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Characterization of the precipitation of Oxide Dispersion Strengthened (ODS) ferritic steels reinforced by $Y_2Ti_2O_7$ pyrochlore phase

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ODS ferritic steels as SFR cladding materials

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

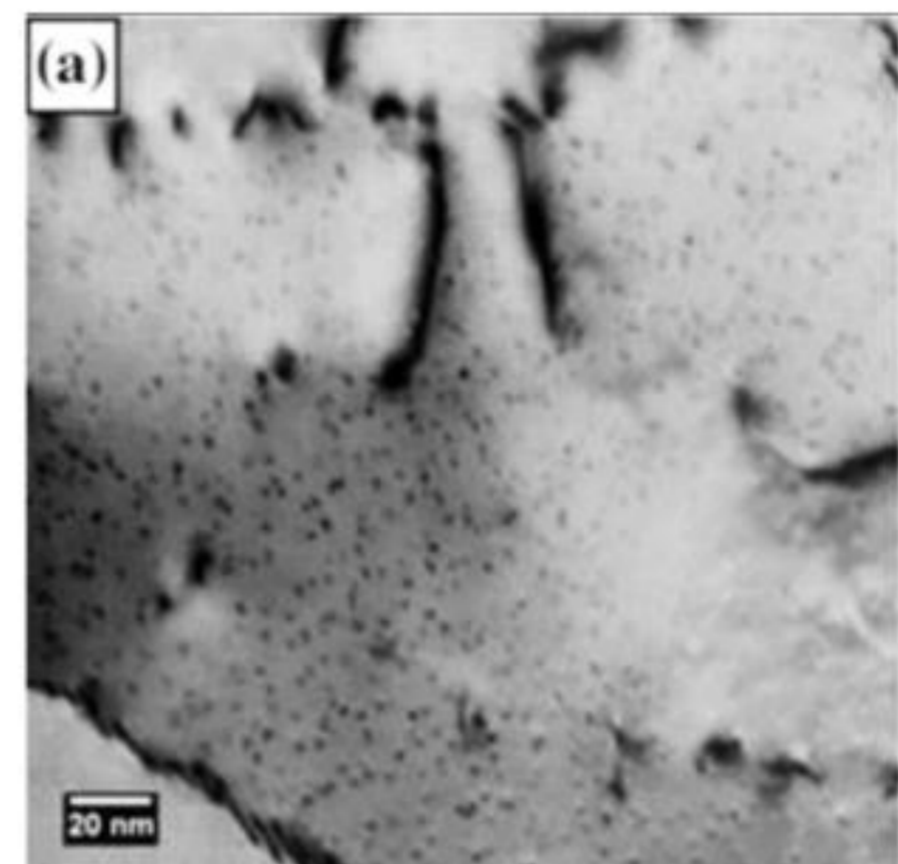
Cladding tube solicitations : $\approx 650^\circ\text{C}$, >150 dpa, up to 100 MPa, 30,000 h

⇒ **new materials for fuel cladding.**

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

- Nanometric precipitation :

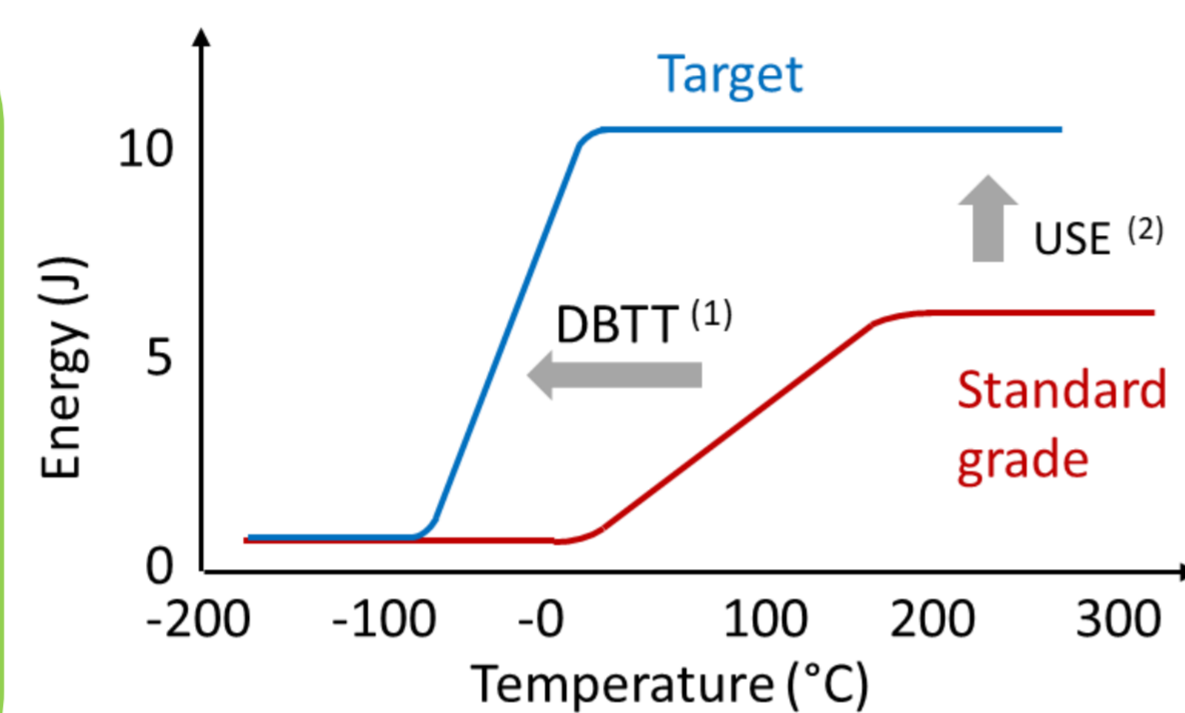
- $Y_xTi_yO_z$ stable up to 1200°C
- Dense ($N > 10^{23}/\text{m}^3$)
- Homogeneous (no segregation)



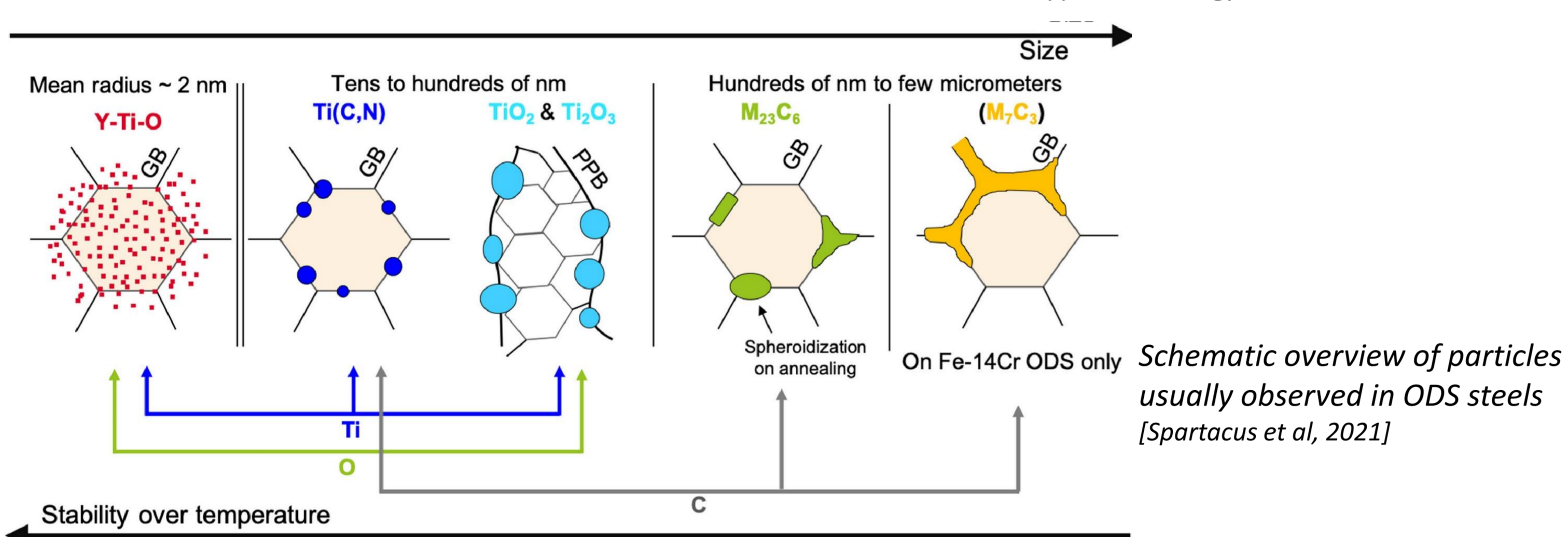
High density of $Y_xTi_yO_z$ [Oksiuta et al, 2013]

ODS ferritic steels concerns :

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

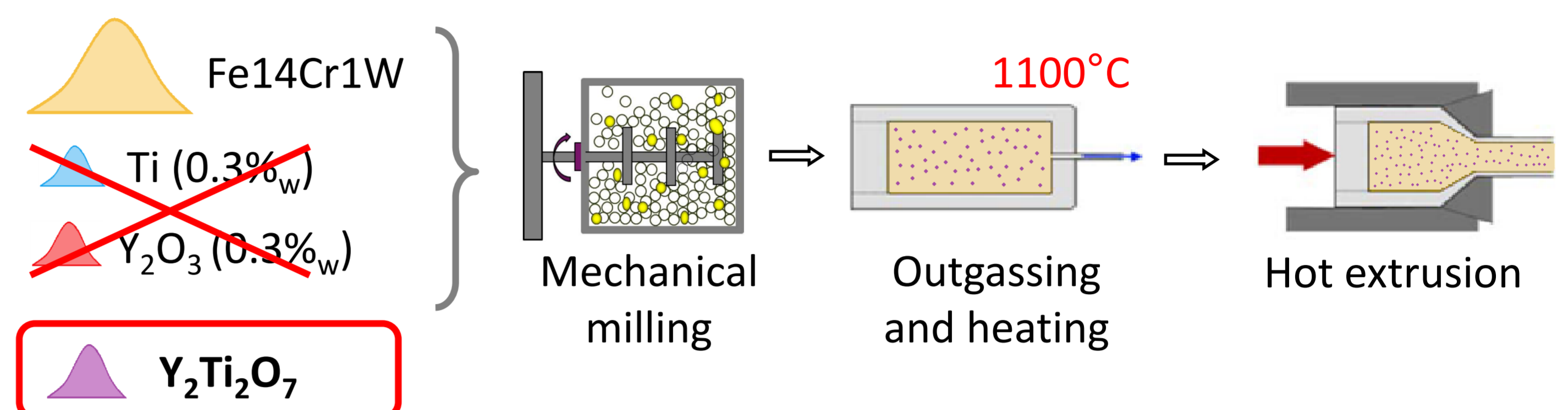


(1) Ductile-to-brittle transition temperature
(2) Upper-shelf energy



Approach

- **Modified ODS elaboration process :**



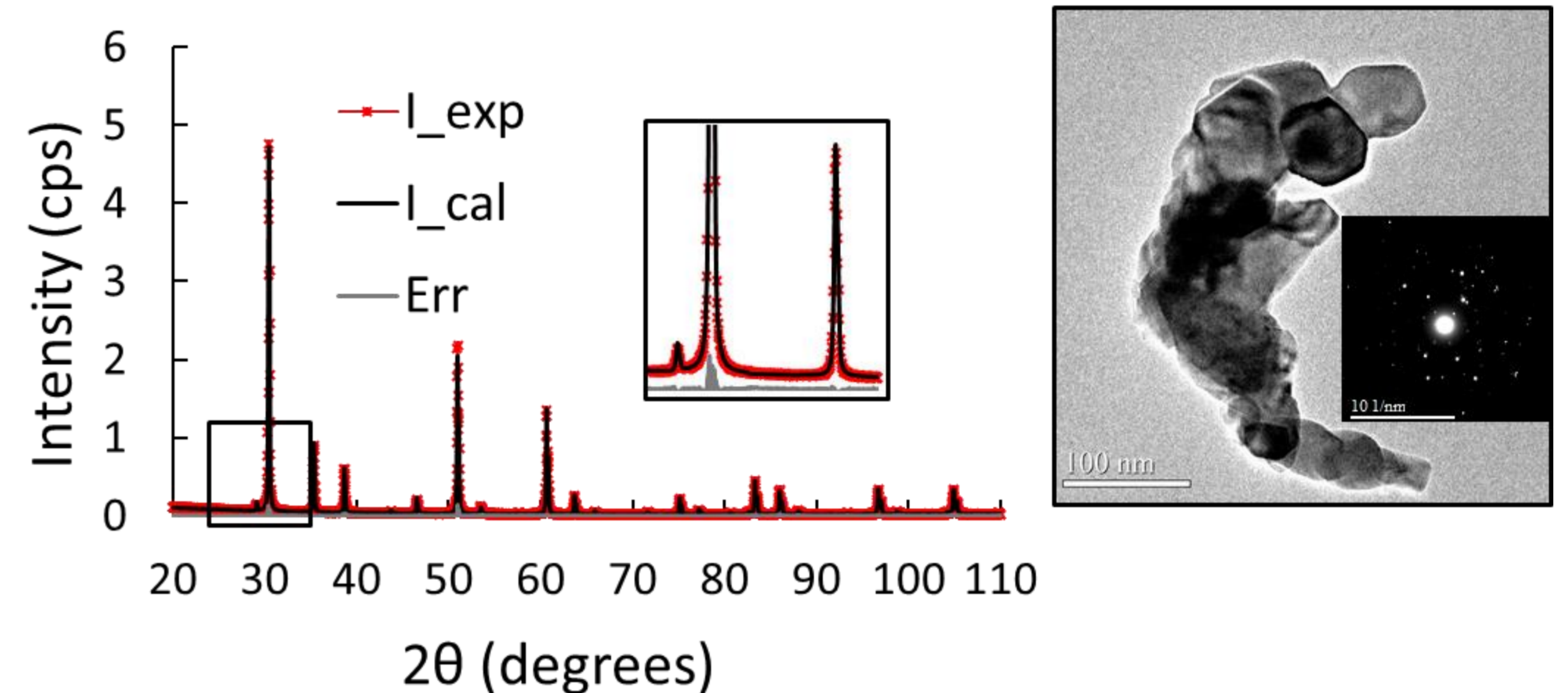
Replacement of $(Y_2O_3 + Ti)$ by $Y_2Ti_2O_7$ pyrochlore phase to avoid secondary phase precipitation

- **Synthesis of $Y_2Ti_2O_7$ pyrochlore phase :**

- High energy ball milling of $Y_2O_3 : 2TiO_2$
- Thermal annealing under pure O_2

⇒ Pure, nanocrystalline, stoichiometric $Y_2Ti_2O_7$ pyrochlore phase (Rietveld refinement of XRD patterns)

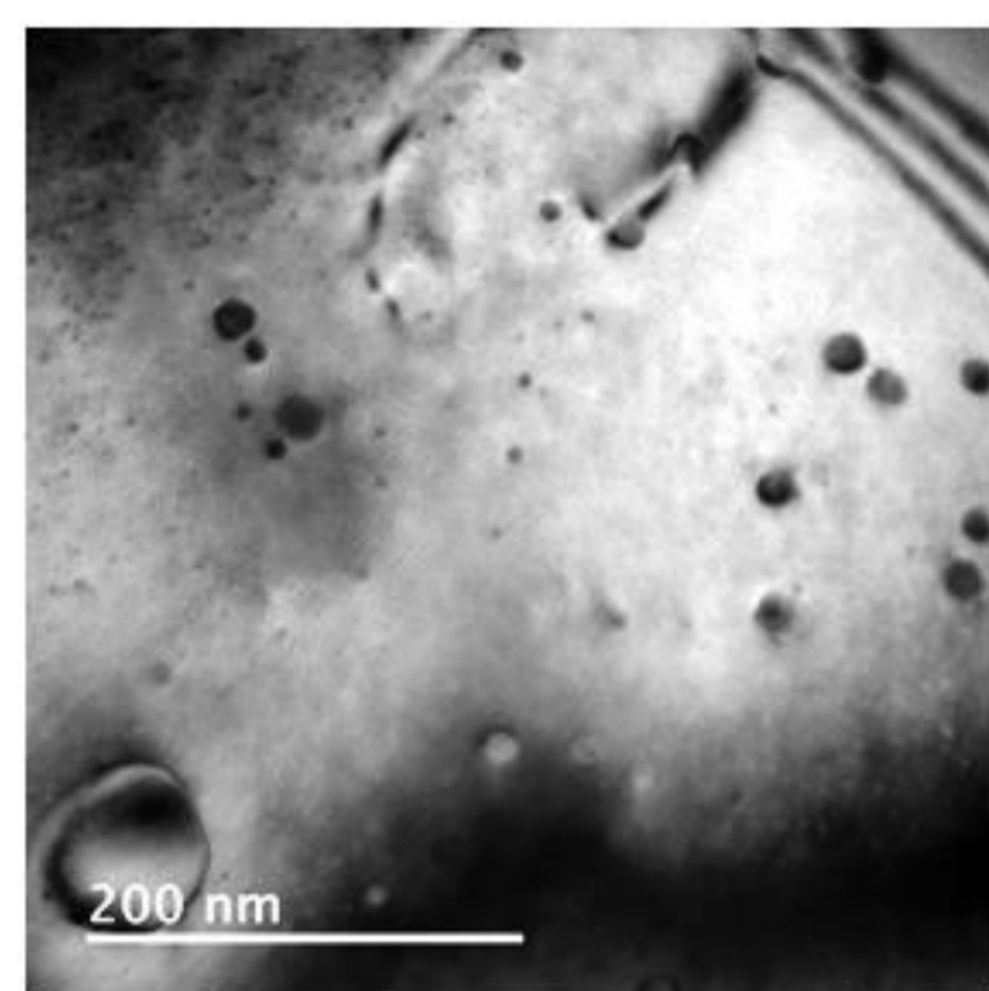
⇒ No precursor left



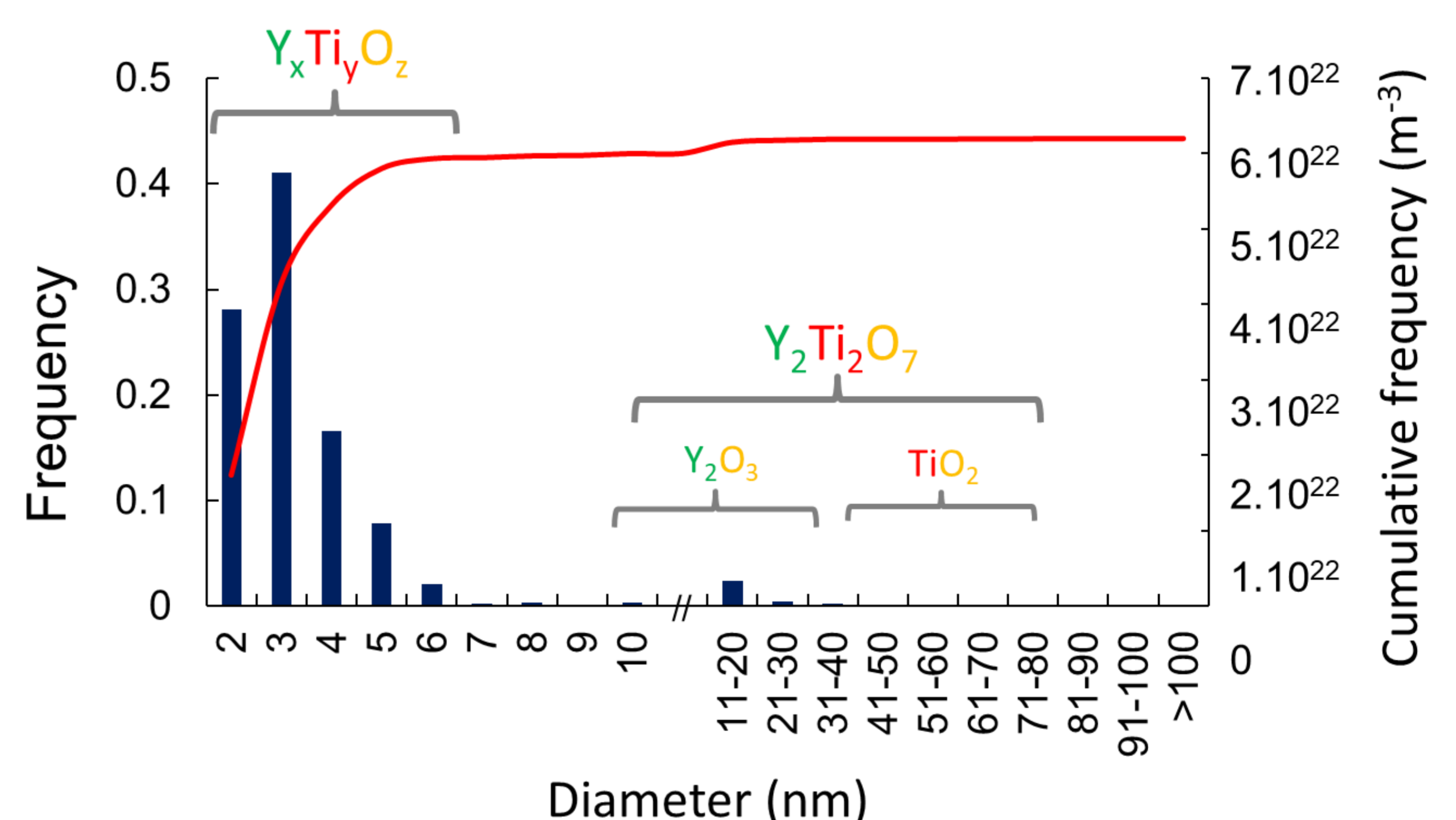
XRD refinement and TEM (BF and associated SAED) of $Y_2Ti_2O_7$ pyrochlore nanostructured powders [Josserand et al, submitted]

Results

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



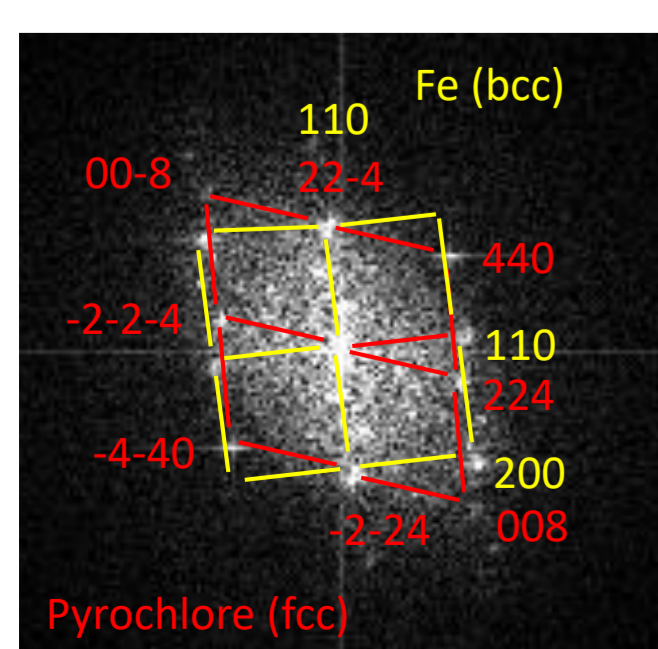
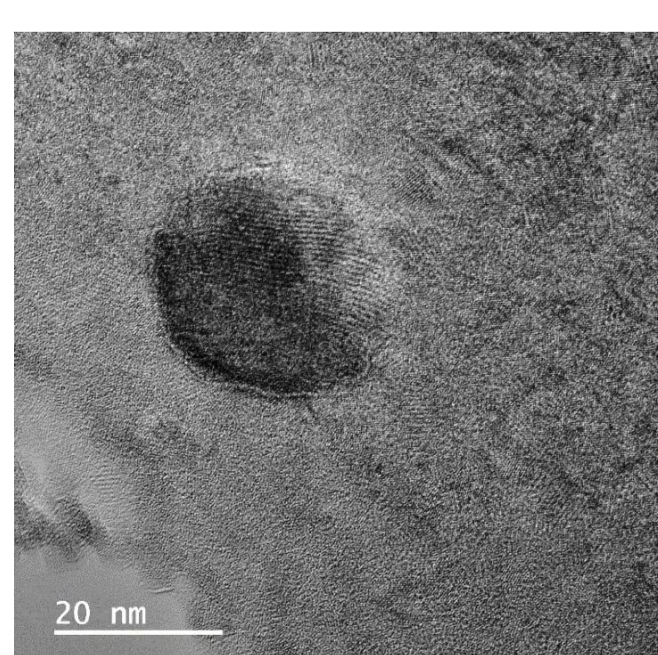
Summary and prospects



- Introduction of $Y_2Ti_2O_7$ pyrochlore phase strengthening particles leads to a multimodal precipitation :
 - Fragmented $Y_2Ti_2O_7$ pyrochlore phase oxides = **new class of stable precipitates**
 - Nucleated $Y_xTi_yO_z$ pyrochlore phase oxides
 - Nucleated Ti-O, Y_2O_3
- Relationship between nature and size of oxides is established
- SAXS data acquired on SOLEIL, SWING line. Data processing should confirm particles size distribution

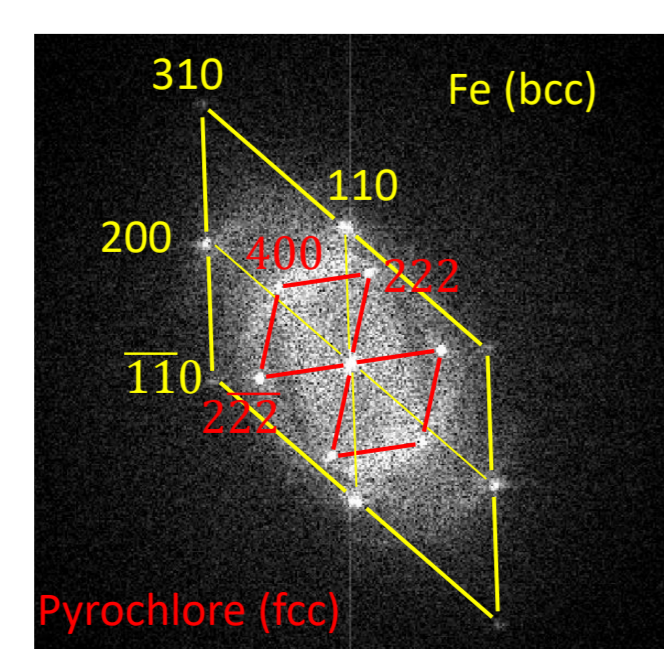
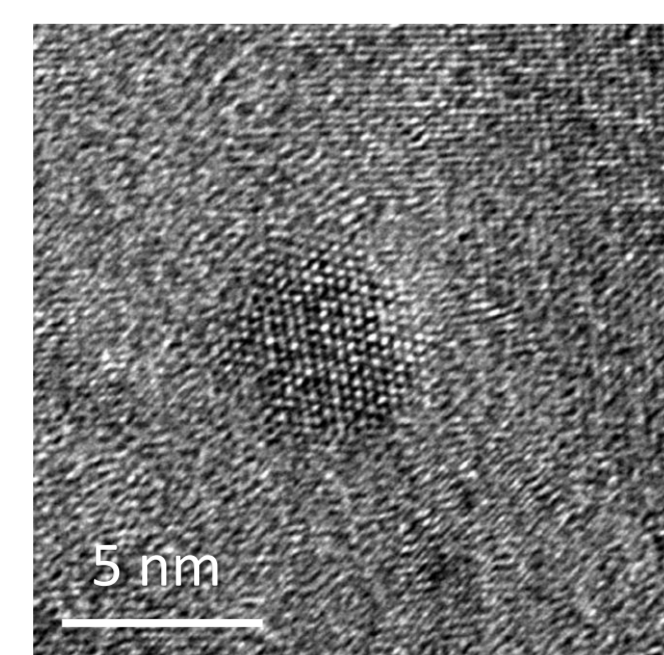
$Y_2Ti_2O_7$ oxide

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



$Y_xTi_yO_z$ oxide

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



$Y_2O_3 / TiO, TiO_2$ oxides

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

