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DE LA RECHERCHE À L'INDUSTRIE



INTERFACE STABILITY UNDER IRRADIATION IN OXIDE DISPERSION STRENGTHENED STEELS

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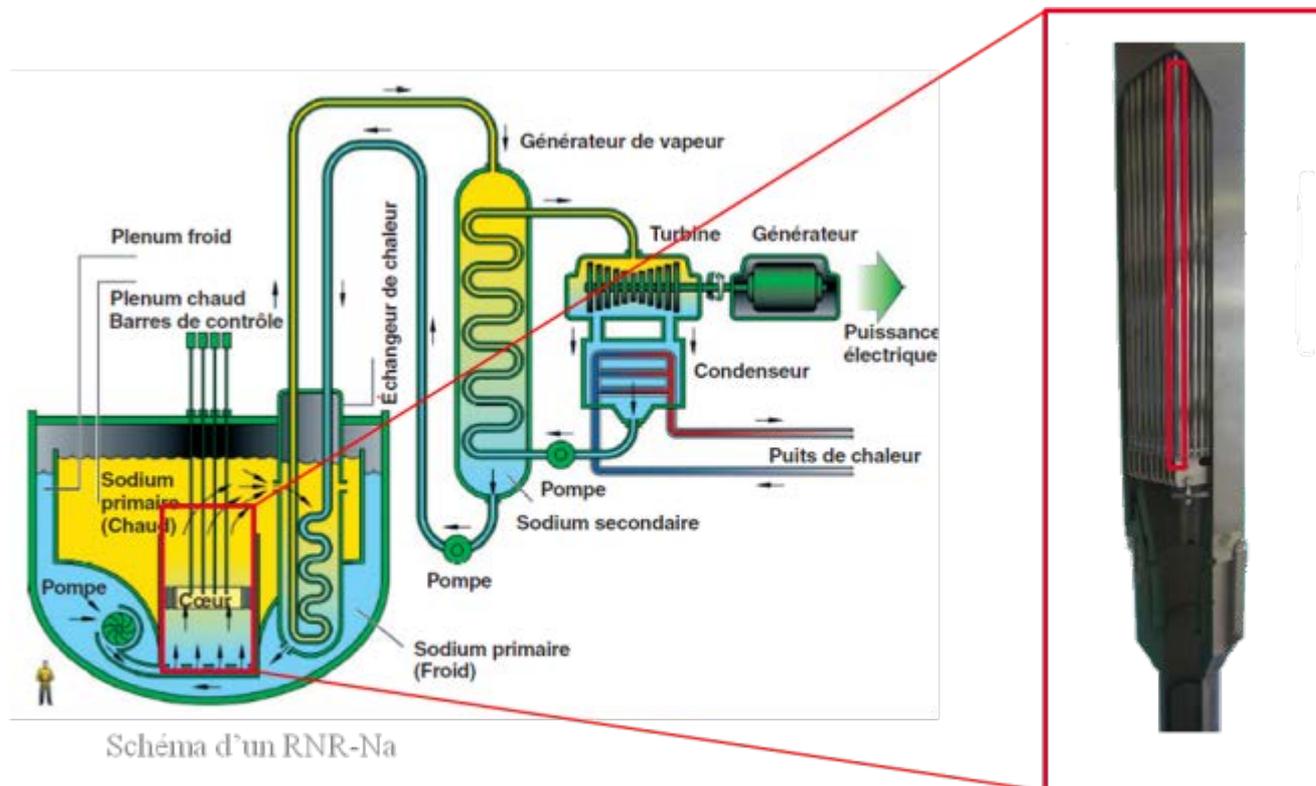
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The Sodium-cooled Fast Reactor: SFR (Gen. IV)

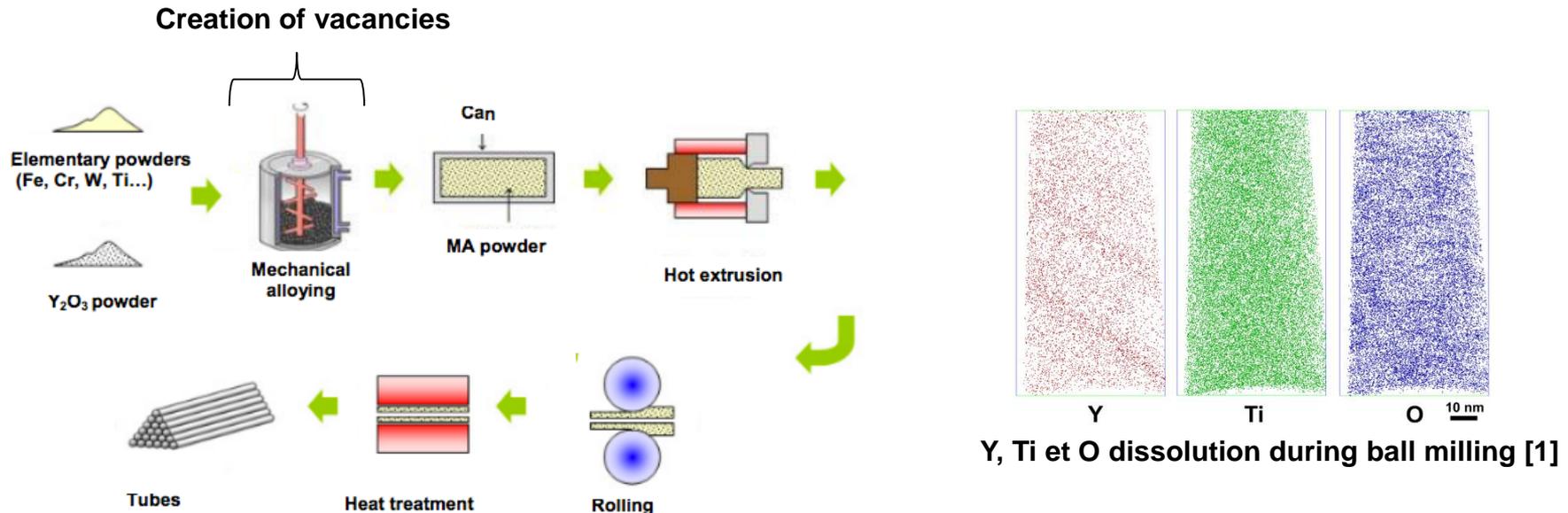


In-service extreme conditions:

- Irradiation dose > 150 dpa
- Temperature $T \approx 400 - 650^\circ \text{C}$

➔ Needs to develop new materials for cladding tubes with excellent mechanical properties at high temperatures:
The Oxide Dispersion Strengthened (ODS) steels

Elaboration process route: mechanical alloying and thermo-mechanical treatment

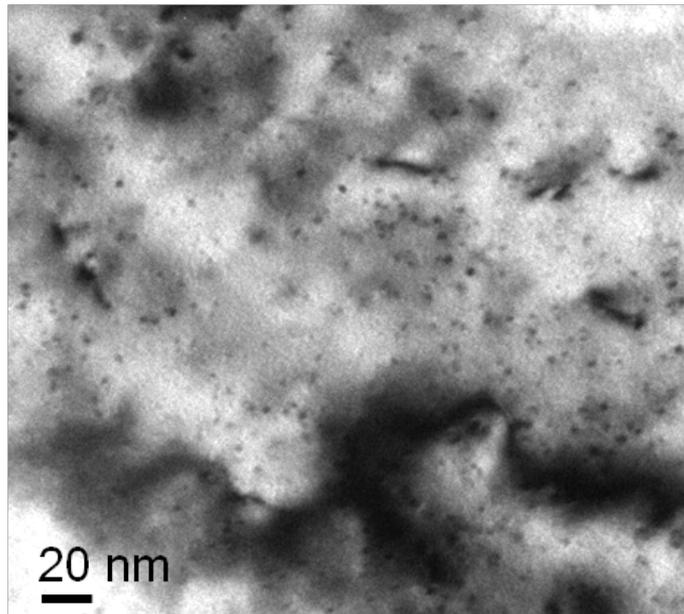


→ The mechanical alloying leads to the Y, Ti, O dissolution in the ferritic matrix [1]. Further, high concentration of vacancies are created [2], these defects are stabilized by the C, N and O solute [3].

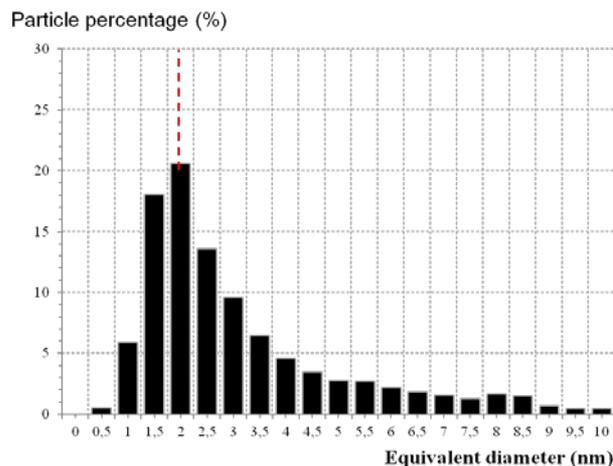
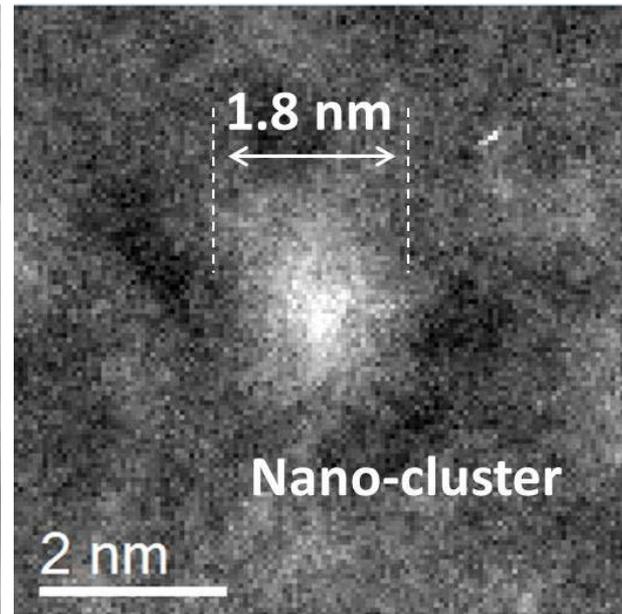
[1] D. Hoelzer, talk at the "académie des sciences" (France, 2008)

[2] G. Martin and P. Bellon, "Driven alloys," *Solid State Physics*, Vol. 50, pp. 189-331 (1997) [3] Barouh et al. PRB 90 054112 (2014)

Nano-oxide distribution observed by TEM:

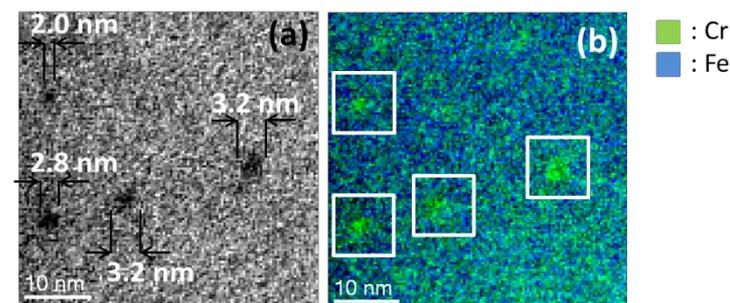
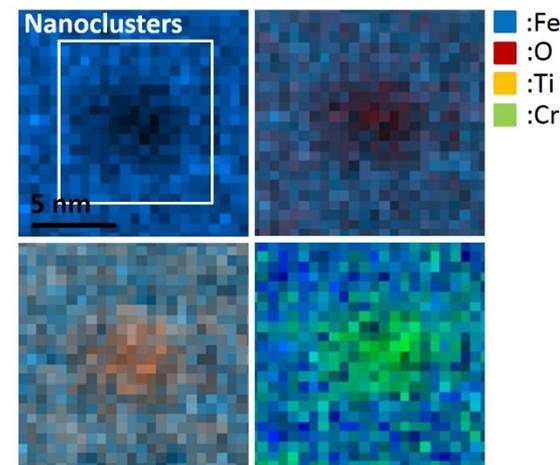
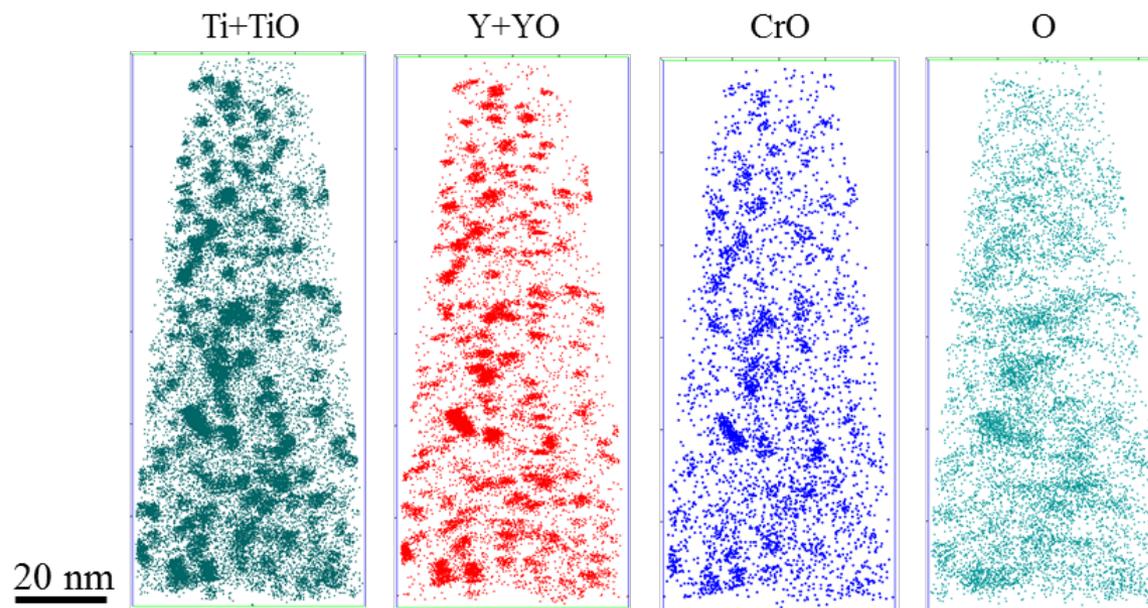


Fe14cr1W0.3Ti-0.3Y₂O₃ [1]



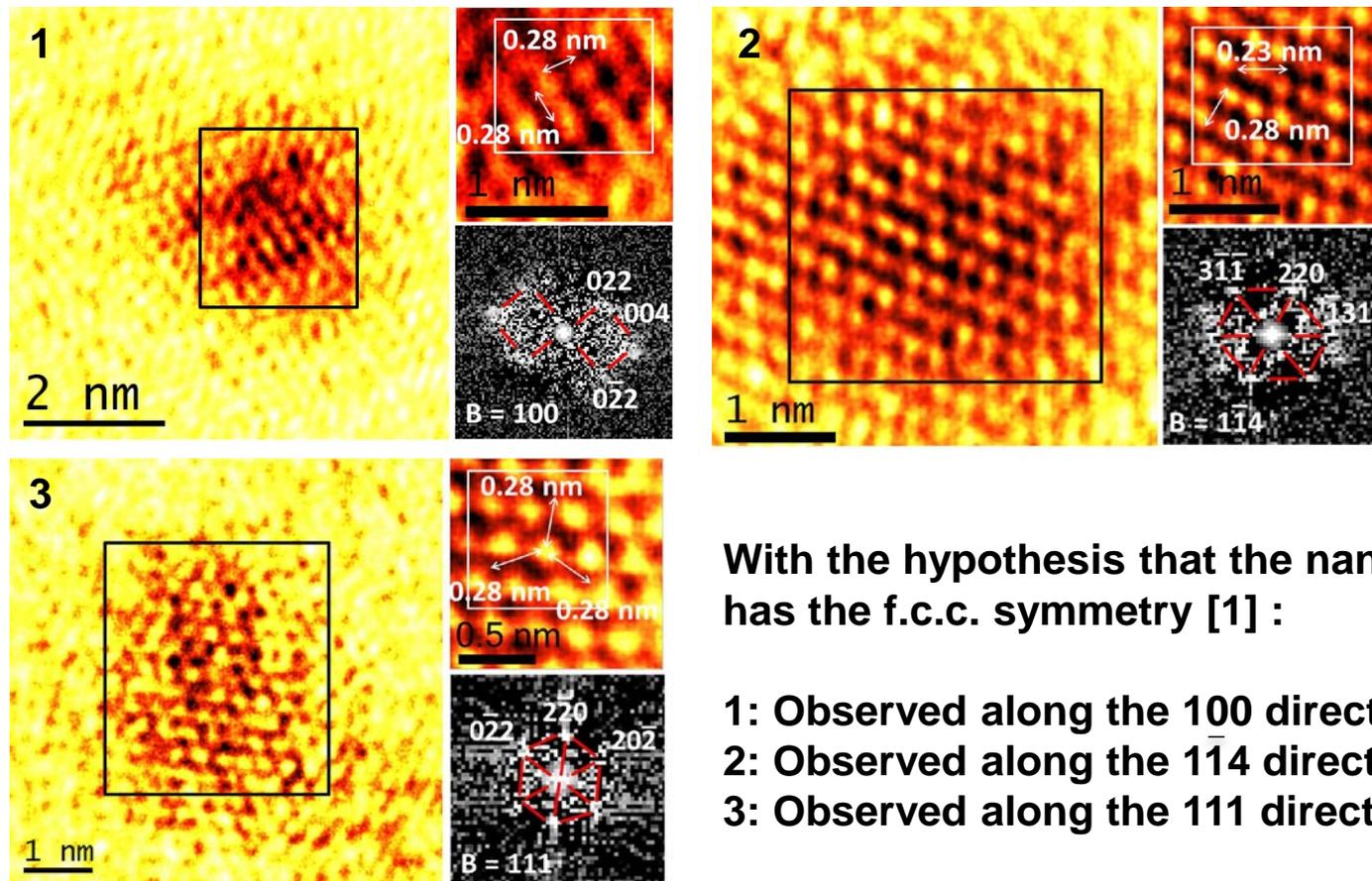
→ The ODS materials are mainly composed of nanoclusters which size is ranging between [1-3 nm]. Some oxide particles with a size larger than 3 nm coexist with these nanoclusters, but the nanoclusters are in majority ($\rho = 3 \times 10^{23} \text{ m}^{-3}$).

Chemical composition of the nanoclusters



- ➔ APT: the nanoclusters are $Y_xTi_yCr_hO_z$ compounds [1,2]
- ➔ EFTEM: confirmation of the Cr enrichment of the nanoclusters

Nanoclusters with size smaller than 3 nm were imaged using HRTEM



With the hypothesis that the nanocluster has the f.c.c. symmetry [1] :

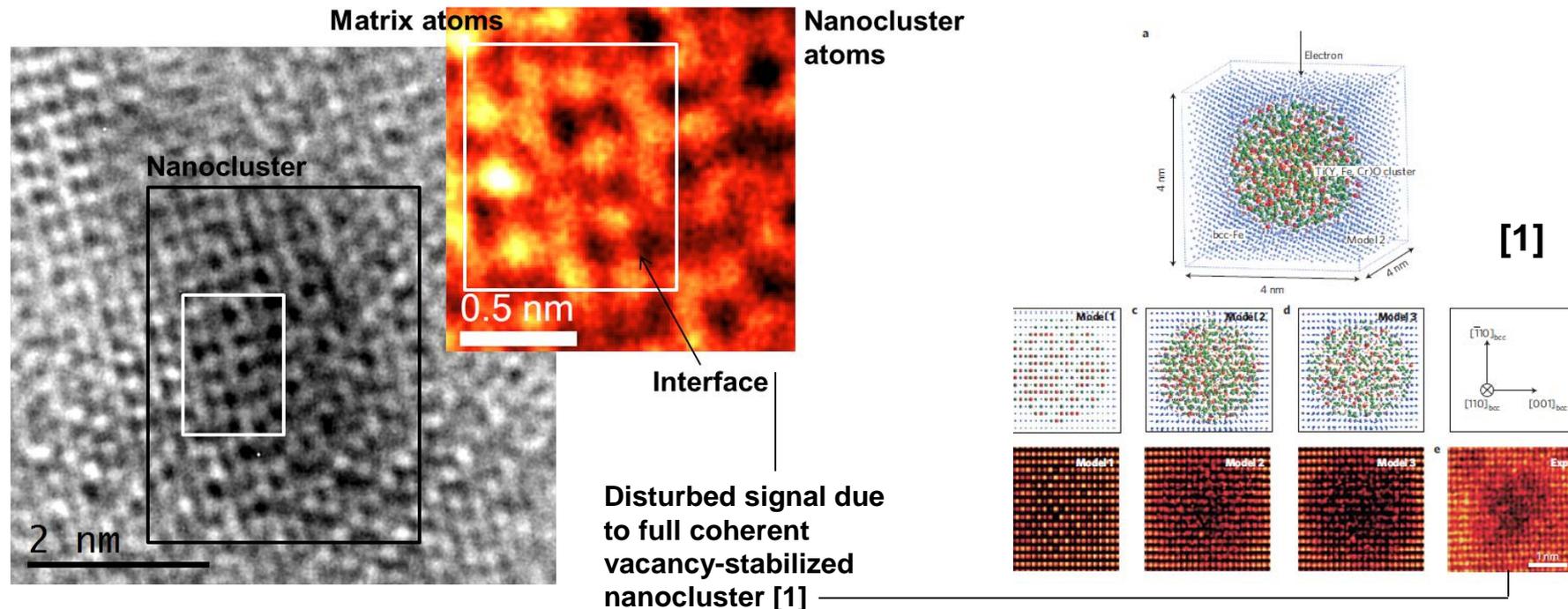
- 1: Observed along the 100 direction
- 2: Observed along the $\bar{1}\bar{1}4$ direction
- 3: Observed along the 111 direction

→ The nanoclusters display an unknown cubic structure (very likely to be fcc like structure as previously found [1]) .

→ Shape stability of such nanocluster under thermal annealing?

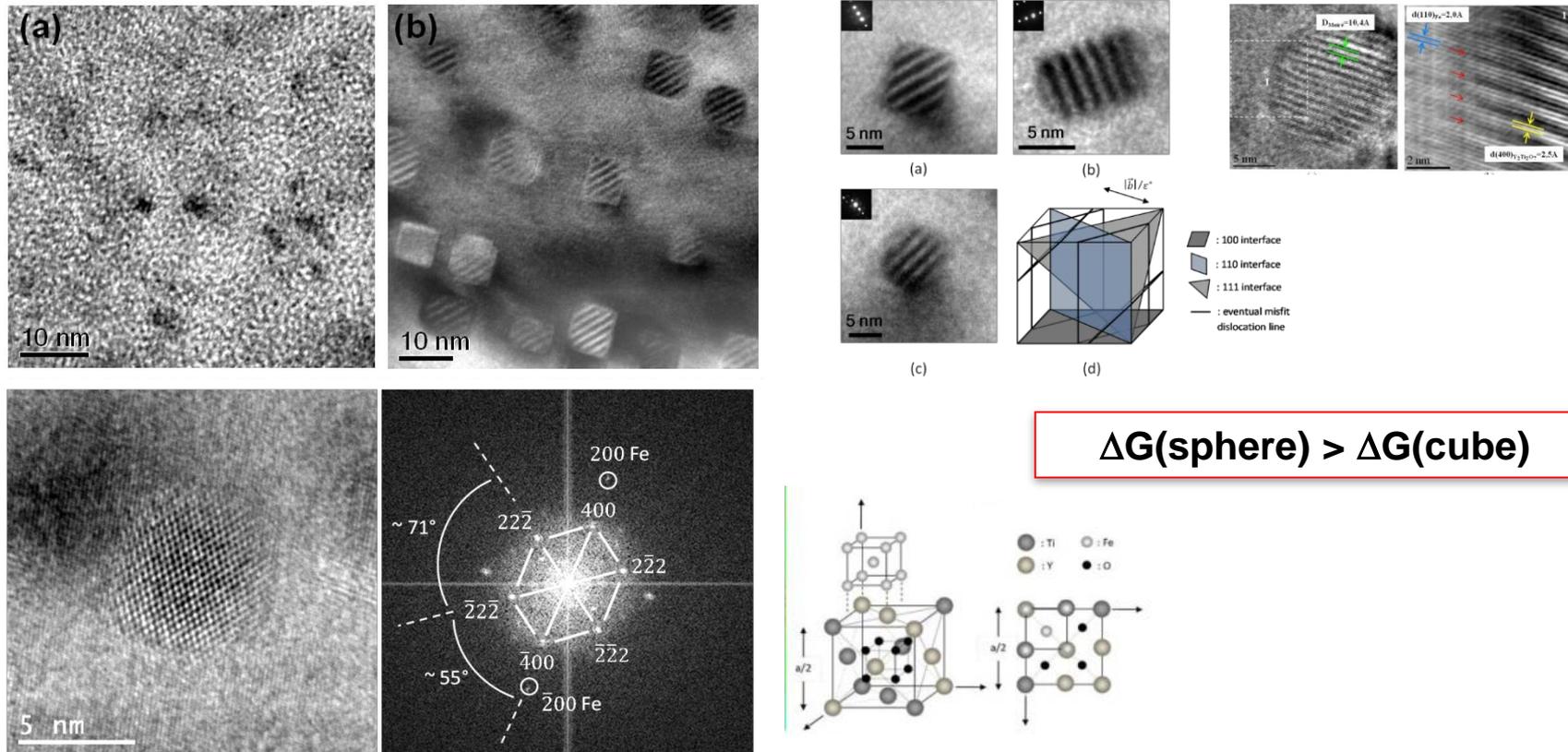
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Nanocluster interface structure imaged using HRTEM:



→ The nanoclusters appear to be embedded in the matrix with a full coherent relationship with the matrix, as already proposed [1]. The full coherent relationship could be at the origin of the stabilisation of the unknown fcc structure of the nanoclusters (minimization of the interface energy).

Structure change during relaxation after thermal annealing: 1h 1300° C [1]

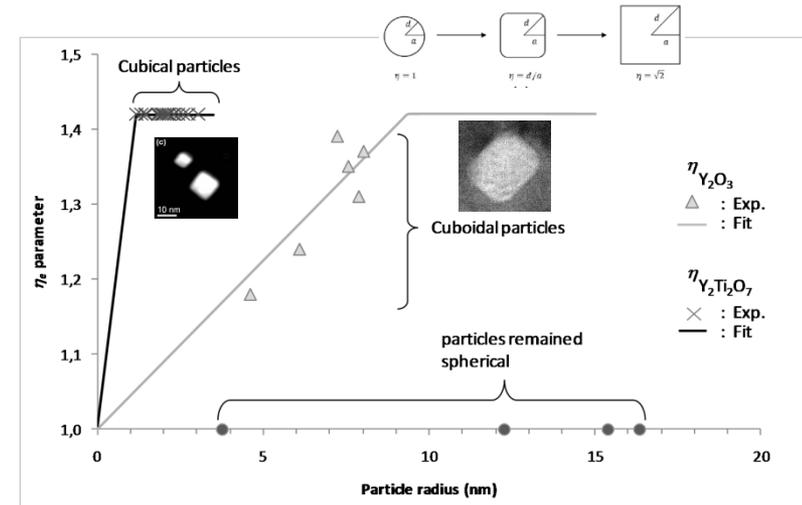
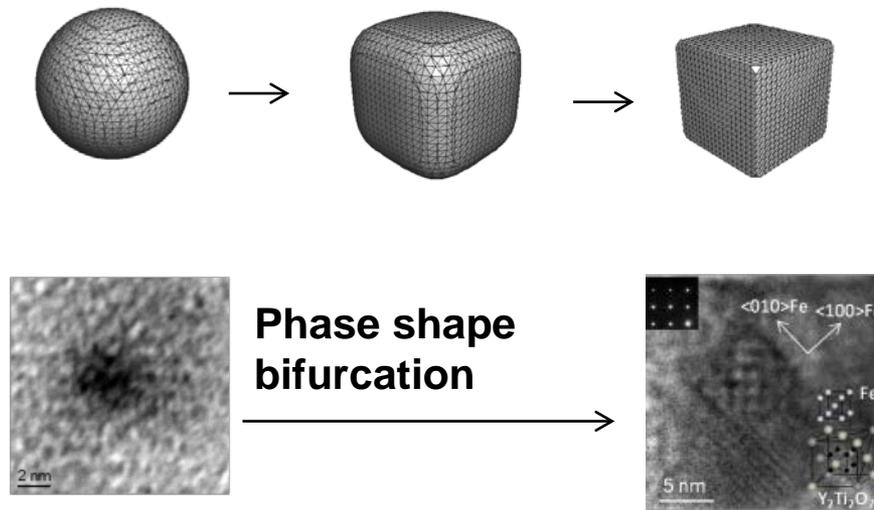


$$\Delta G(\text{sphere}) > \Delta G(\text{cube})$$

After thermal annealing at 1300° C during 1h most of the particles display a fcc $\text{Y}_2\text{Ti}_2\text{O}_7$ -like structure and are embeded with cube-on-cube orientation relationships with the matrix. The pyrocholre have different lattice parameter than the matrix which is at the origin of a strain energy stored at the interface → Shape bifurcation from spheric to cubic and introduction of misfit dislocations [1]

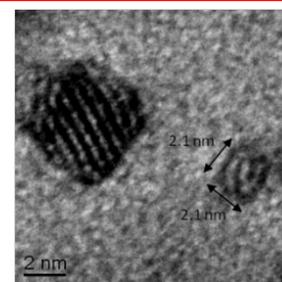
Phase shape stability during thermal annealing [1,2]

Calculation of the shape bifurcation radius for $Y_2Ti_2O_7$ and Y_2O_3 type phase



$$\eta_e \approx 1 + \frac{h(A_r, \nu_{100})[1 - \alpha(A_r, \nu_{100})]C_{44}e^{*2}}{6(\sqrt{2} - 1)K\sigma_{oxide}^{(100)}} r$$

Elasticity calculations allow to estimate the bifurcation shape radius. It is found to be equal to 2 nm for the $Y_2Ti_2O_7$ like particles and to 10 nm for the Y_2O_3 particle. The interface energy has been found to be equal to 290 mJm⁻² for $Y_2Ti_2O_7$ like particles [1,2]



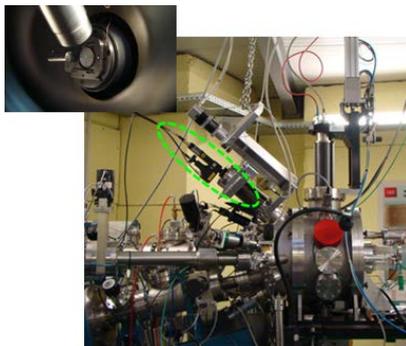
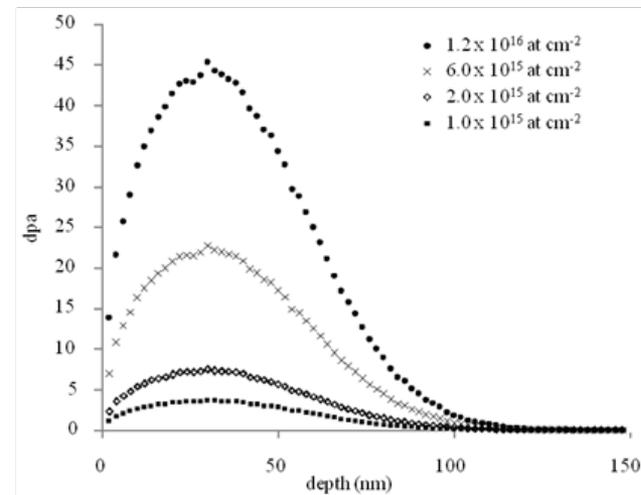
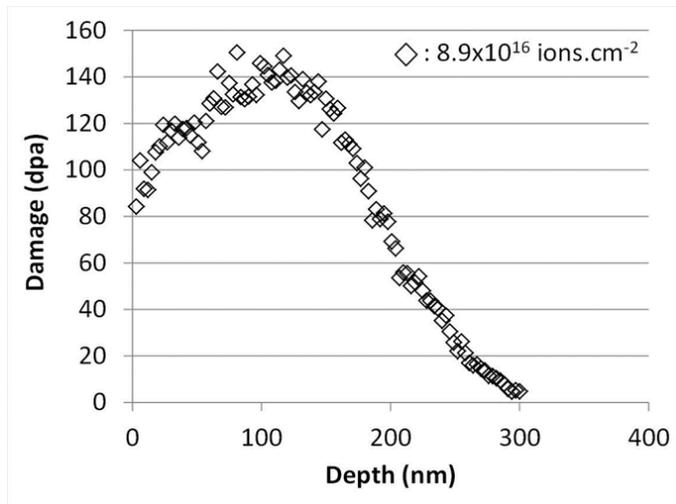
➔ **Shape instability** : from spherical to cubic (metastable shape)

→ Shape stability of such cubical particle under irradiation?

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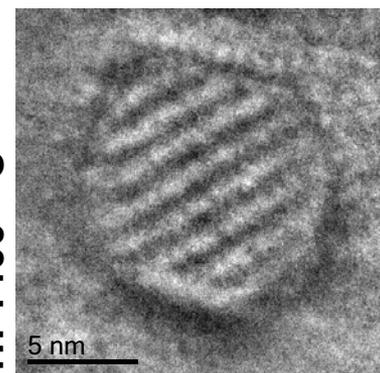
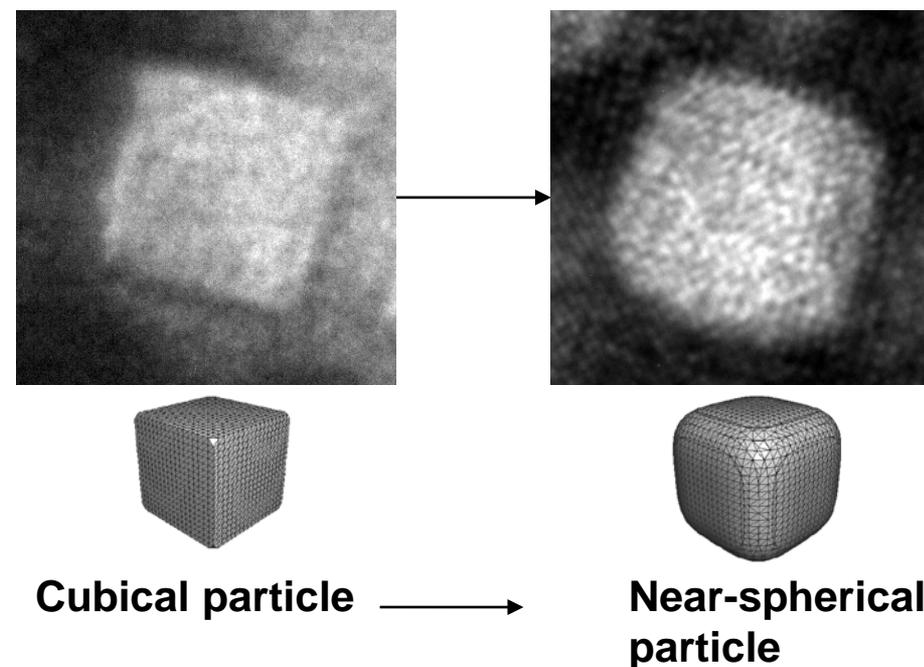
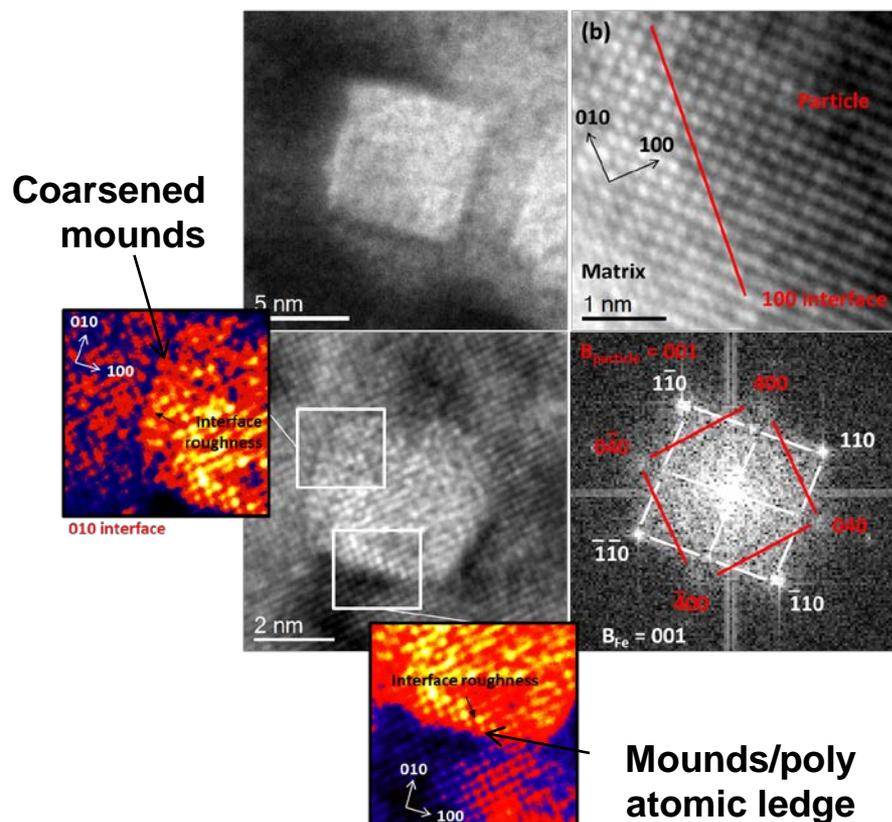
How the cubical/faceted shape evolve under irradiation?

To answer this questions two irradiations were conducted, ex-situ in Jannus saclay (up to 150 dpa at 500° C), and in-situ in Jannus Orsay (up to 45 dpa at 500° C) using Fe⁺ ions.



Shape stability under irradiation: 150 dpa 500° C

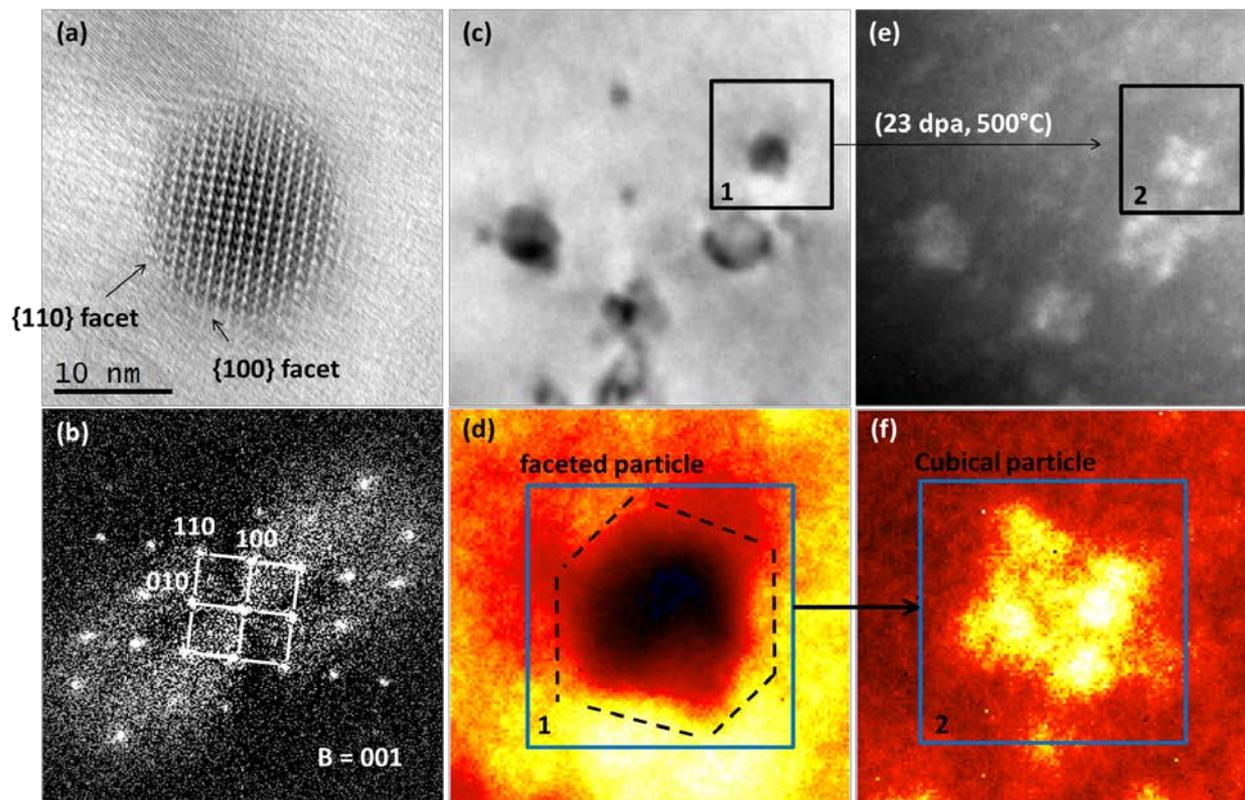
- Precipitate shape transformation (for cubical particles)



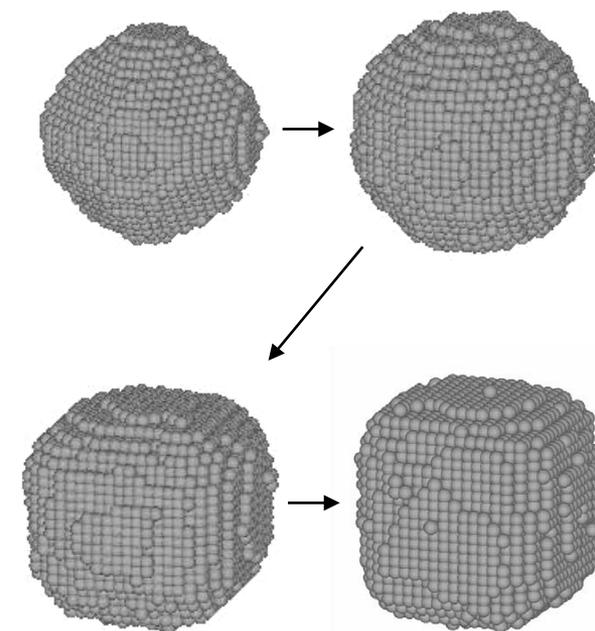
Due to the Radiation-Enhanced Diffusion, the irradiation drives the particle from the metastable cubical shape toward the spherical shape (equilibrium) by coarsening of mounds. The particle adopt at 500° C under irradiation the shape it would have adopted at high temperatures.

Shape stability under irradiation: 23 dpa 500° C

- Precipitate shape transformation (for faceted particles)



KMC calculation, $\Gamma_b = 10 \text{ s}^{-1}$, 400K [1]

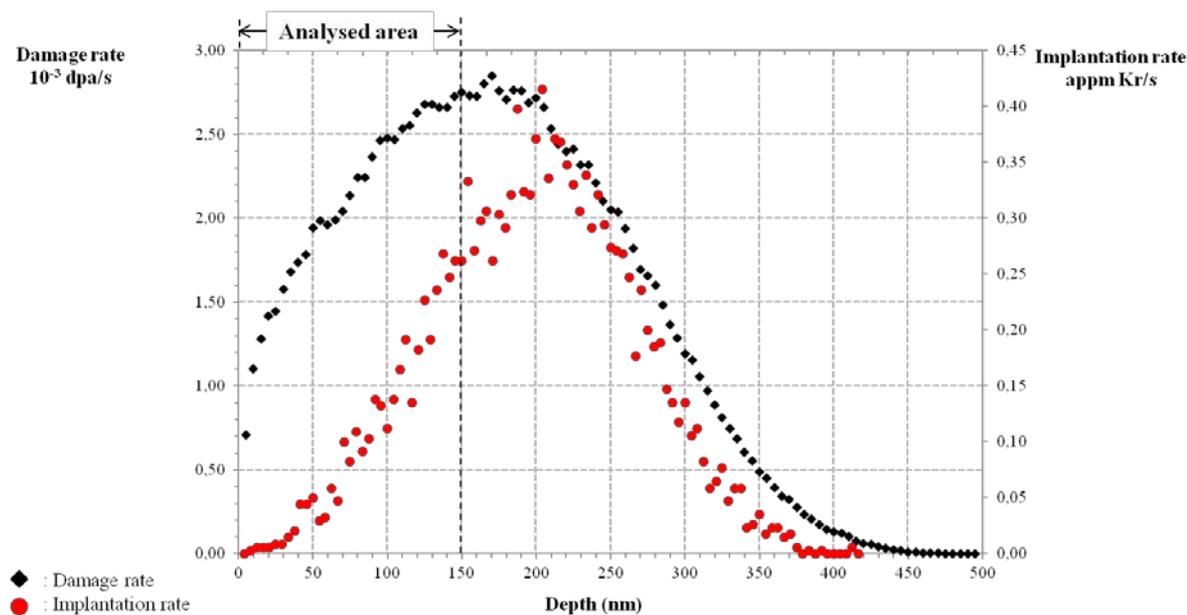
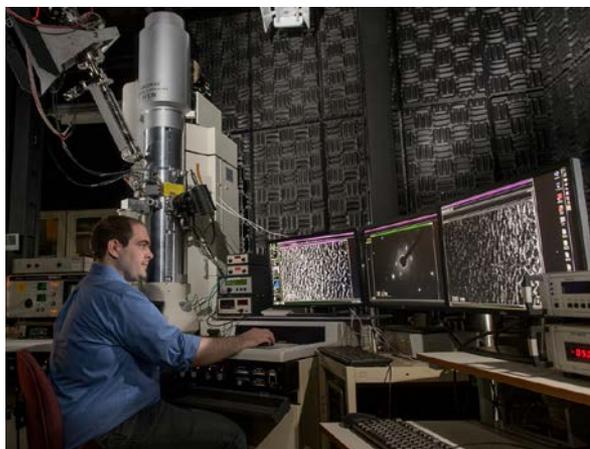


If the initial precipitate is faceted, the dynamical shape transformation produces non-equilibrium cubical shape. The explanation of this phenomenon could reside in the fact that $\{111\}$ interfaces locally develop $\{100\}$ facets since they have larger fraction of nearest neighbours out-of-plane [1].

- **Stability of the interfaces under irradiation is a key point since interface are supposed to be site for point defect annealing**
- **This could prevent the formation of bubbles in case of neutron irradiation (for fusion reactor)**
- **What is the interaction between oxide interfaces and bubbles?**

What are the interaction between interfaces and bubbles?

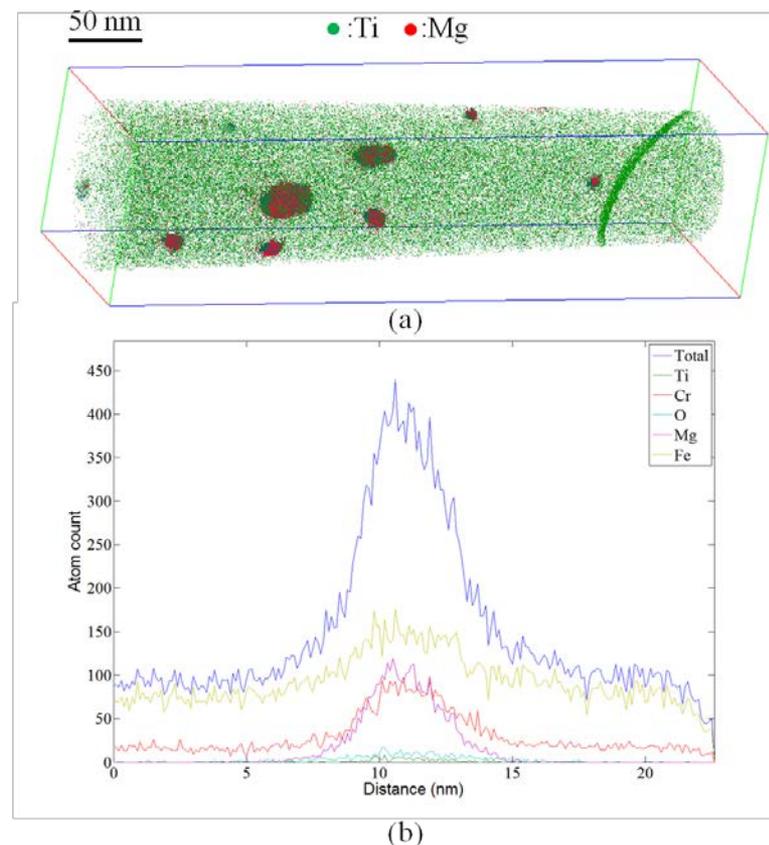
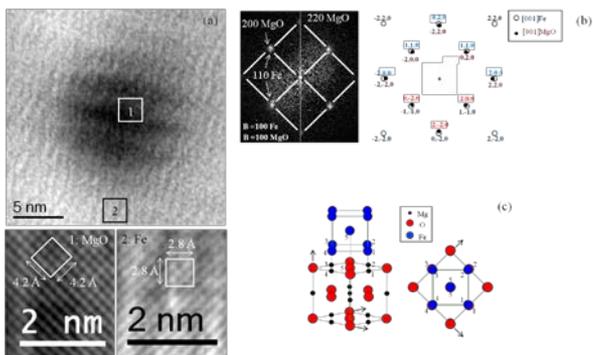
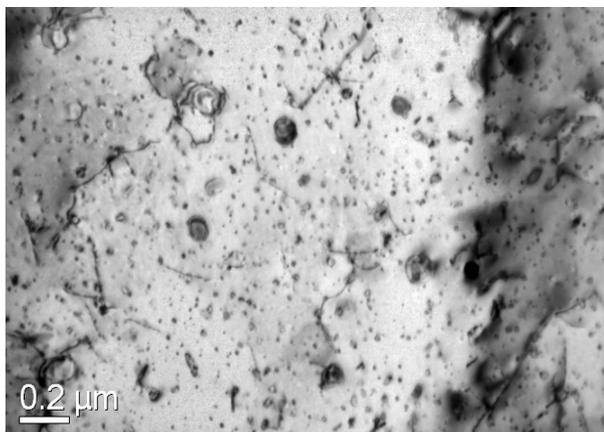
- Implantation of Kr using IVEM facility (ANL) in MgO ODS



Irradiation up to 58 dpa at RT
Implantation of Kr up to 0,8%

The MgO ODS steels:

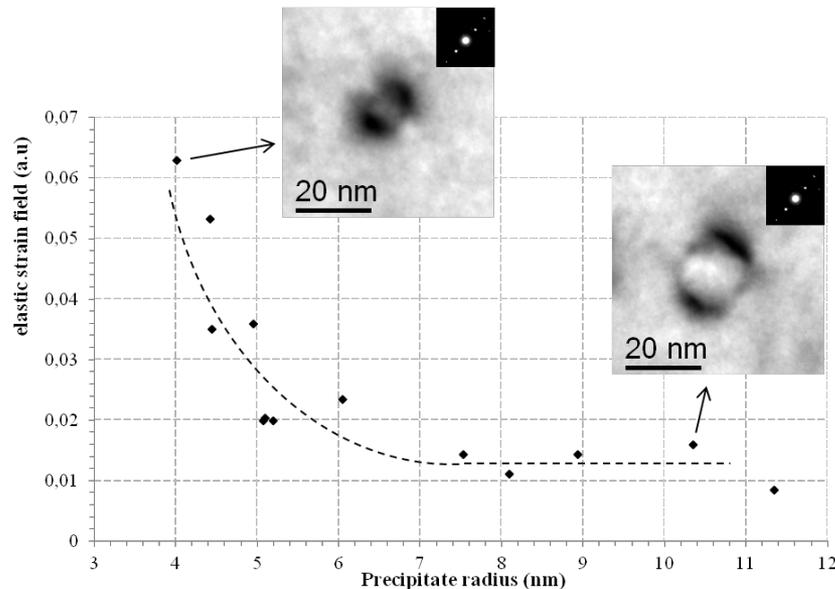
- Fe-1W-18Cr-0.6Ti-0.3 MgO



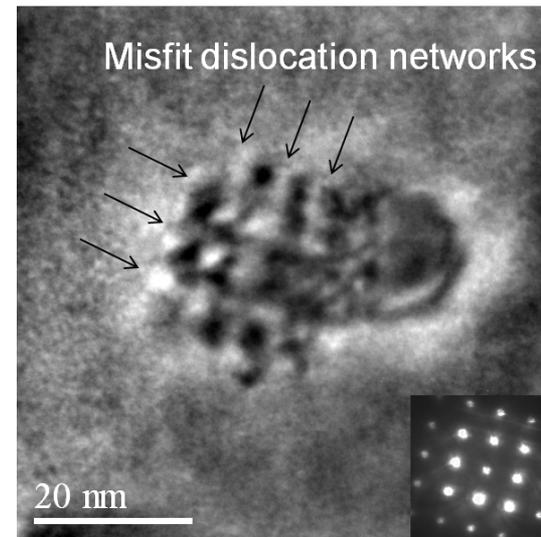
The nano-particle are identified as MgO type particles with a fcc cubic structure. The particle appear to be coherent and have Baker and Nutting orientation relationship.

Interface strained structure of the particle:

- Particles are coherent with a misfit strain



(a)

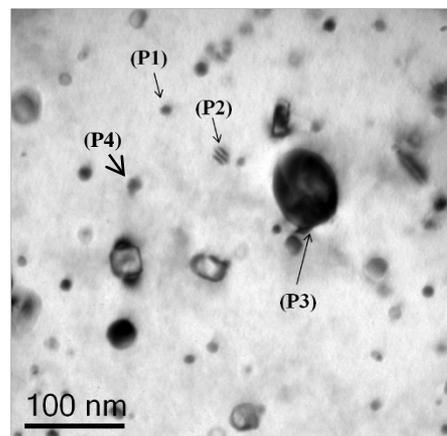


(b)

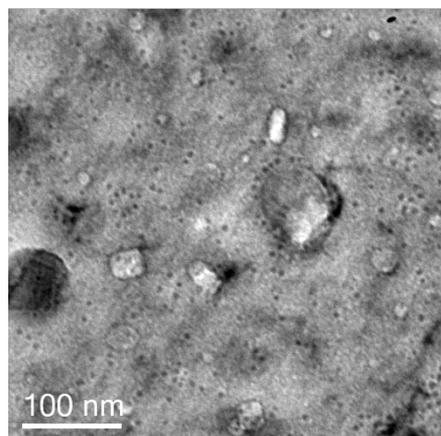
Evolution of the Ashby-Brown contrast regarding the coherency degree of the particles. The Ashby-Brown contrast tends to disappear as the particles become larger → the interface strain is relaxed by the appearing of misfit dislocations.

Formation of Kr-cavities under irradiation:

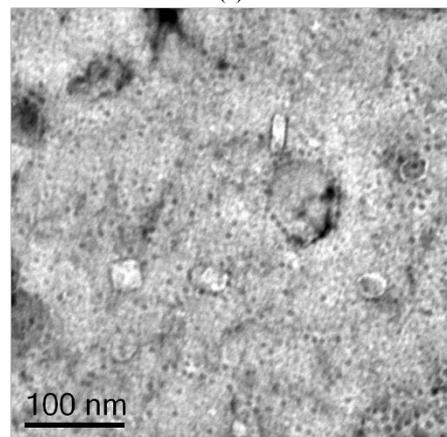
- Interaction with coherent particles, semi-coherent particles and incoherent particles



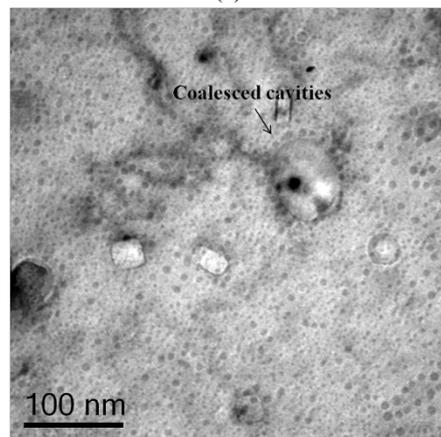
(a)



(b)



(c)



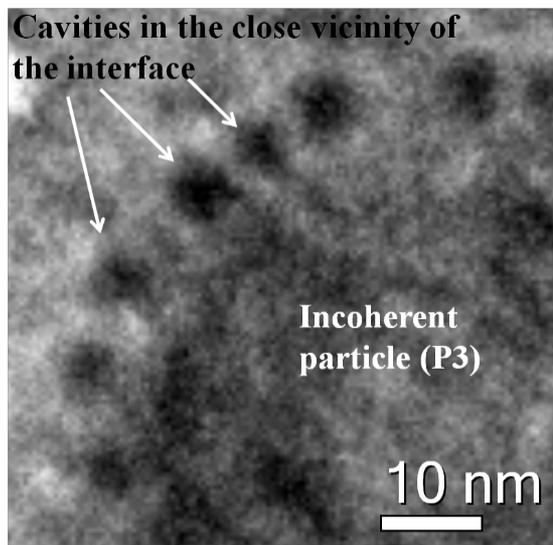
(d)

P1: semicoherent particle
P2: semicoherent particle
P3: incoherent particle
P4: coherent particle

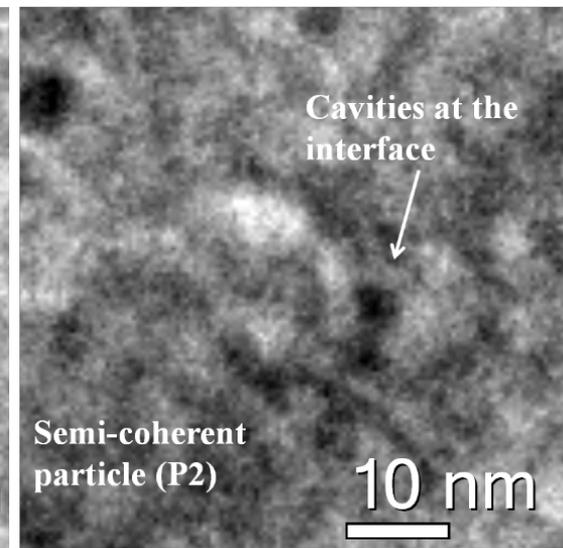
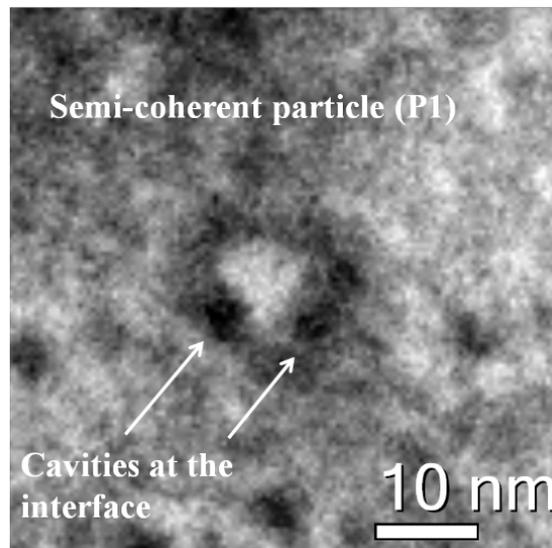
(a): Non irradiated
(b): 28 dpa RT
(c): 40 dpa RT
(d): 58 dpa RT

Formation of Kr-bubbles under irradiation:

Incoherent



Semicoherent



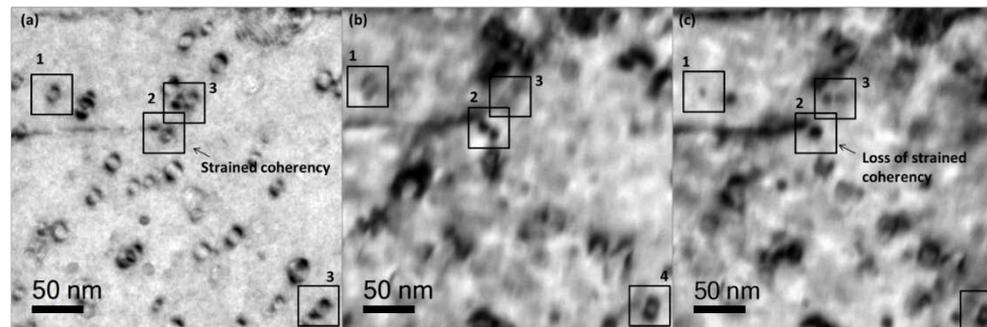
For incoherent particles, the bubbles form in the close vicinity of the particle but not strictly at the interface. For the semi-coherent particles, the bubbles form at the interface, possibly at the free volume of the misfit dislocations.

Formation of Kr-bubble under irradiation:

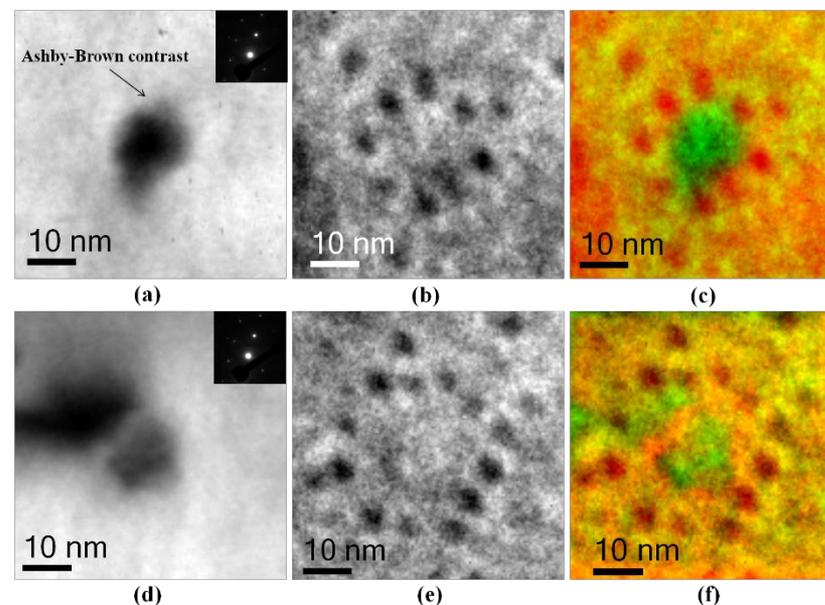
- Coherent with misfit strain

For the coherent particles which present a misfit strain, the bubbles form around the particle but quite far from it. Further, it seems that under irradiation the Ashby-Brown contrasts disappear.

Point defects may be trapped by the strained interface (relaxing the strain), the trapped defects may annihilate with other defects in the vicinity of the interface, explaining why cavities only form at few nanometers away from the particles.



Loss of strained coherency by trapping the point defects



The nano-oxide dispersion in ODS steels:

- ODS steels are mainly composed of nanoclusters
- The nanoclusters display an unknown fcc cubic symmetry structure
- The nanoclusters are YTiCrO rich

Shape stability during thermal annealing:

- During thermal annealing (1h 1300° C) the unknown fcc structure change into pyrochlore type structure
- Due to the full coherency of the nanoclusters, the elasticity at the interface leads the particle to adopt a cubical shape

Shape stability under irradiation:

- Ex situ irradiation (150 dpa 500° C) cubical shaped particle evolve toward near-spherical shaped particle
- In situ irradiation (23 dpa 500° C) faceted particle evolve toward cubical shaped particle

Cavity interaction with interfaces:

- Incoherent particles: bubbles form in the close vicinity of the interface
- Semi-coherent particles: bubbles form at the interface on the misfit dislocations
- Coherent particle with a misfit strain: bubbles form around the particles few nanometers away from the particle probably due to the point defect trapping effect