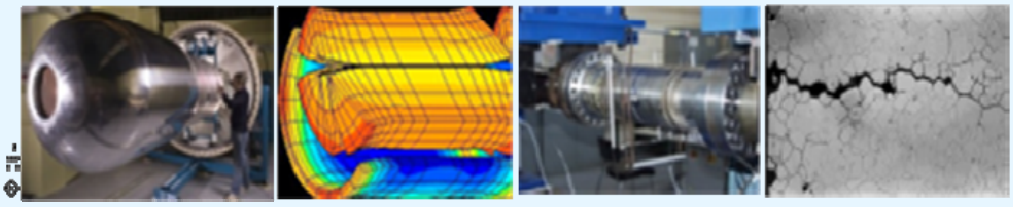
MINOS

Centre of Excellence for Nuclear Materials

Workshop

Materials Innovation for Nuclear Optimized Systems



December 5-7, 2012, CEA – INSTN Saclay, France

Yann De CARLAN

CEA (France)

Relationship between Microstructure and Mechanical Properties in ODS Materials for Nuclear Application

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Workshop Materials Innovation for Nuclear Optimized Systems
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Relationship between Microstructure and Mechanical Properties in ODS Materials for Nuclear Application

Yann De CARLAN[1]

[1]CEA-DEN-DMN, Service de Recherches Métallurgiques Appliquées, SRMA (Saclay, France)

Oxide Dispersion Strengthened ferritic/martensitic alloys are developed as prospective cladding materials for future Sodium-Cooled-Fast-Reactors (GEN IV) [1]. These advanced alloys present a good resistance to irradiation and a high creep rupture strength due to a reinforcement by the homogeneous dispersion of hard nano-sized particles (such as Y_2O_3 or YTiO). ODS alloys are elaborated by powder metallurgy, consolidated by hot extrusion and manufactured into cladding tube using the pilger cold-rolling process [2, 3]. ODS alloys present usually low ductility and high hardness. The aim of this talk is to present the specificity of the metallurgy of ODS materials in relationship with the main mechanical properties (tensile and creep properties, toughness, transition temperature…). Two types of alloys will be presented: Fe-9Cr martensitic ODS and Fe-14Cr ferritic ODS alloys. Mechanical properties of the materials depend on the metallurgical state (fine grains, recrystallized, martensitic) and very different behaviors are observed as a function of final microstructure. For example, for a Fe-9CrODS alloy, tempered martensite lets obtaining material with high strength whereas softened ferrite see figure 1 [4] tolerates high deformation levels.

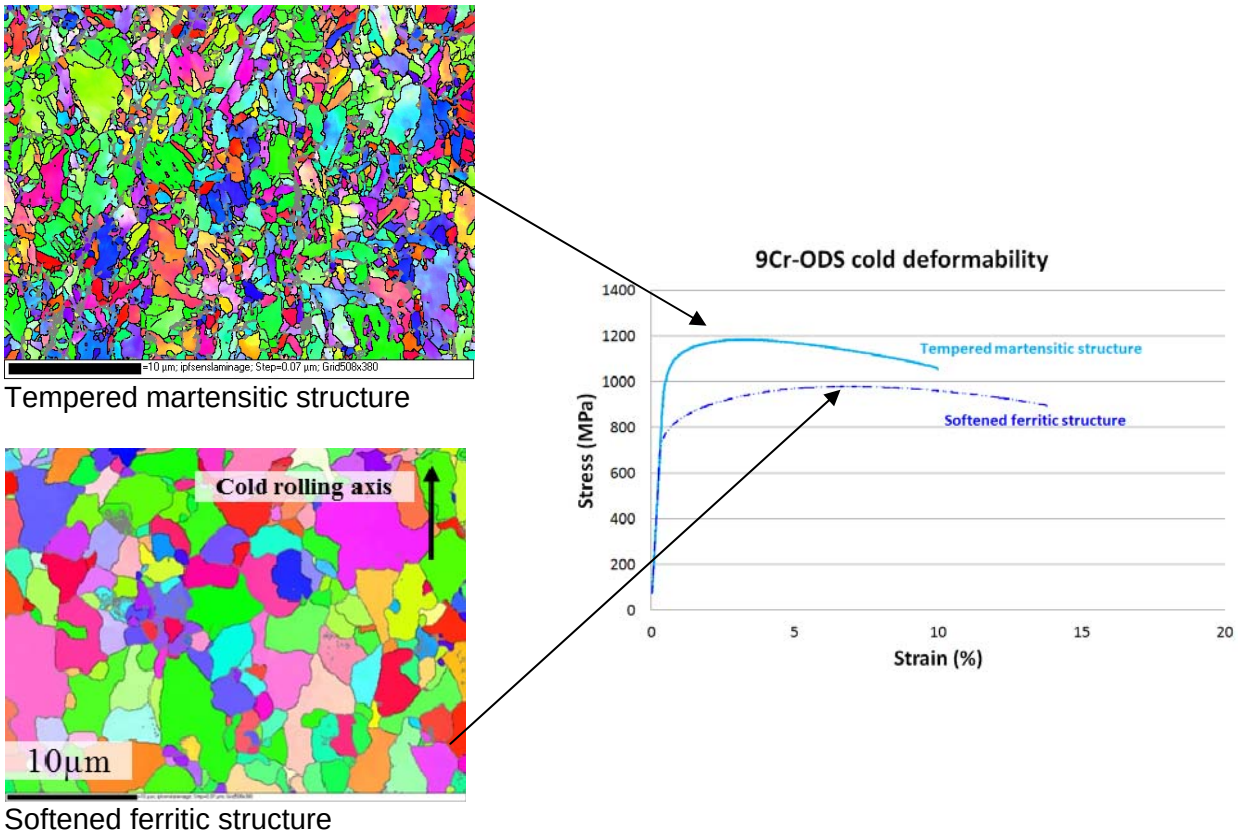

Fig. 1: Electron Backscatter Diffraction maps for a Fe-9Cr ODS associated with the tensile properties at room temperature.

References

[1] P. Dubuisson, Y. de Carlan, V. Garat, M. Blat, ODS Ferritic/martensitic alloys for Sodium Fast Reactor fuel pin cladding. Journal of Nuclear Materials 428 (2012) 6–12.

[2] T. Narita, S. Ukai, T. Kaito, S. Ohtsuka, T. Kobayashi, Development of Two-Step Softening Heat Treatment for Manufacturing 12Cr–ODS Ferritic Steel Tubes. Journal of Nuclear Science and Technology, Vol. 41, No. 10, p. 1008–1012 (October 2004).

[3] S. Ohtsuka, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, Improvement of 9Cr-ODS martensitic steel properties by controlling excess oxygen and titanium contents. Journal of Nuclear Materials 329-333(1): 372-376.

[4] L. Toualbi, C. Cayron, P. Olier, R. Logé, Y. de Carlan. Relationships between mechanical behavior and microstructural evolutions in Fe 9Cr-ODS during the fabrication route of SFR cladding tubes. Submitted in Journal of Nuclear Materials, April 2012.

DE LA RECHERCHE À L'INDUSTRIE

RELATIONSHIP BETWEEN MICROSTRUCTURE AND MECHANICAL PROPERTIES IN ODS MATERIALS FOR NUCLEAR APPLICATION

Yann de Carlan et al., CEA , DEN, SRMA, 91191 GIF/YVETTE FRANCE

MINOS Workshop, Materials Innovation for Nuclear Optimized Systems
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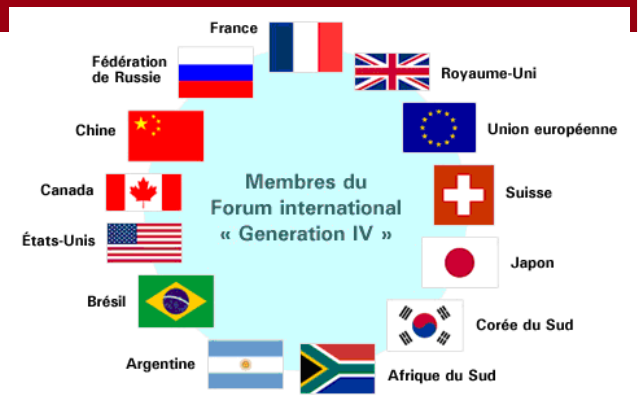
ODS FOR SODIUM FAST REACTORS

MINOS

Rapsodie

Phénix

Superphénix



Forum Gen IV : reactors

Beginning of the studies on the Fast Sodium Reactors to produce electricity

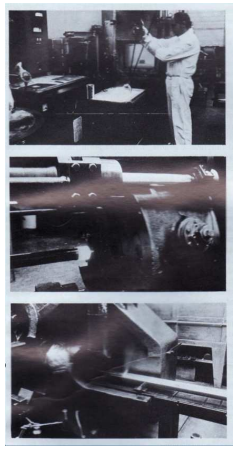
1960 | 1967 | 1973 | 1985 | 1987 | 2000

Materials

Optimization of the austenitic steels

New reinforced ferritic alloys : **ODS alloys**

First ODS (SCK-CEN)

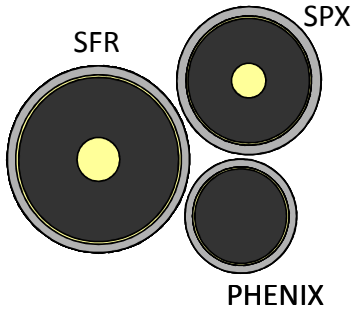


First irradiated tubes

Industrial Production (DourMetal)

ODS tubes in Phenix reactor

Need of optimized materials is still relevant !



L. Toualbi

Why ODS materials for Nuclear Applications ?

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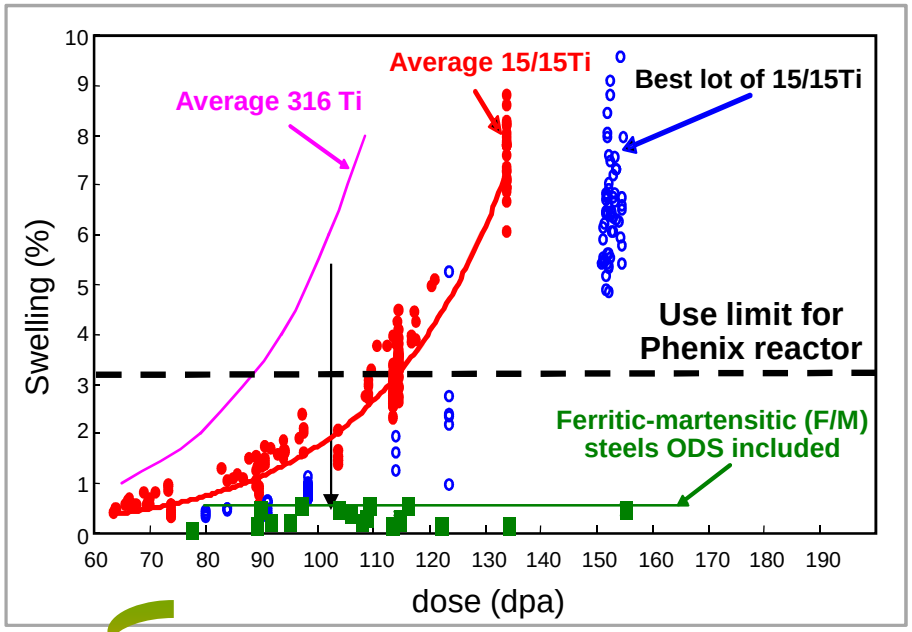
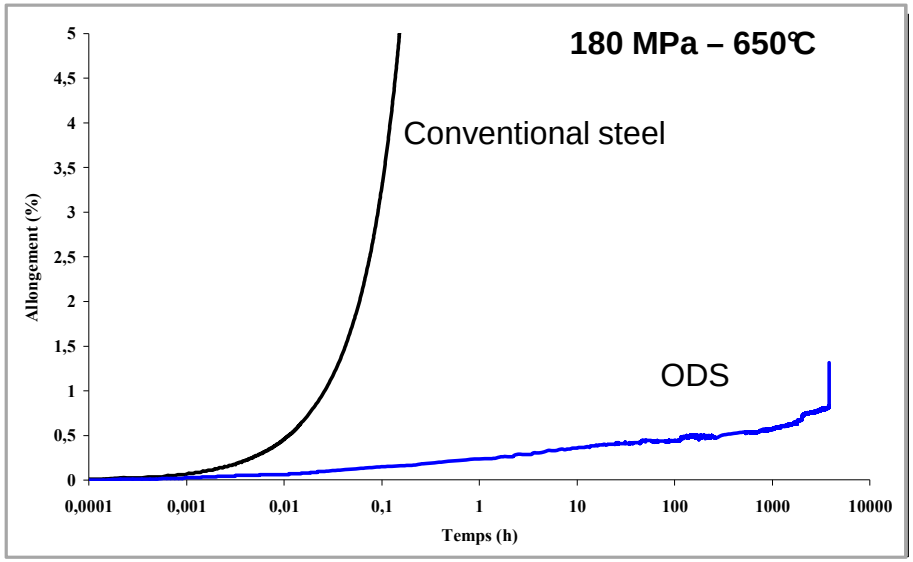
SFR
Dose > 150 dpa
Stress ~ 100 MPa
Temperatures : 400℃ – 670℃

ODS materials

(Oxide Dispersion Strengthened)



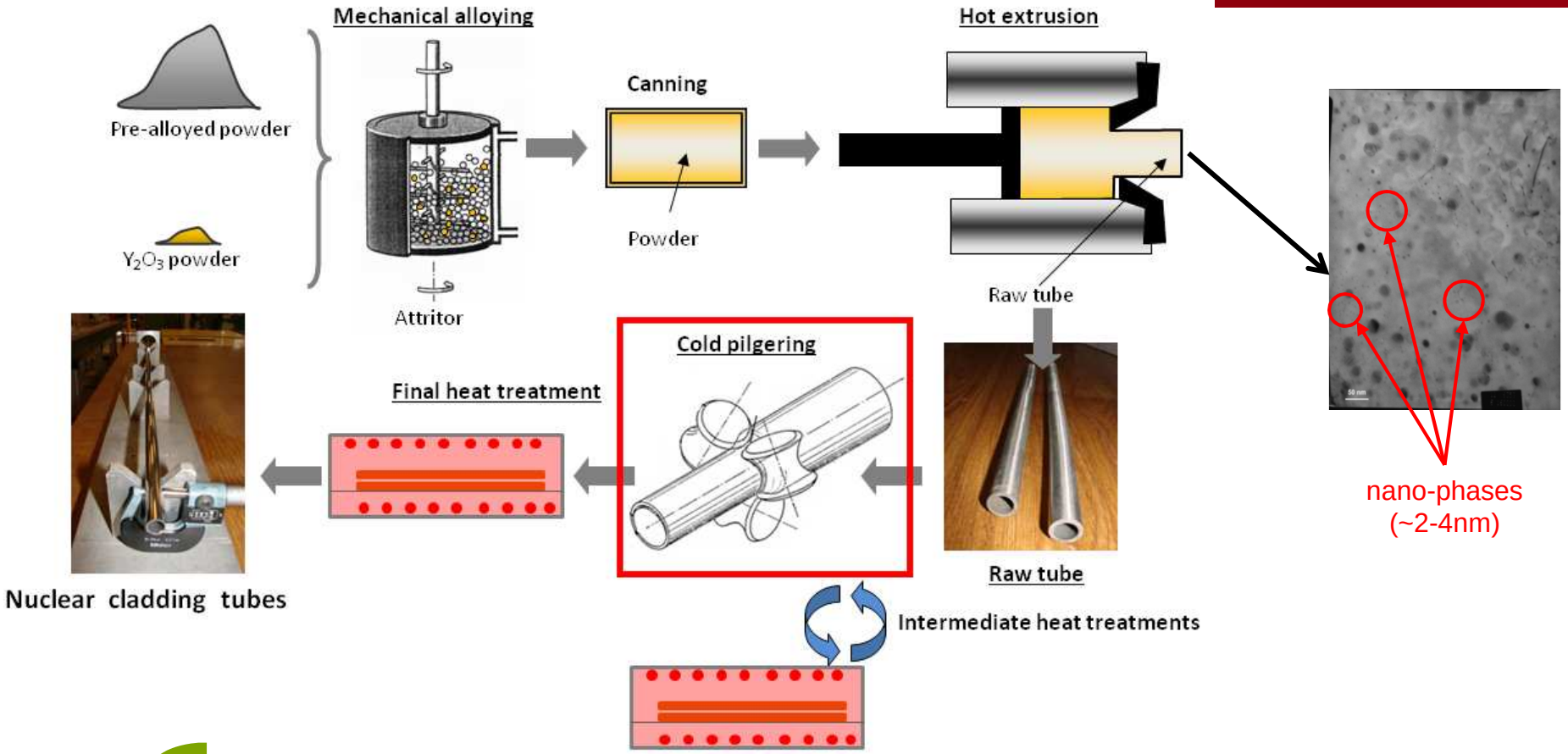
Limited swelling under irradiation



Low thermal creep

Nano dispersion

METALLURGY OF ODS MATERIALS

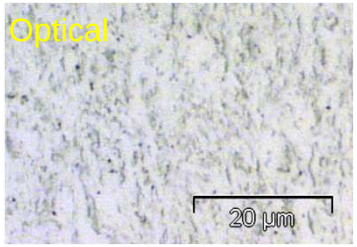


Complex manufacturing

- ODS steels present low ductility and limited consolidation at room temperature
- Low deformability
- Intermediate heat treatments needed for stress relief purpose

METALLURGY OF ODS MATERIALS

MINOS

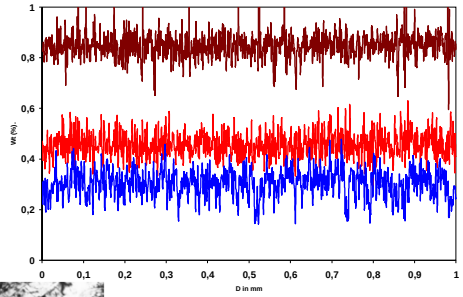


Ex:ODS steel
18 Cr-1W Ti 0,5 Y_2O_3

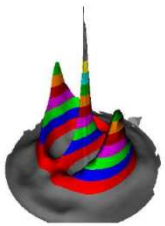
Uniform distribution of nano oxides

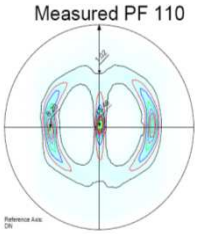
Plate

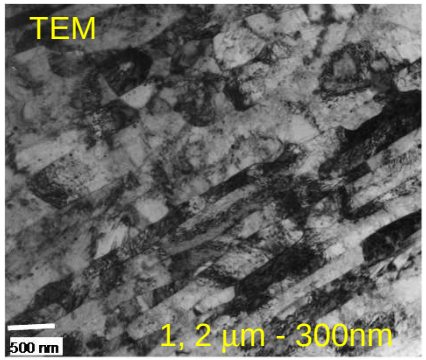
Microprobe



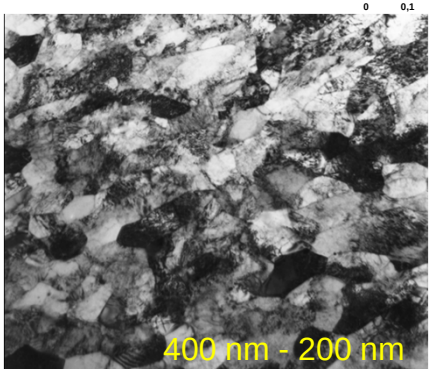
Texture





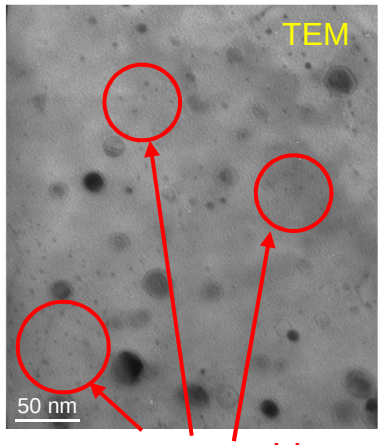


Longitudinal direction



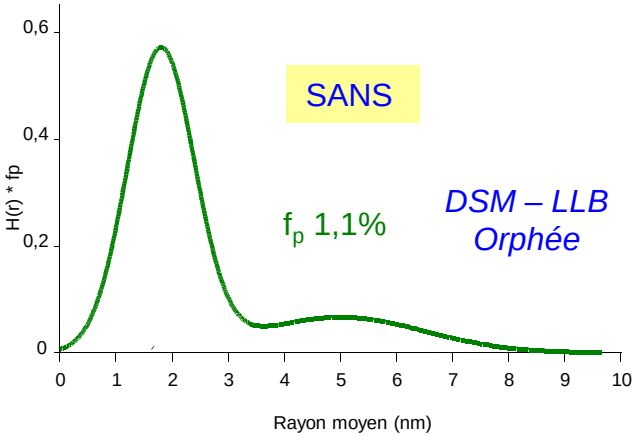
Transverse direction

Homogeneous microstructure
Fine ferritic grains
elongated
in extrusion direction

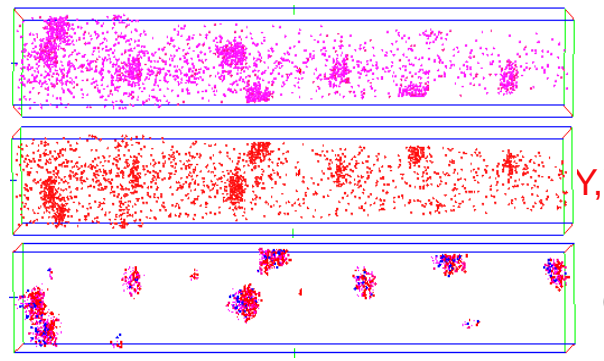


EFTEM
HRTEM
nano-oxides
$8\ 10^{22}\ m^{-3}$ ϕ 3 nm

SANS

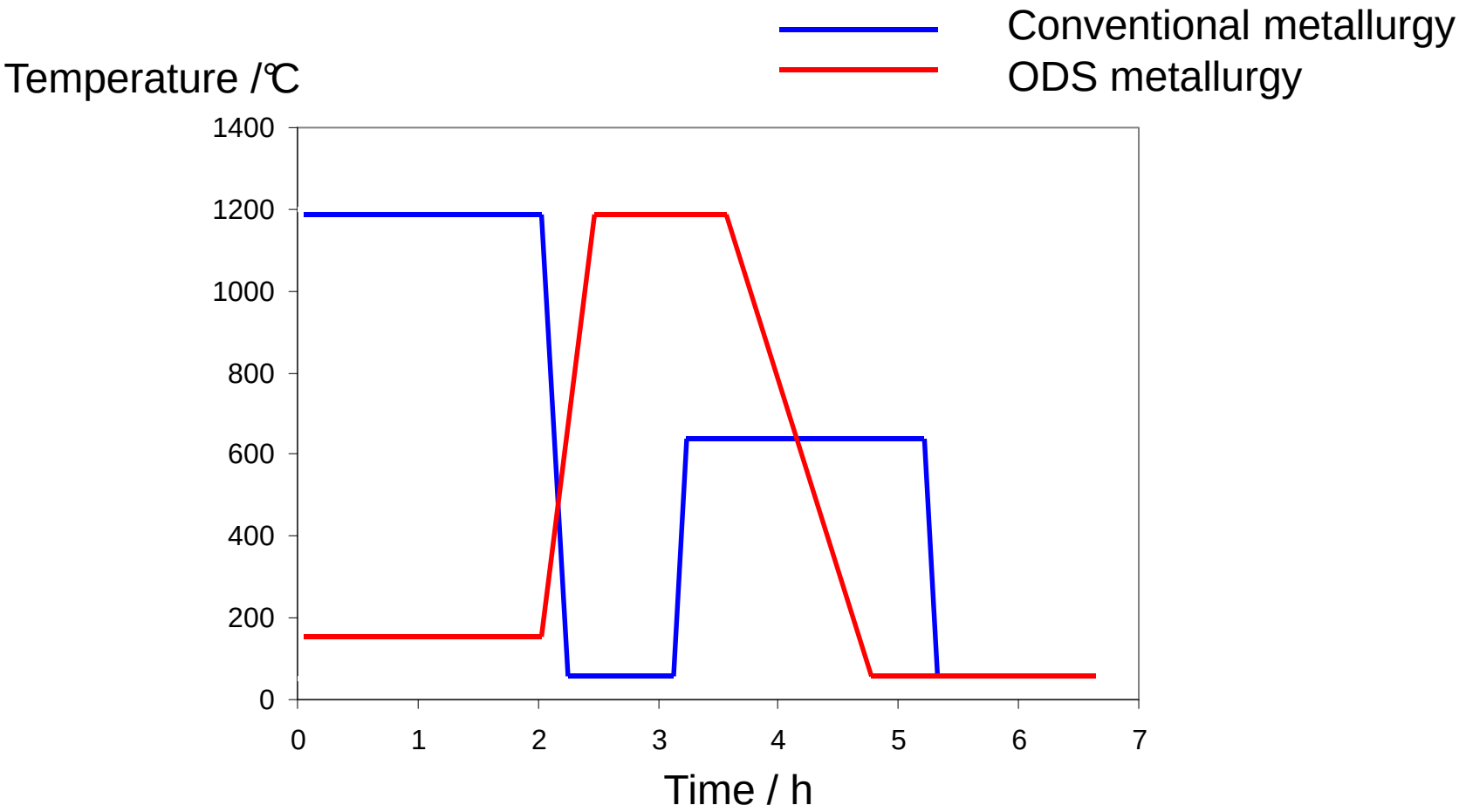


TAP



GPM Rouen
O. Kalokhtina, B. Radiguet, Ph. Pareige

METALLURGY OF ODS MATERIALS



METALLURGY OF ODS MATERIALS

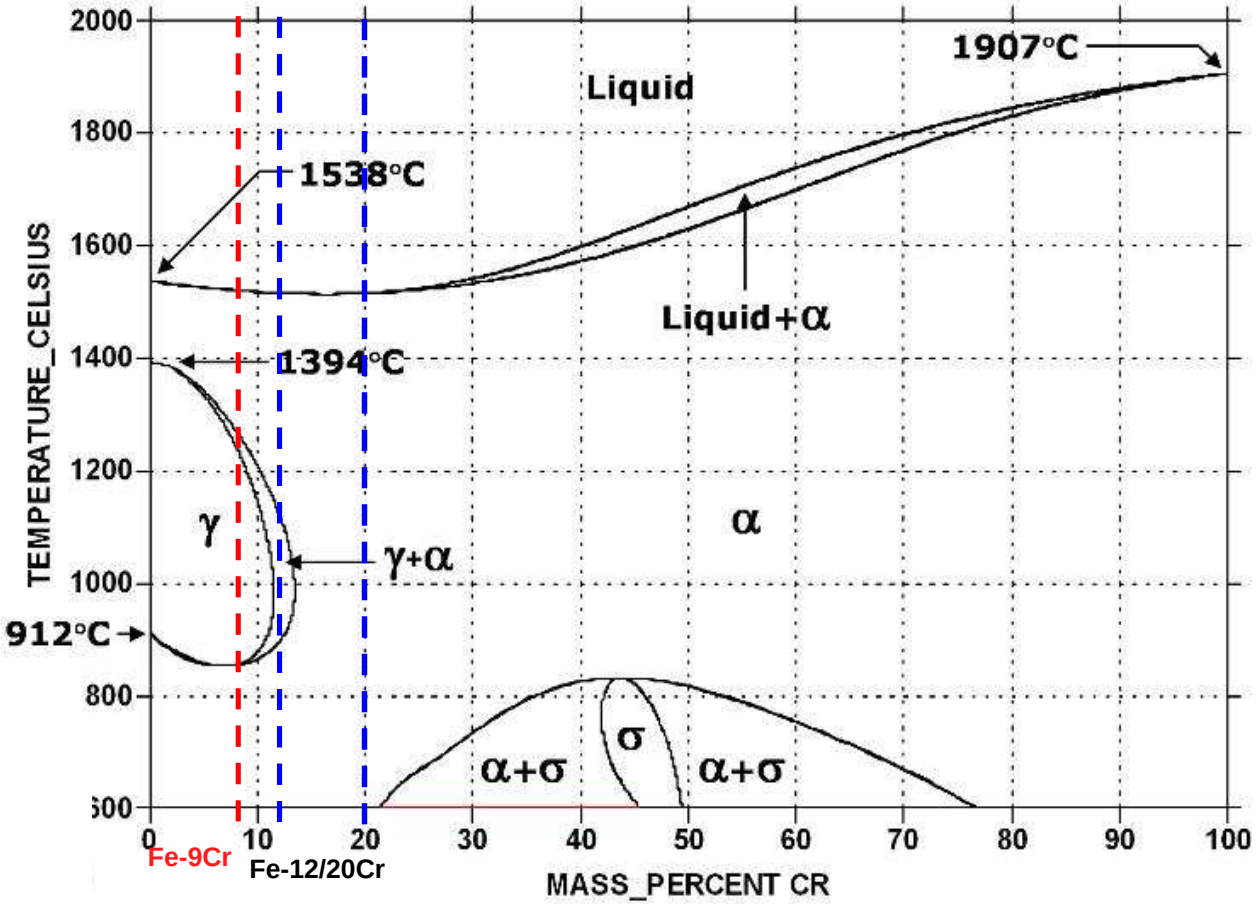
MINOS

- Ferritic grades

Fe -12/20Cr

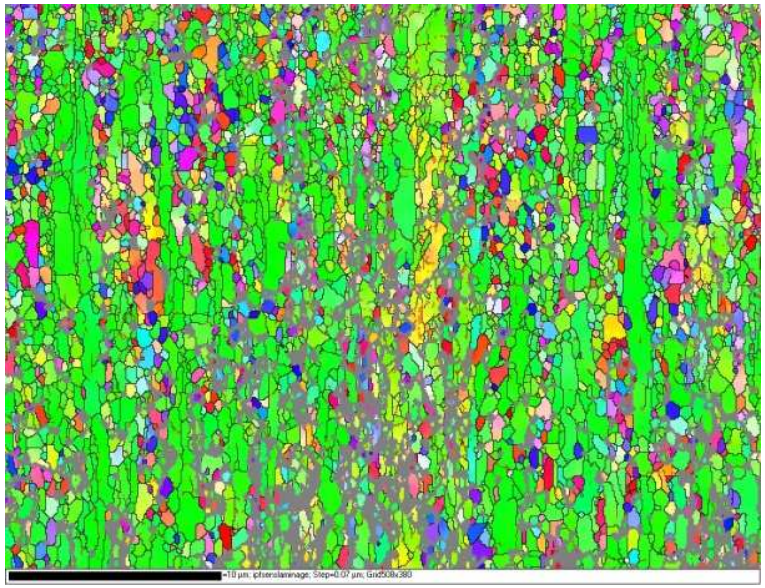
- Martensitic grades

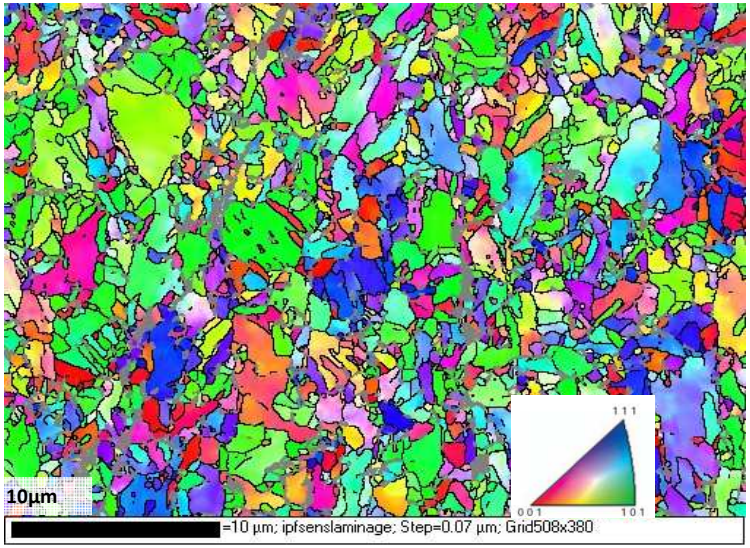
Fe - 9 Cr



Large differences between both type of alloys as a function of their final microstructure but generic creep behavior is noticed.

Typical microstructures





<u>Ferritic Fe-14Cr ODS :</u>
Extruded at 1100℃ + 1h at 1050℃ (very difficult to recrystallize)

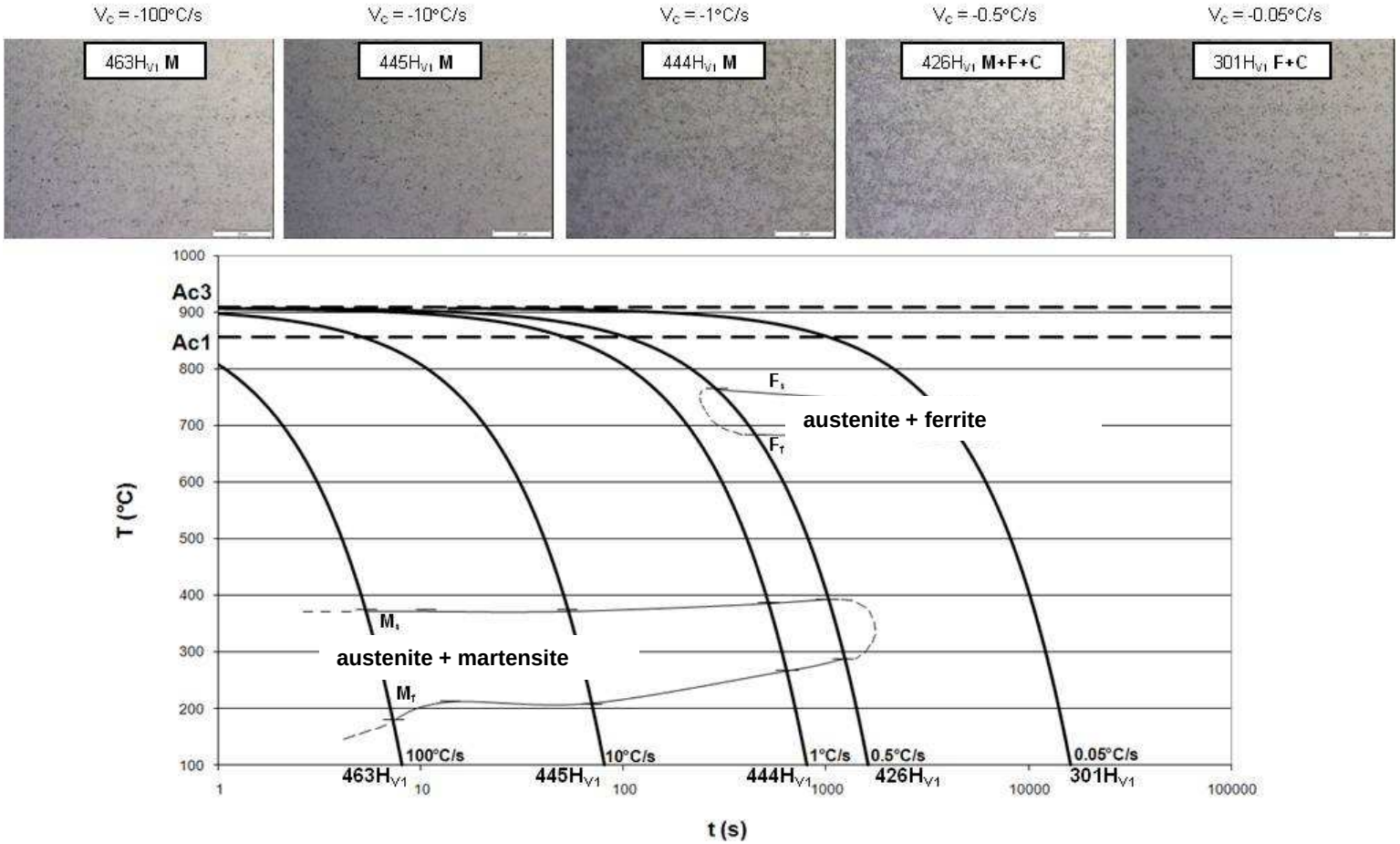
L. Toualbi, Phd Thesis

<u>Martensitic Fe-9Cr ODS :</u>
Tempered martensite
(very high critical cooling rates)

METALLURGY OF ODS MATERIALS

Martensitic grade: Fe-9Cr-1W-0.2Ti-0.3Y_2O_3

- CCT diagram
  - Transformation temperatures
  - Critical cooling rates ~ 1°C/s



L. Toualbi, Phd Thesis

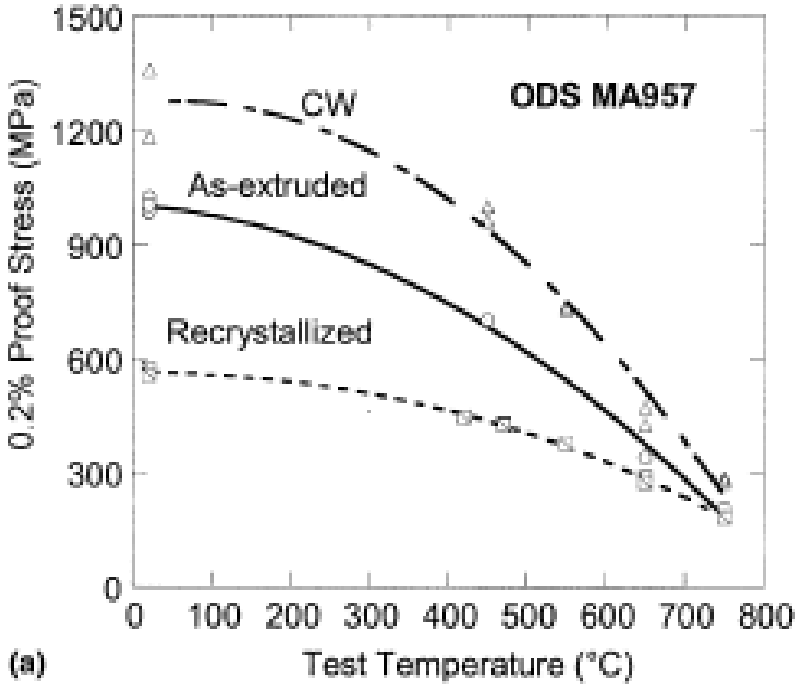
MECHANICAL PROPERTIES OF ODS

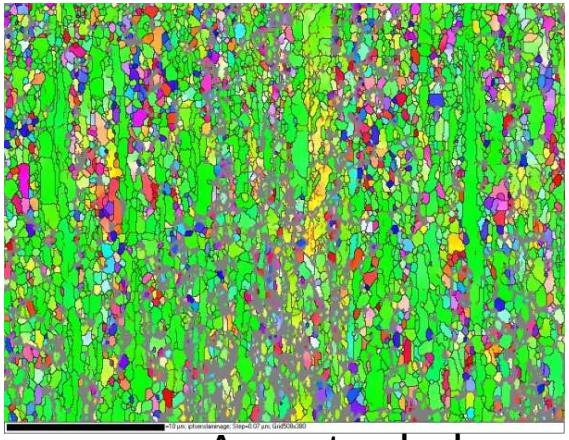
- Tensile properties
- Creep properties
- Impact properties

TENSILE PROPERTIES OF ODS ALLOYS

MINOS

- **Ferritic (Fe-14 Cr ODS)**





As-extruded

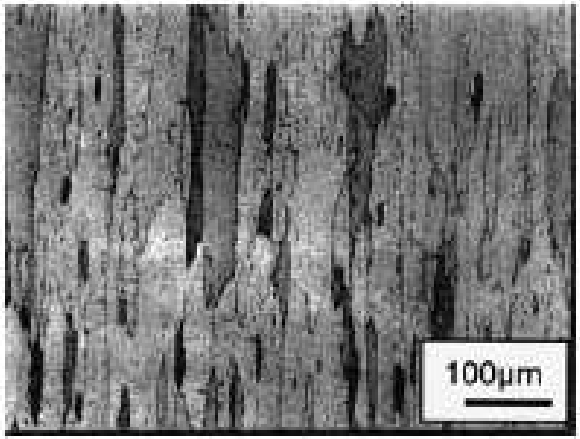
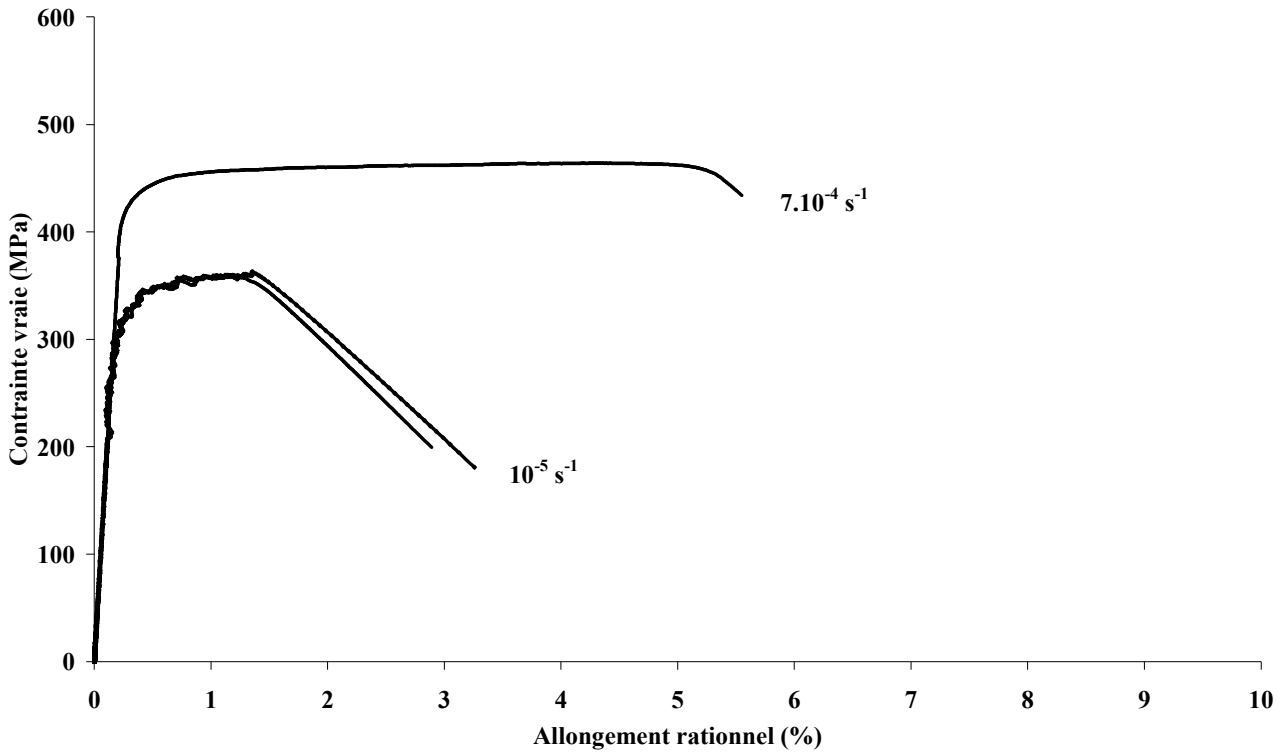


Fig. 1. Morphology of MA957 recrystallized grains obtained after deformation by cold-drawing and recrystallization heat-treatment at 1100 °C.

A. Alamo et al. Journal of Nuclear Materials 329–333 (2004) 333–337

TENSILE PROPERTIES OF ODS ALLOYS

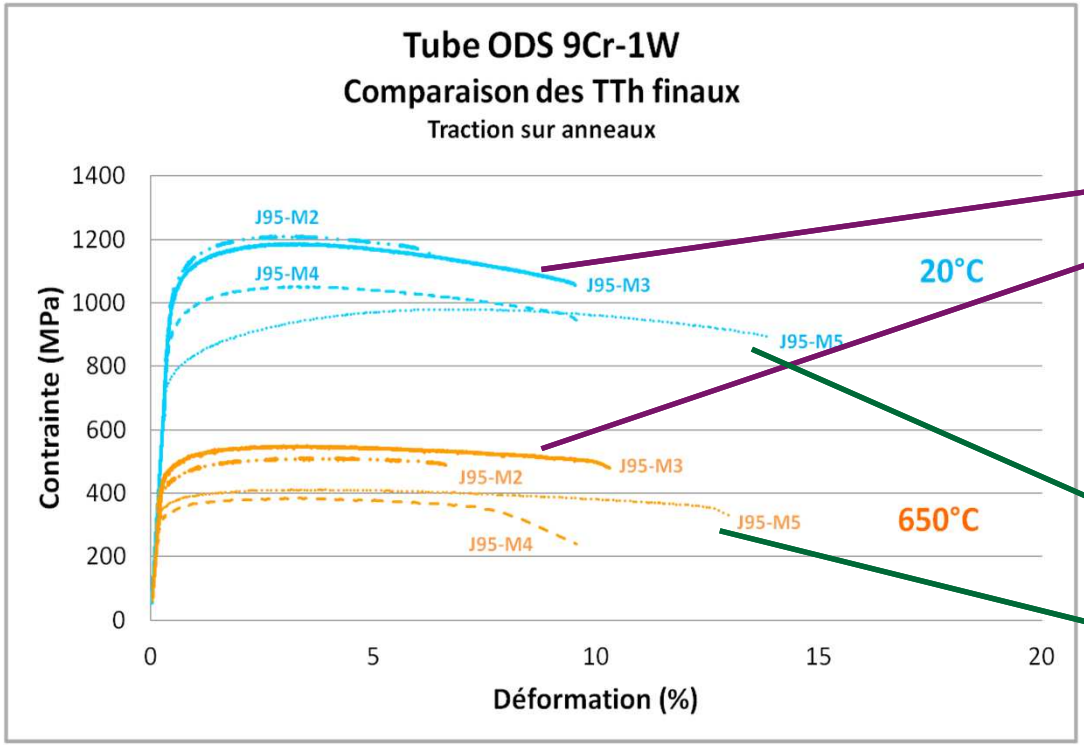


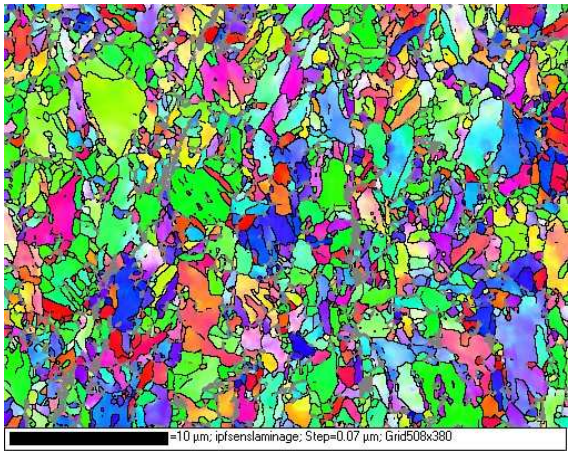
Extruded and annealed ferritic ODS (Fe-18Cr), tensile test at 650℃ in the transvers direction, large decrease of the ductility when the deformation rate is decreasing.

M. Ratti, PhD thesis

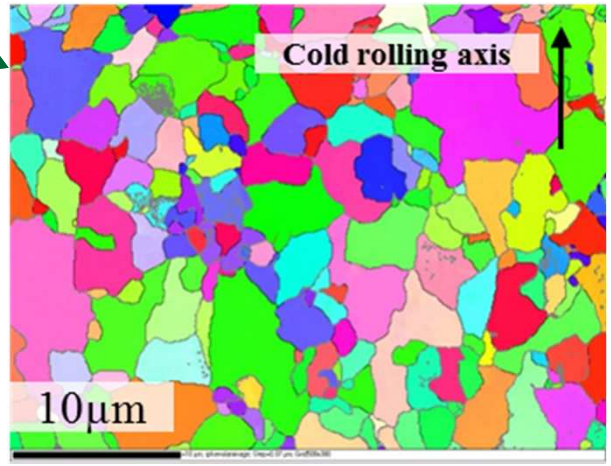
TENSILE PROPERTIES OF ODS ALLOYS

- **Martensitic (Fe-9Cr ODS)**





Tempered martensite



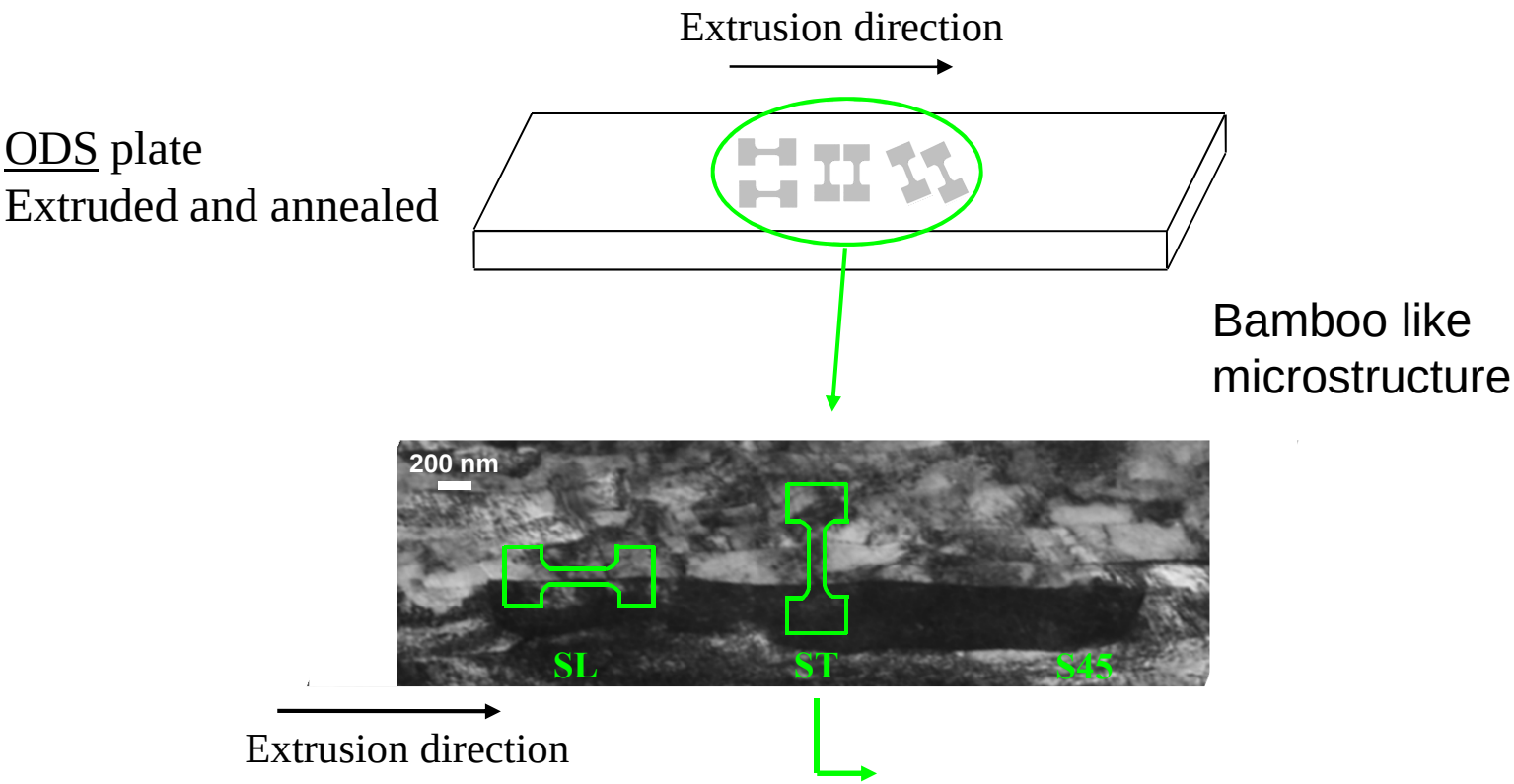
Softened ferrite

Tensile properties in different metallurgical state :
- J95-M3, tempered martensite
- J95- M5, Softened ferrite

L. Toualbi, Phd Thesis

CREEP PROPERTIES OF ODS ALLOYS

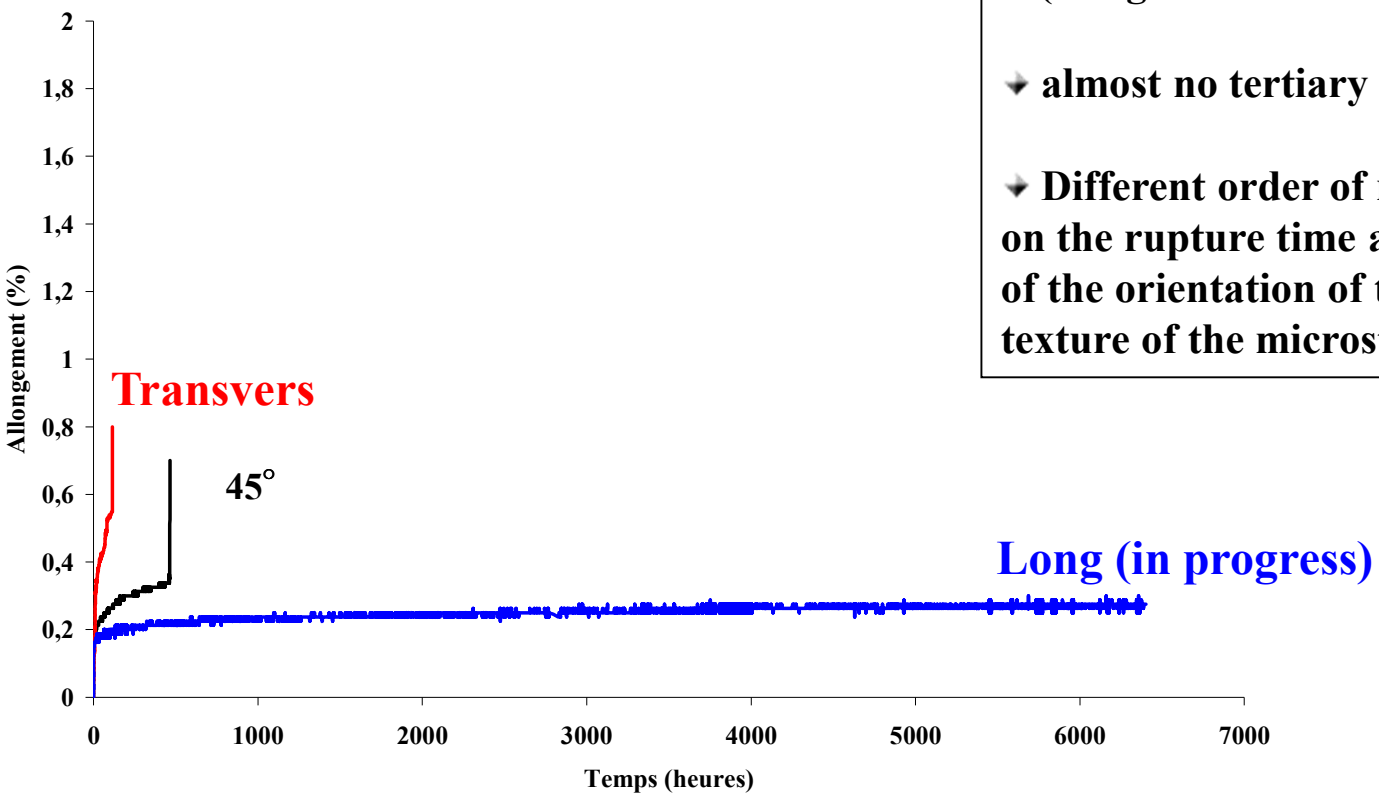
- **Ferritic (Fe-18 Cr ODS)**



M. Ratti, Phd Thesis

CREEP PROPERTIES OF ODS ALLOYS

- **Ferritic Fe-18 Cr ODS at 650℃ 250 MPa**



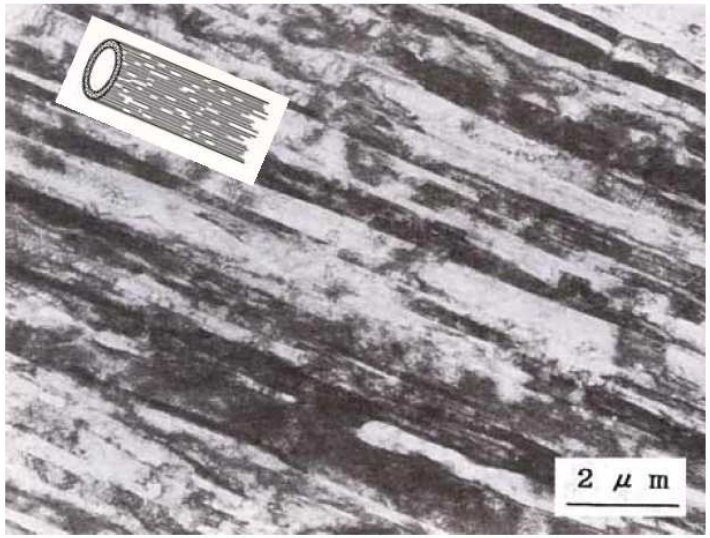
- Very low deformation rates (Long-250 MPa : 7.10^{-8} h^{-1}).
- almost no tertiary domain .
- Different order of magnitude on the rupture time as a function of the orientation of the sample / texture of the microstructure

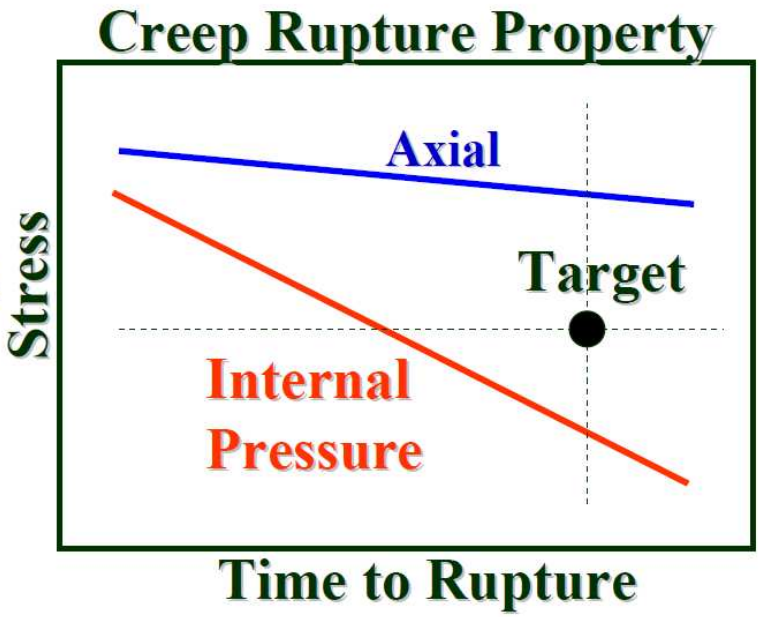
M. Ratti, Phd Thesis

Grain Morphology Anisotropy Results in Poor Creep Rupture Strength under Internal Pressure

Finely elongated grain structure along rolling direction results in

- degradation of internal creep rupture strength and
- significant loss of ductility around 673 K



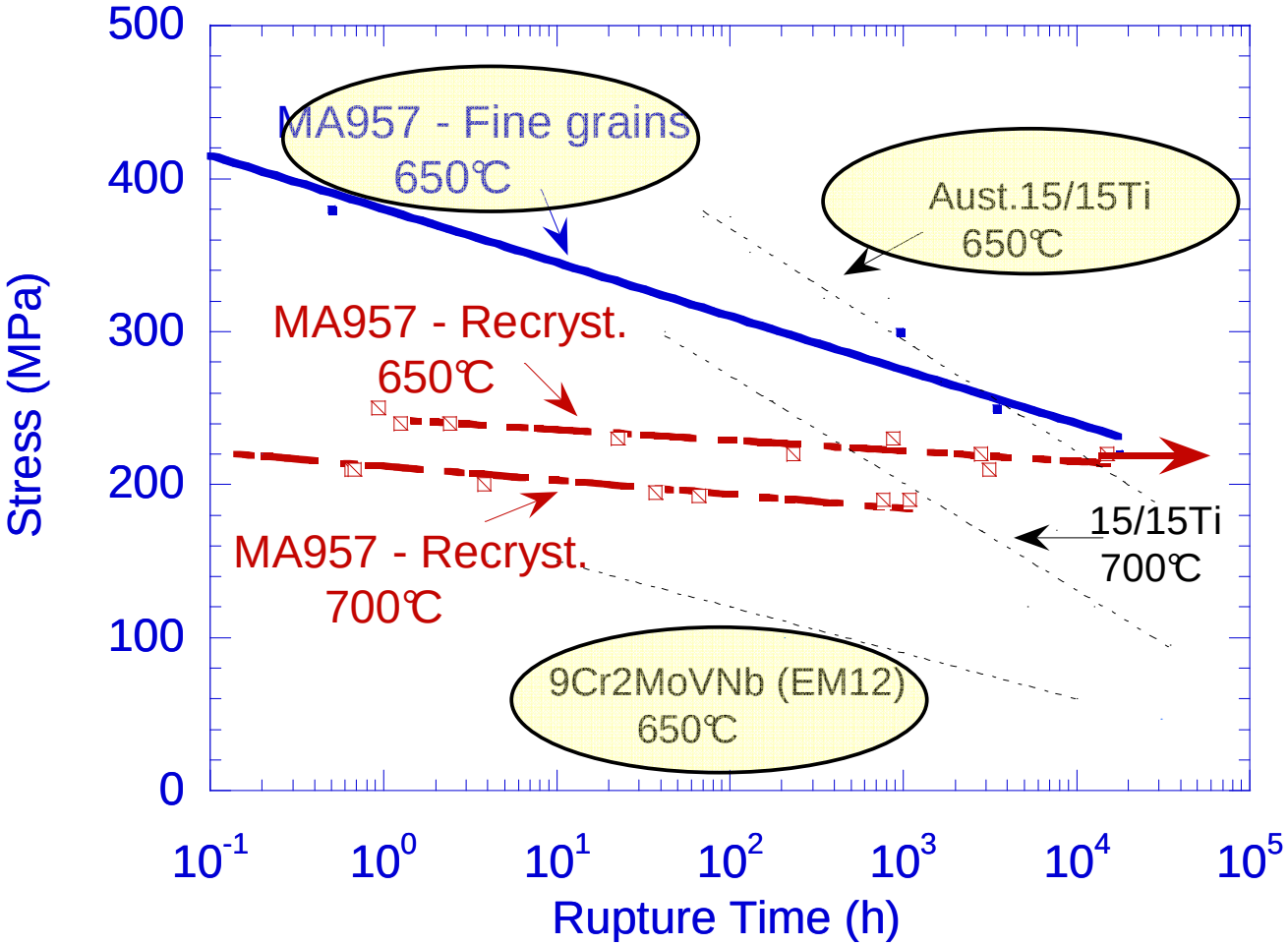


Inoué et al.

CREEP PROPERTIES OF ODS ALLOYS

MINOS

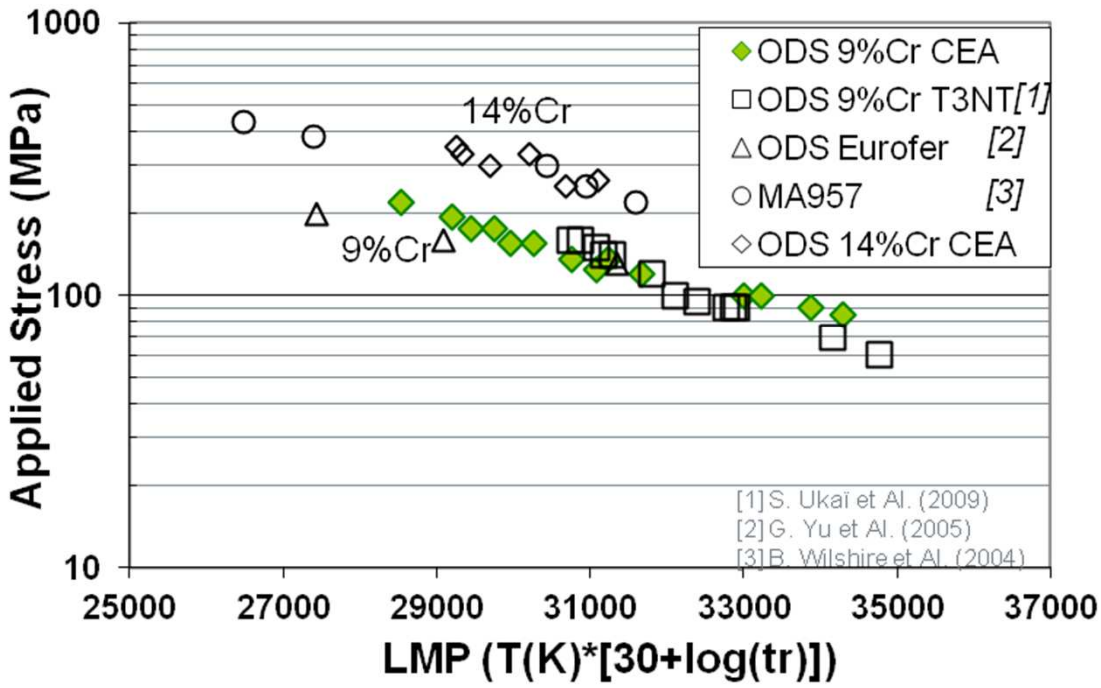
- **Ferritic ODS**



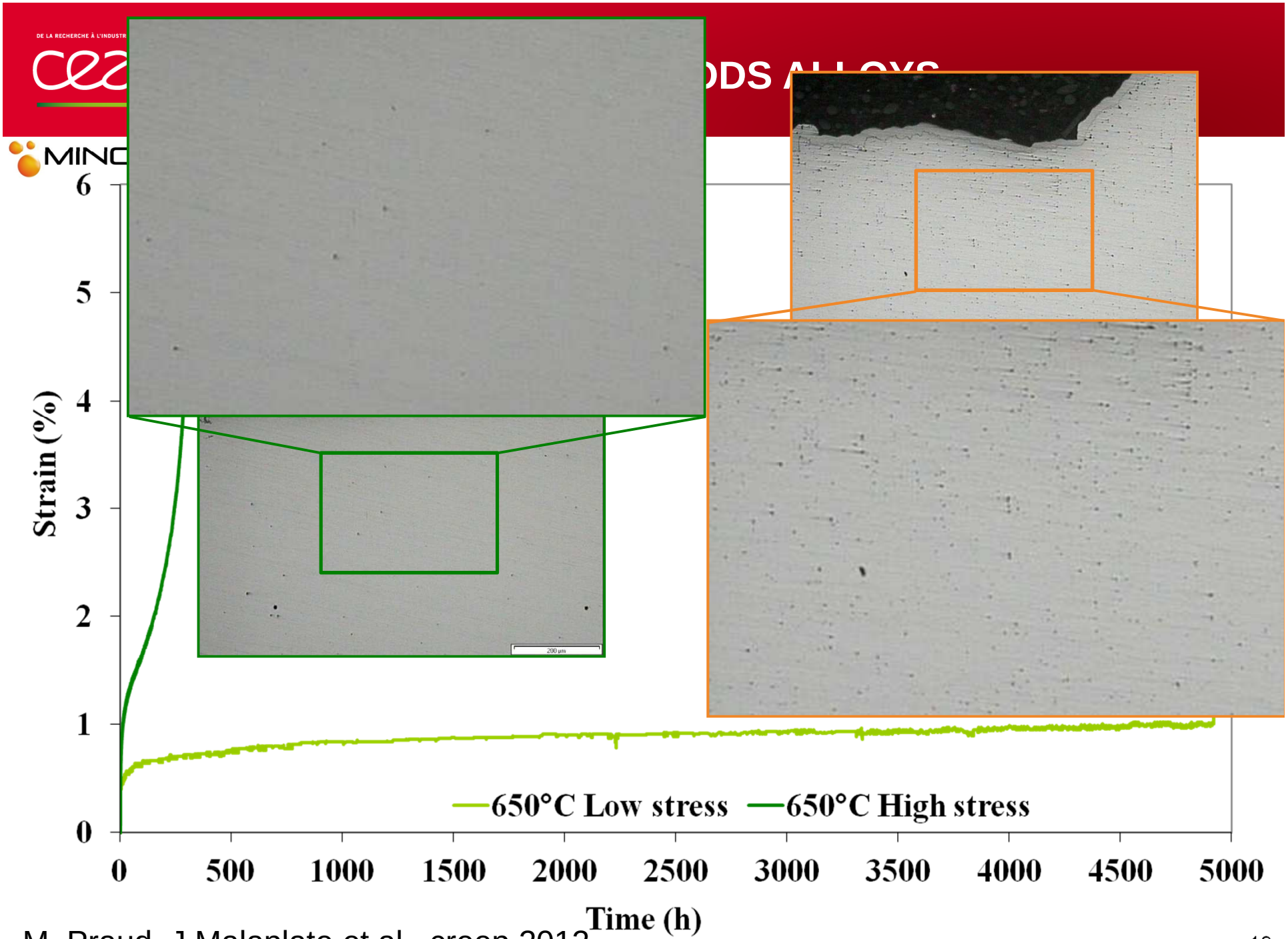
A. Alamo et al. Journal of Nuclear Materials 329–333 (2004) 333–337

CREEP PROPERTIES OF ODS ALLOYS

Higher performances of Fe-14Cr ODS alloys (fine grains) in the longitudinal direction compared to Fe-9Cr ODS (softened ferrite)



M. Praud, J.Malaplate et al., creep 2012



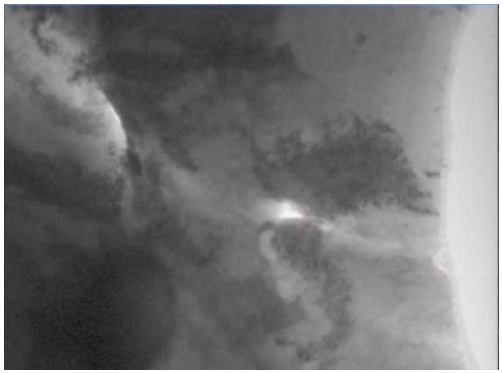
M. Praud, J.Malaplate et al., creep 2012

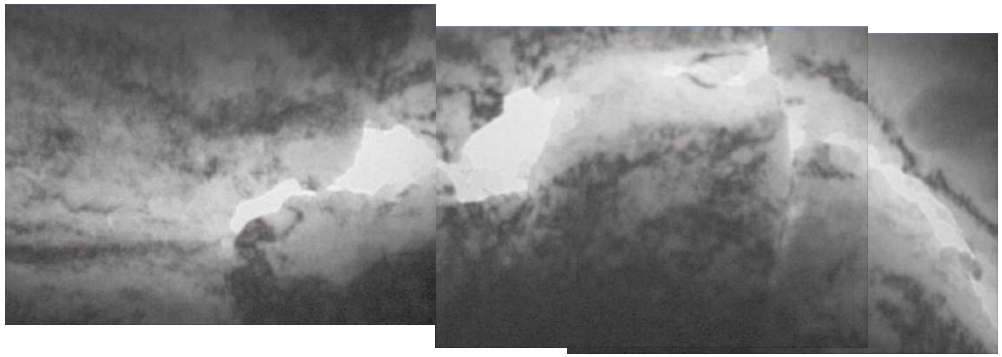
CREEP PROPERTIES OF ODS ALLOYS

In situ relaxation tests at 550°C

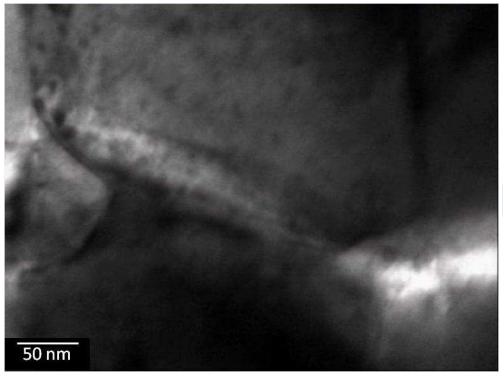
- **Creation of cavities**
- **Decohesion along the grain boundaries**

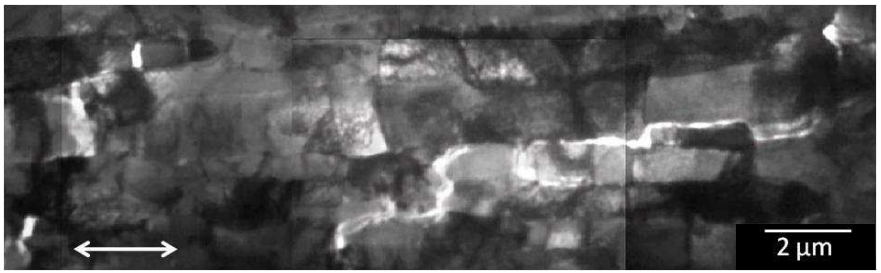
ODS 9%Cr





ODS 14%Cr





INTERGRANULAR mechanism

Consistent with the macroscopic observations

M. Praud, J.Malaplate et al., creep 2012

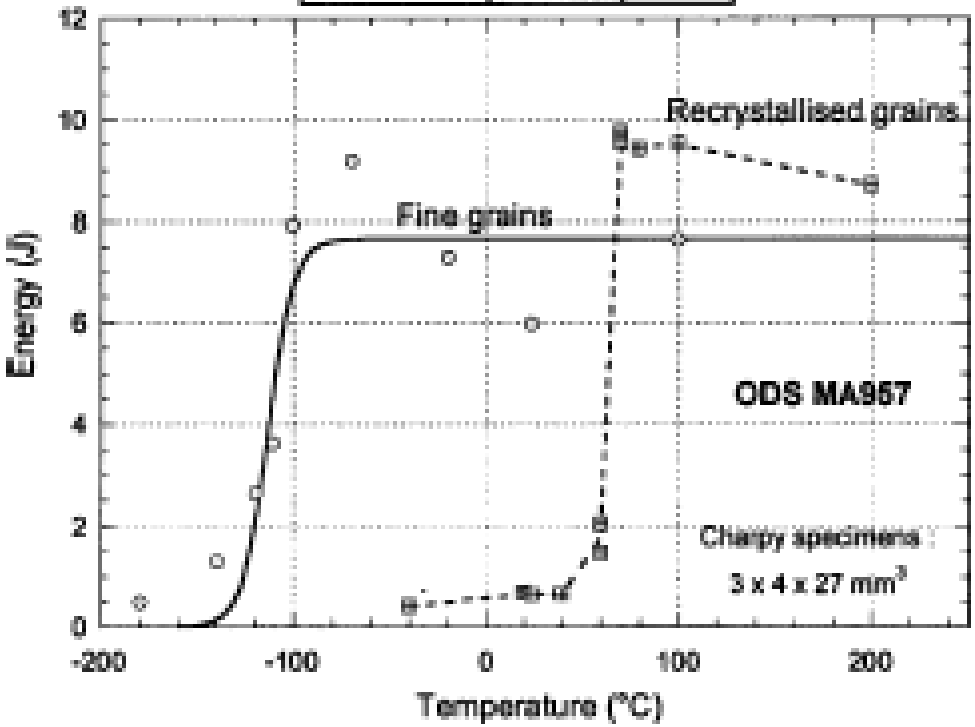
- **Dislocation still pinned on oxides**
- **Viscous motion**
- **Activation of sources of dislocations at the grain boundary**

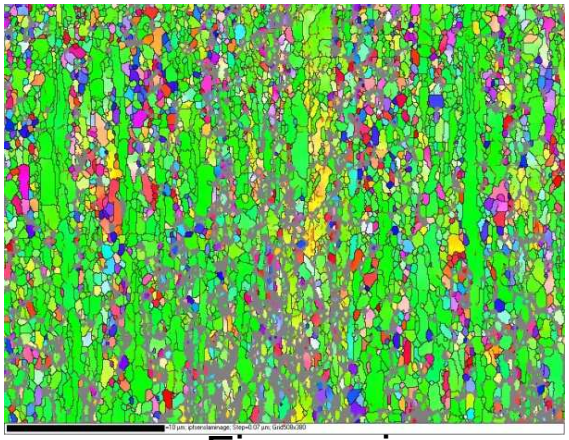
IMPACT PROPERTIES OF ODS ALLOYS

MINOS

- **Ferritic Fe-14 Cr ODS alloys**

MA957	DBTT (°C)	USE (J)
Fine Grains	- 110	7.7
Recrystallised	+ 60	9.2





Fine grain

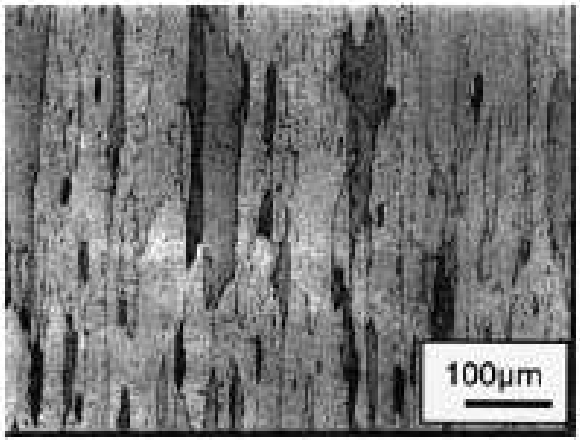
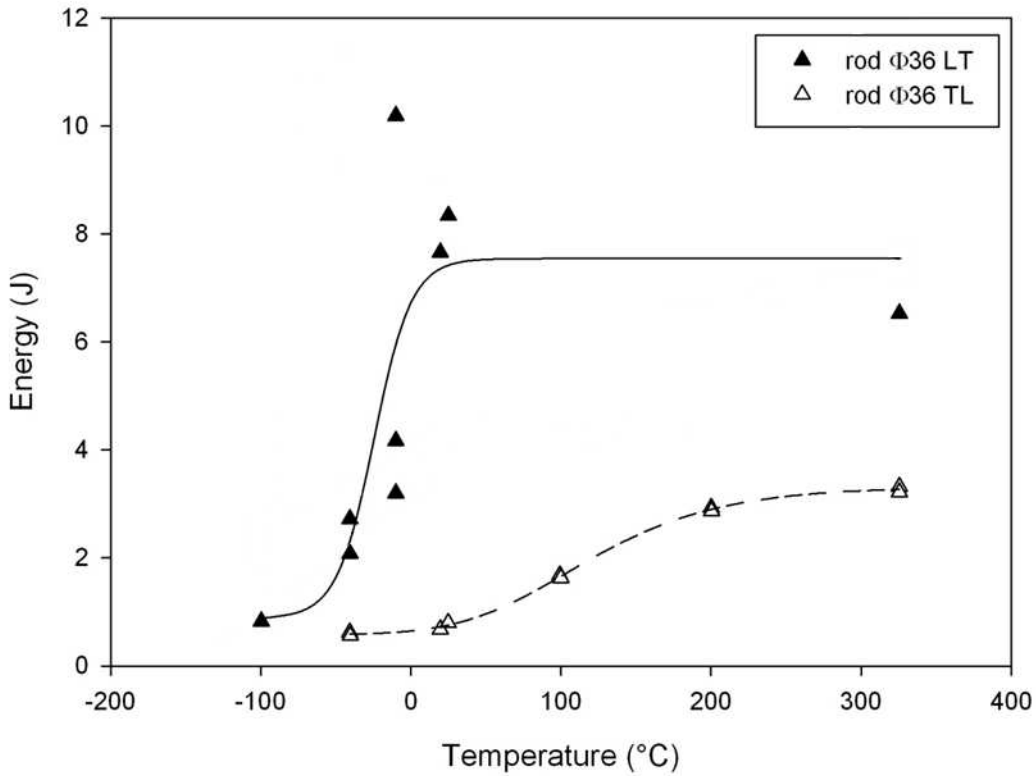


Fig. 1. Morphology of MA957 recrystallized grains obtained after deformation by cold-drawing and recrystallization heat-treatment at 1100 °C.

A. Alamo et al. Journal of Nuclear Materials 329–333 (2004) 333–337

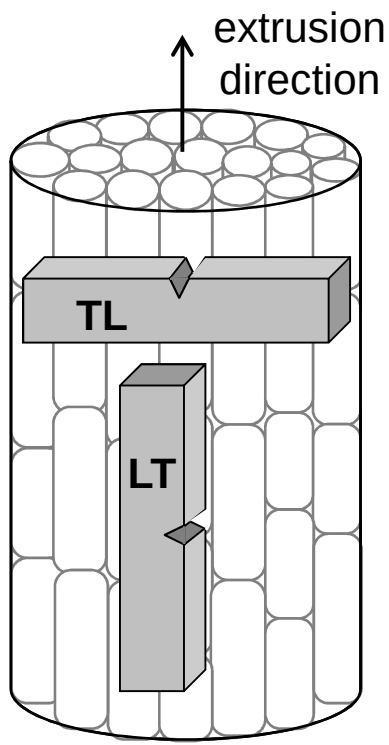
IMPACT PROPERTIES OF ODS ALLOYS

Ferritic Fe-14 Cr ODS alloys



	DBTT (℃)		USE (J)	
	LT	**TL**	**LT**	**TL**
rod Ø36	**-29**	**108**	**7.6**	**3.8**

- **anisotropic** impact behaviour for the **rod**: higher USE and lower DBTT under LT loading



diameter: 36mm

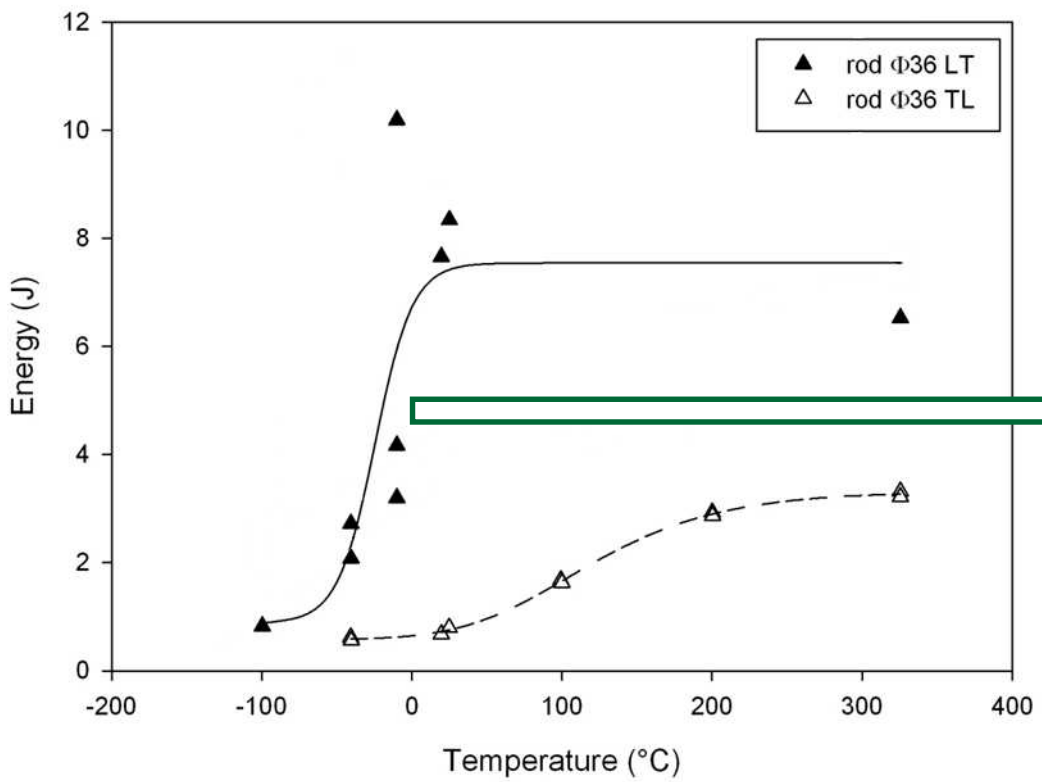
Fe-14Cr 1W ODS "Fine grain microstructure"

AL Rouffié et al. http://dx.doi.org/10.1016/j.jnucmat.2012.08.050

IMPACT PROPERTIES OF ODS ALLOYS

MINOS

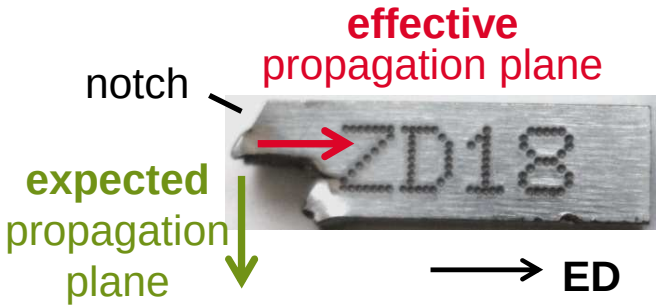
- **Ferritic Fe-14 Cr ODS alloys**



- **anisotropic** impact behaviour for the **rod**: higher USE and lower DBTT under LT loading

	DBTT (℃)		USE (J)	
	LT	**TL**	**LT**	**TL**
rod Ø36	**-29**	**108**	**7.6**	**3.8**

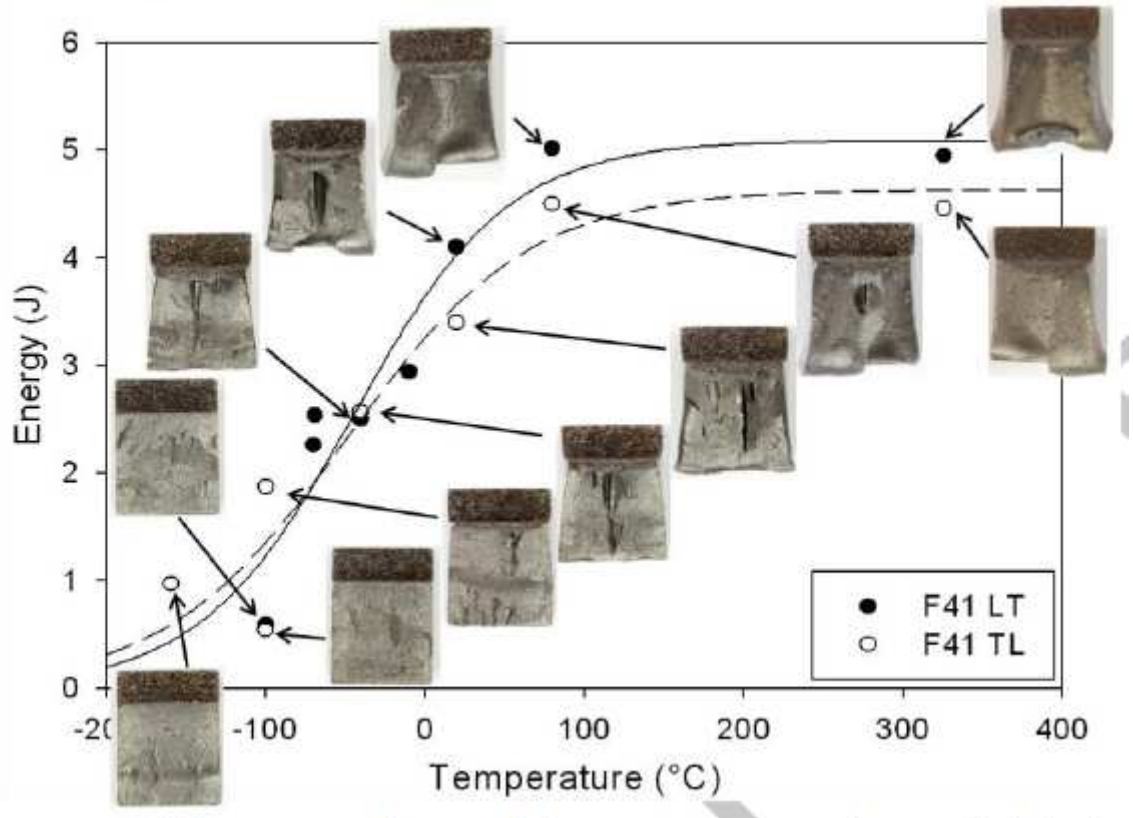
For different samples, tendency to delamination



AL Rouffié et al. http://dx.doi.org/10.1016/j.jnucmat.2012.08.050

IMPACT PROPERTIES OF ODS ALLOYS

- **Ferritic Fe-9Cr ODS alloys (thick plate)**



Quasi-isotropic behaviour

AL Rouffié et al. http://dx.doi.org/10.1016/j.jnucmat.2012.08.050

CONCLUSIONS

The mechanical properties of nuclear F/M alloys depend strongly on the chemical composition and the microstructure of the product

Tensile properties

- Large dependence on the grain size / morphology / dislocation density / nano-precipitation
- Collapse of the strength from 400℃
- Strain rate effect on the ductility

Creep properties

- No tertiary creep but high performances
- Reduced fracture strain
- Damage more severe at high temperature low strain rate / low stress => **Intergranular mechanism of failure**

Impact properties

- Low toughness and in general “low” Upper Shelf Energy and sometimes tendency to delamination
- Very important impact of the microstructure (morphological texture) on the impact properties

The fabrication route of ODS materials with the appropriate microstructure is a key issue for the nuclear applications