Characterization of the precipitation of Oxide Dispersion Strengthened (ODS) ferritic steels reinforced by Y_2 Ti_2O_7 pyrochlore phase

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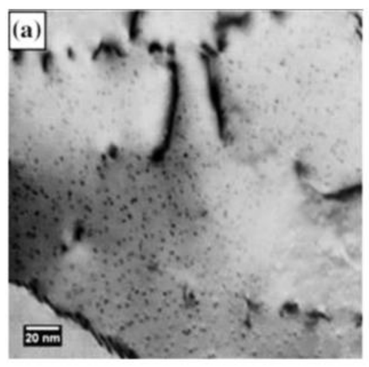
ODS ferritic steels as SFR cladding materials

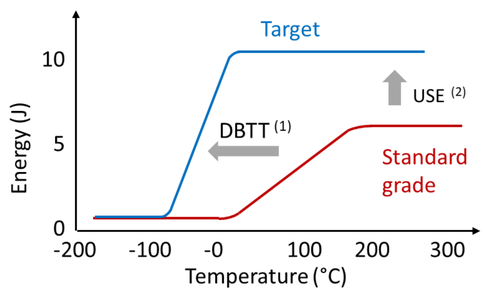
- **Sodium-cooled Fast Reactors (SFR)** : natural resources optimization, enhanced safety, efficiency, waste reduction…
  Cladding tube solicitations : ≈650°C, >150 dpa, up to 100 MPa, 30,000 h
  ⇨ **new materials for fuel cladding.**
- **ODS ferritic steels : high creep lifetime** and stability under irradiation
- Nanometric precipitation :
  - $Y_xTi_yO_z$ stable up to 1200°C
  - **Dense** ($N>10^{23}/m^3$)
  - **Homogeneous** (no segregation)

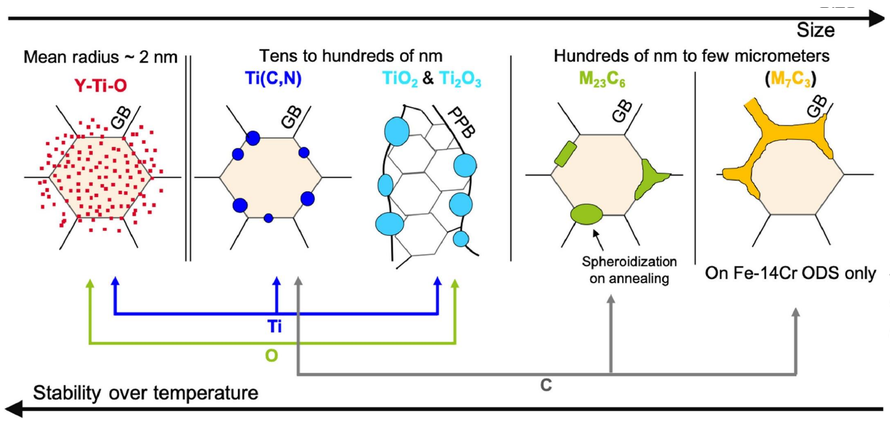


High density of $Y_xTi_yO_z$ [Oksiuta et al, 2013]

ODS ferritic steels concerns :

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

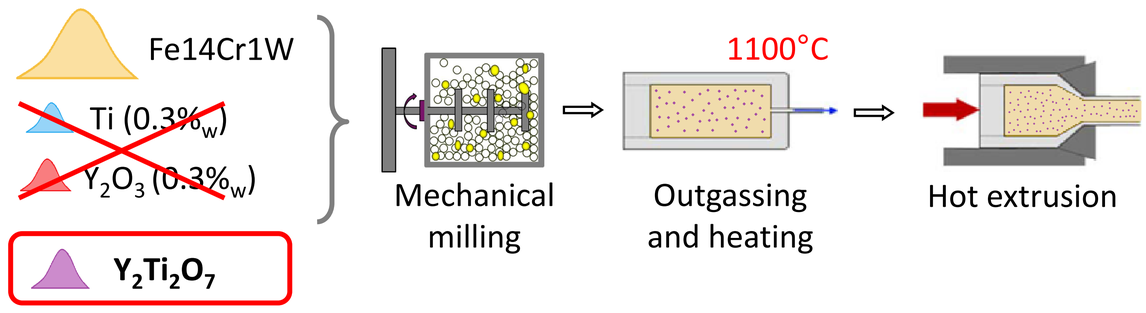




Schematic overview of particles usually observed in ODS steels [Spartacus et al, 2021]

Approach

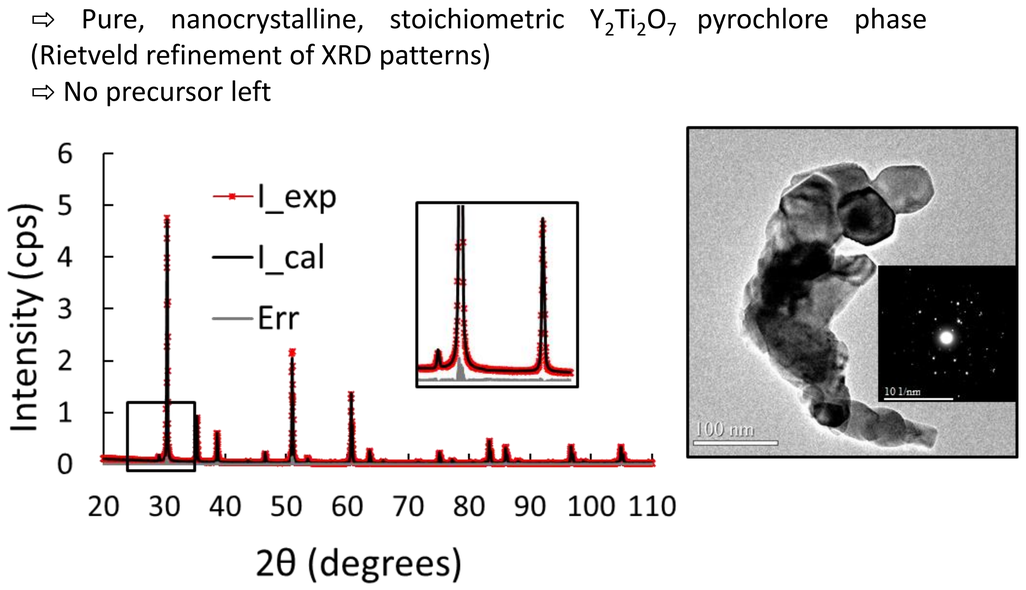
- **Modified ODS elaboration process** :



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

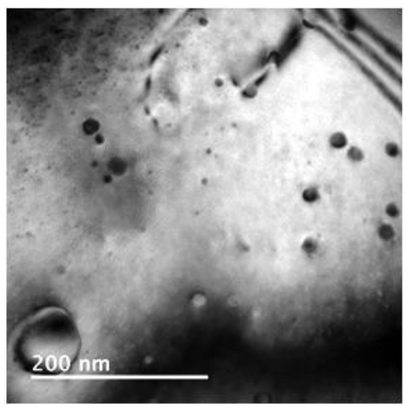
⇨ Pure, nanocrystalline, stoichiometric $Y_2Ti_2O_7$ pyrochlore phase (Rietveld refinement of XRD patterns)
⇨ No precursor left



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

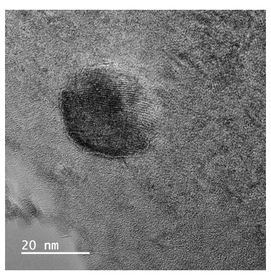
Results

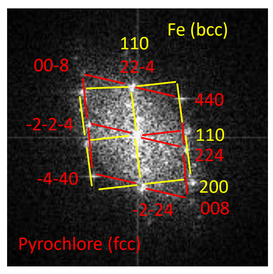
- Single type particles introduced lead to **multimodal precipitation**
- 95% of numbered particles are ≤ 5 nm
- Density of precipitates is « low » for ODS : $10^{22}/m^3$ (vs 10^{23}-$10^{24}/m^3$)
- Nature of the precipitates ?



$Y_2Ti_2O_7$ oxide

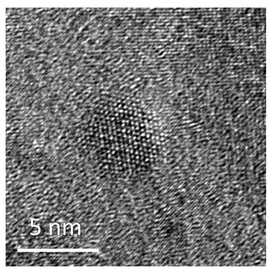
- Size : 10-80 nm
- Incoherent interfaces
- Origin : fragmentation

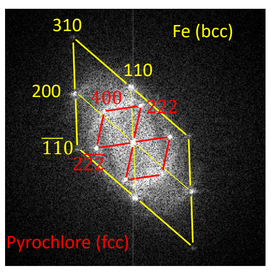




$Y_xTi_yO_z$ oxide

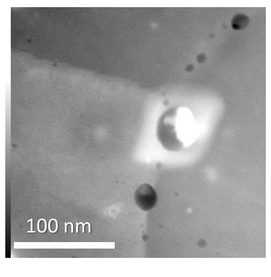
- Size : 2-6 nm
- Semi-coherent interfaces
- Origin : nucleation

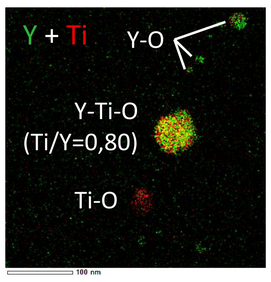




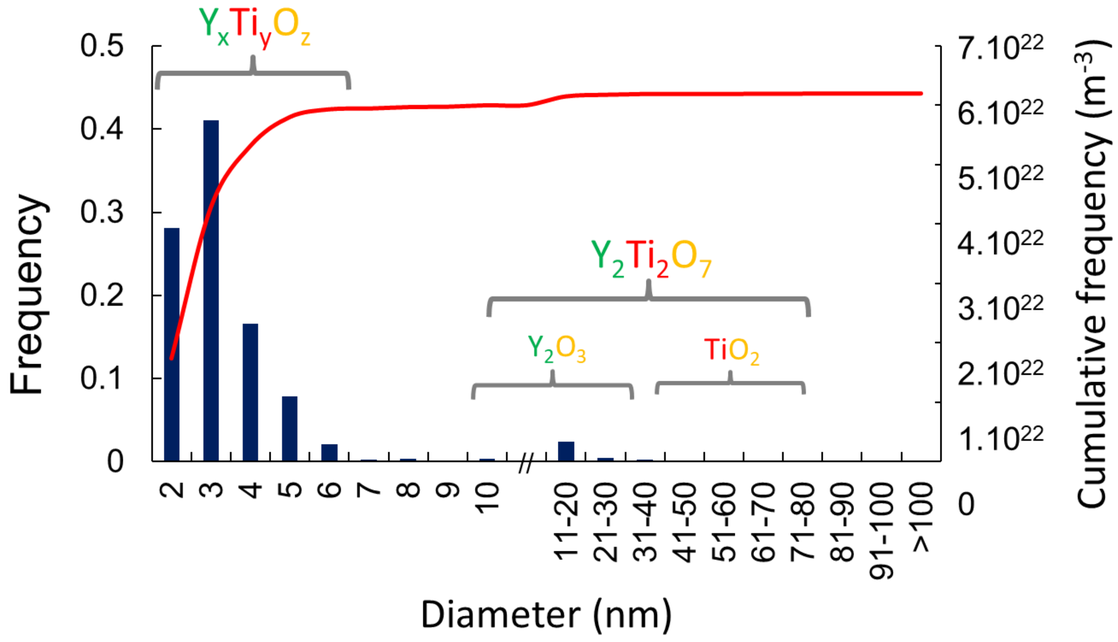
Y_2O_3 / TiO, TiO_2 oxides

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





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