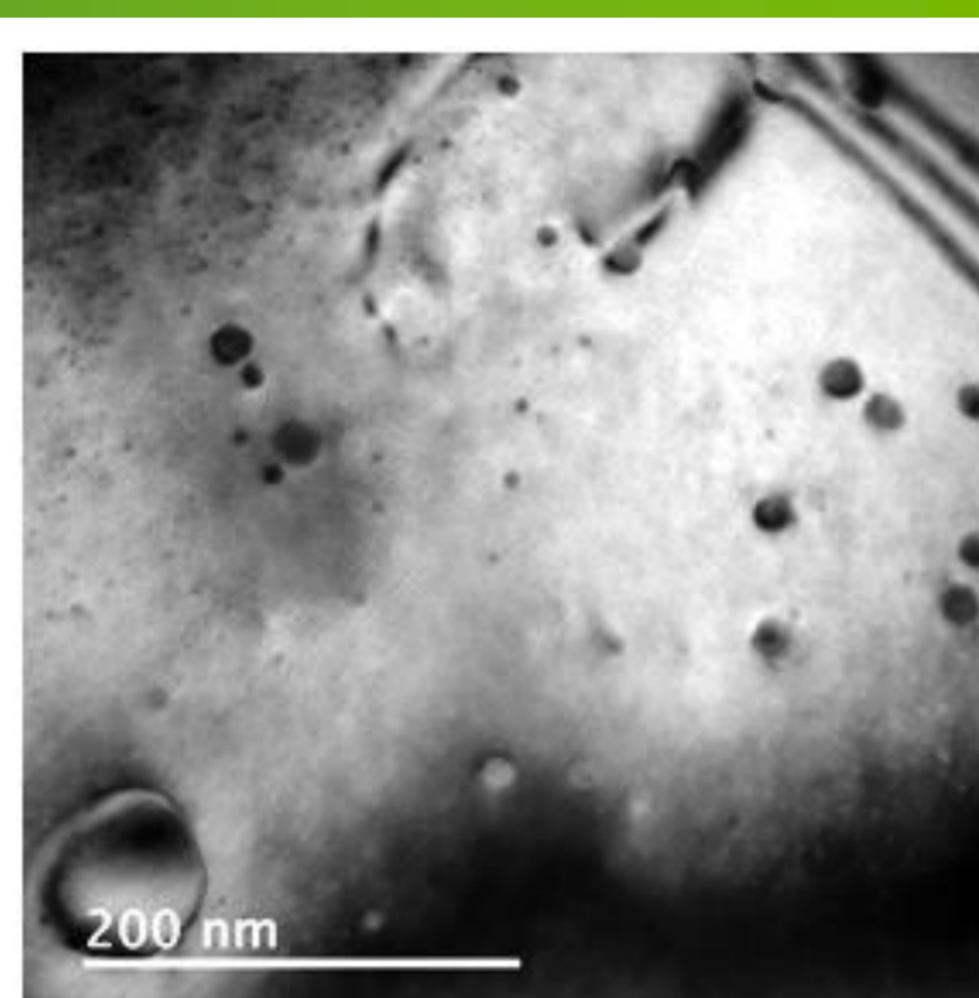
### ODS ferritic steels concerns :

- Embrittlement by secondary coarsened and segregated particles
- Low cold formability
- Anisotropic strain, high DBTT and low USE, no tertiary creep



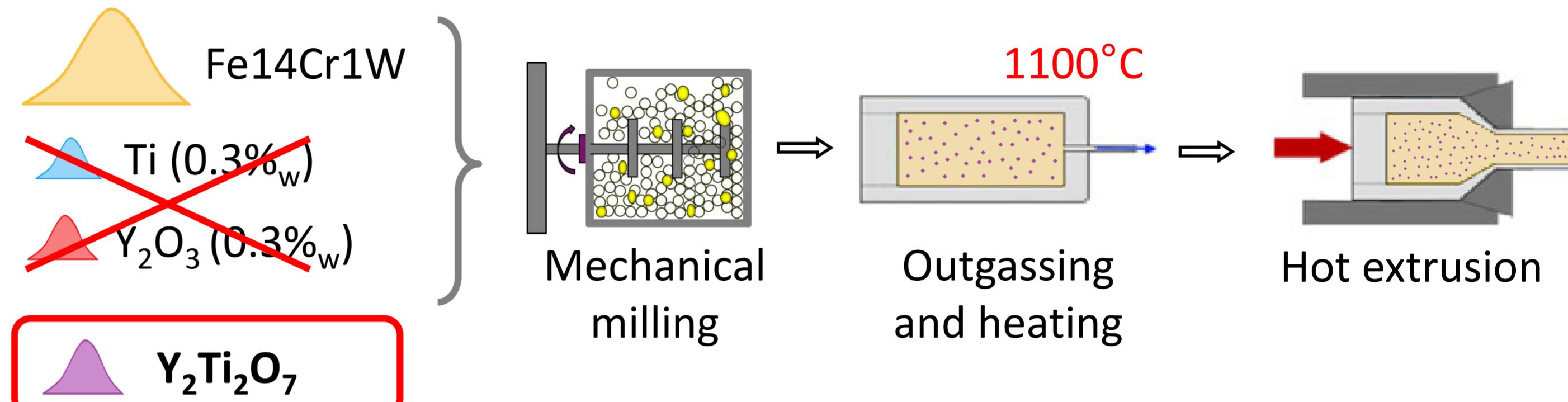
## Results

- Single type particles introduced lead to **multimodal precipitation**
- 95% of numbered particles are  $\leq 5$  nm
- Density of precipitates is « low » for ODS :  $10^{22}/\text{m}^3$  (vs  $10^{23}\text{-}10^{24}/\text{m}^3$ )
- Nature of the precipitates ?



## Approach

- Modified ODS elaboration process :

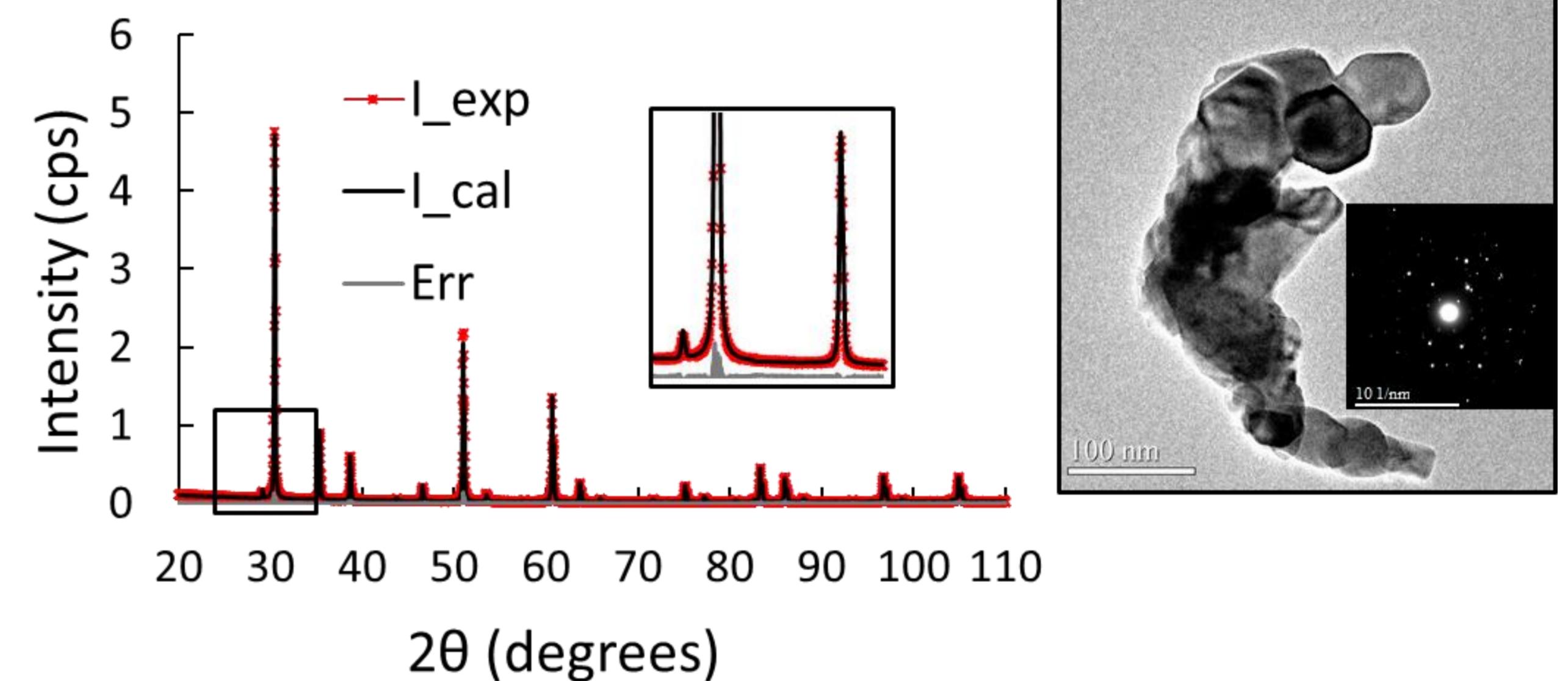


Replacement of  $(\text{Y}_2\text{O}_3 + \text{Ti})$  by  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore phase to avoid secondary phase precipitation

- Synthesis of  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore phase :

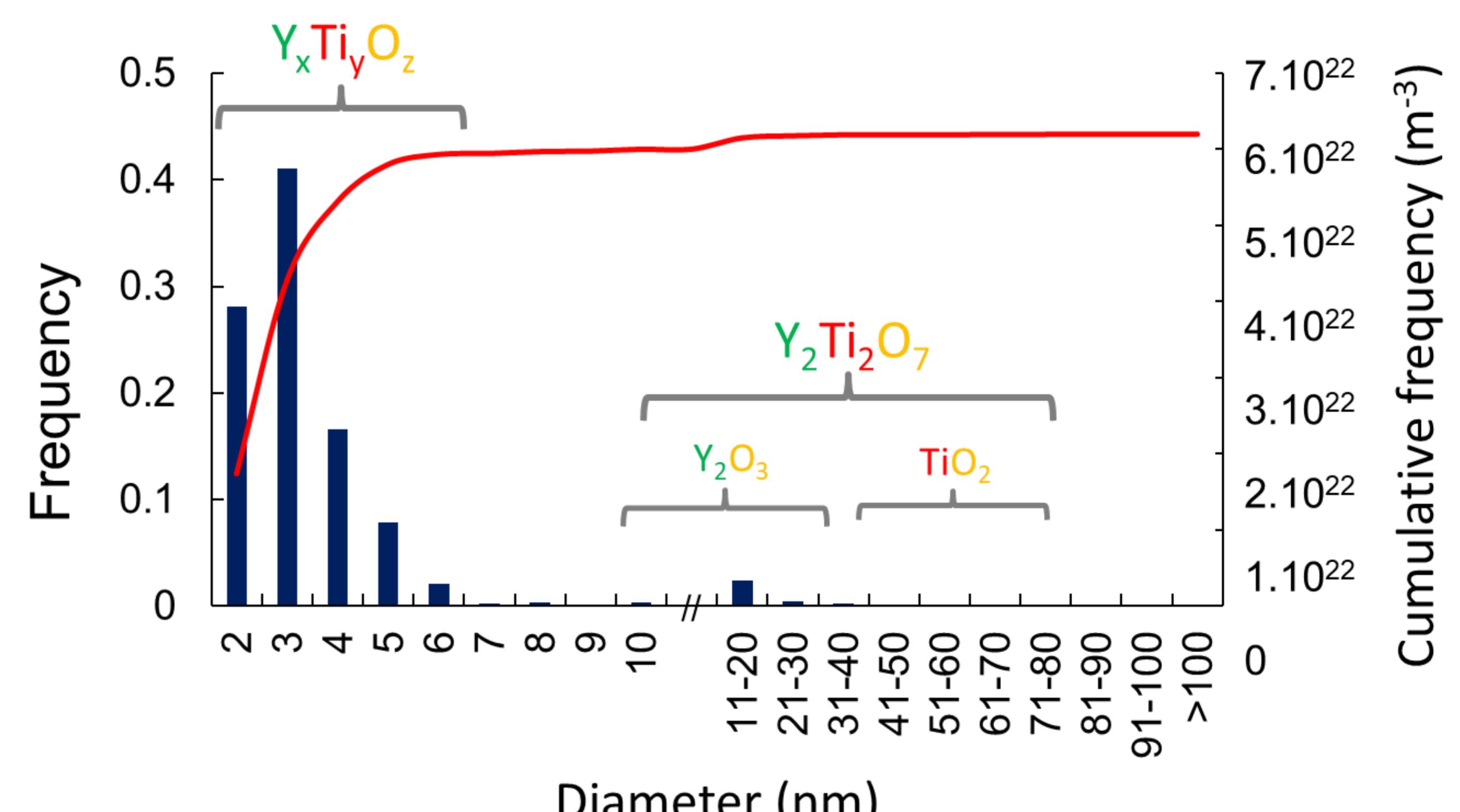
- High energy ball milling of  $\text{Y}_2\text{O}_3$  :  $2\text{TiO}_2$
- Thermal annealing under pure  $\text{O}_2$

⇒ Pure, nanocrystalline, stoichiometric  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore phase (Rietveld refinement of XRD patterns)  
⇒ No precursor left



XRD refinement and TEM (BF and associated SAED) of  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore nanostructured powders [Josserand et al, submitted]

## Summary and prospects



- Introduction of  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore phase strengthening particles leads to a multimodal precipitation :

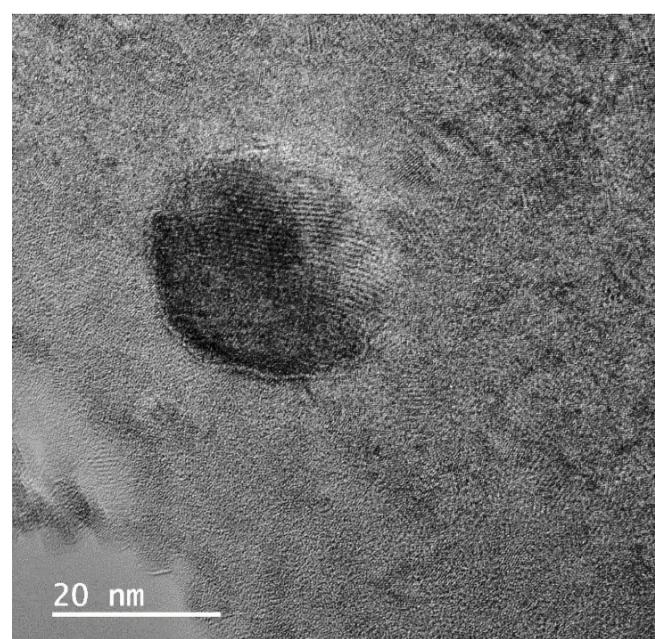
- Fragmented  $\text{Y}_2\text{Ti}_2\text{O}_7$  pyrochlore phase oxides = **new class of stable precipitates**
- Nucleated  $\text{Y}_x\text{Ti}_y\text{O}_z$  pyrochlore phase oxides
- Nucleated  $\text{Ti}-\text{O}$ ,  $\text{Y}_2\text{O}_3$

- Relationship between nature and size of oxides is established

- SAXS data acquired on SOLEIL, SWING line. Data processing should confirm particles size distribution

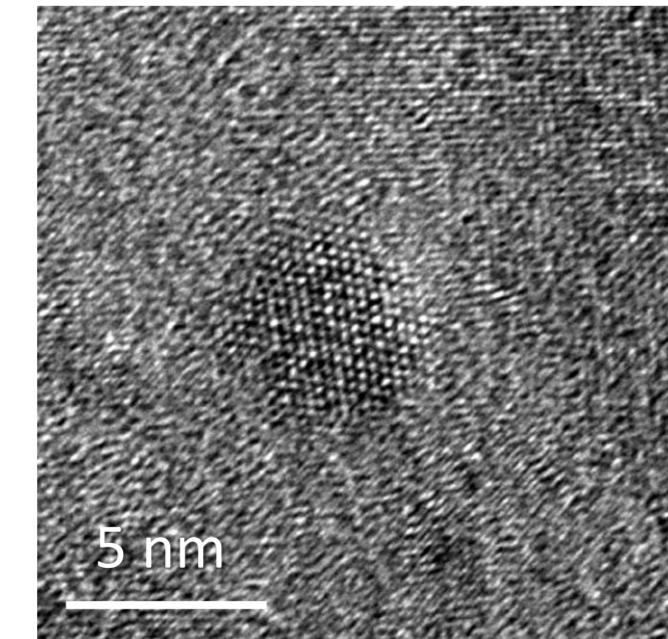
### $\text{Y}_2\text{Ti}_2\text{O}_7$ oxide

- Size : 10-80 nm
- Incoherent interfaces
- Origin : fragmentation



### $\text{Y}_x\text{Ti}_y\text{O}_z$ oxide

- Size : 2-6 nm
- Semi-coherent interfaces
- Origin : nucleation



### $\text{Y}_2\text{O}_3 / \text{TiO}_2, \text{TiO}_2$ oxides

- Size : 10-40 / 30-80 nm
- Origin : nucleation ?

