Mechanical Properties of ultrafine-grained Iron processed by HIP

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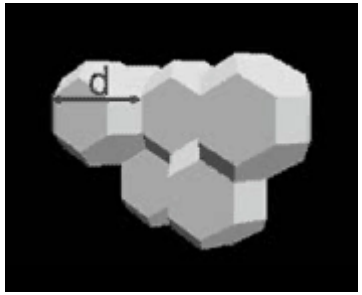
- [1]Standard polycrystals *vs* nanograined polycrystals: what do we know, what do we measure?

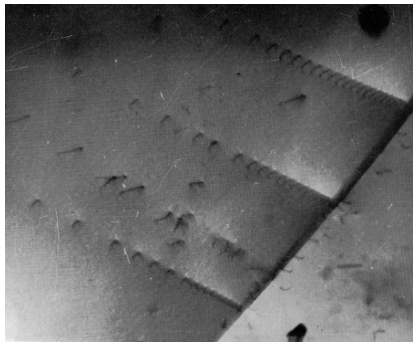
[1] Collaboration: G. Saada. LEM, ONERA, Châtillon

- The general principles underlying the processing of high strength, and ductile, metallic materials have been understood since the beginning of the development of dislocation theory.

- Ductility necessitates a reasonable mobility of the dislocations, strengthening is related to the building of obstacles restricting propagation of dislocations.

- These obstacles (dislocations, atoms in solid solution, second phase inclusions, grain boundaries) are introduced by combining alloying (structural hardening), plastic flow (strain hardening) and **grain size refinement**.

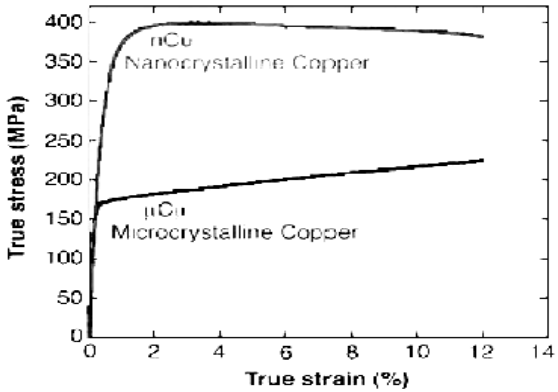




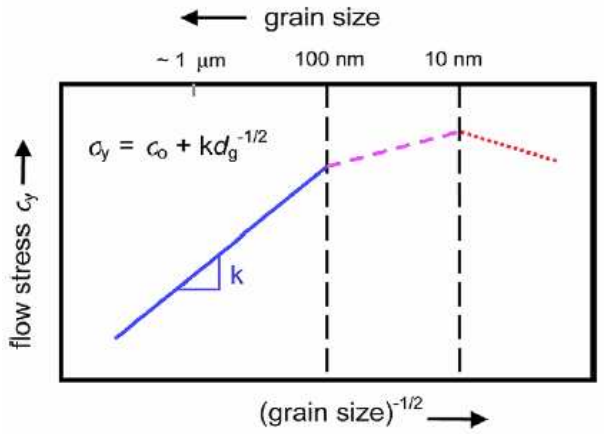
Dash et al. (1956)

Hall – Petch Law: $$\sigma = \sigma_0 + \frac{k}{\sqrt{d}}$$

- Down to very small grained materials, one expects an increase in strength, which has been observed in various circumstances .



(Champion et al. Science **300**, 310 (2003)



Kumar et al. Acta Mater., 51 (2003) 5743-5774)

Various mechanical tests on these products have revealed indeed a remarkable increase of strength, at the expense of ductility. But the measured values of the yield stress significantly differ from those obtained by extrapolating the HPL formula

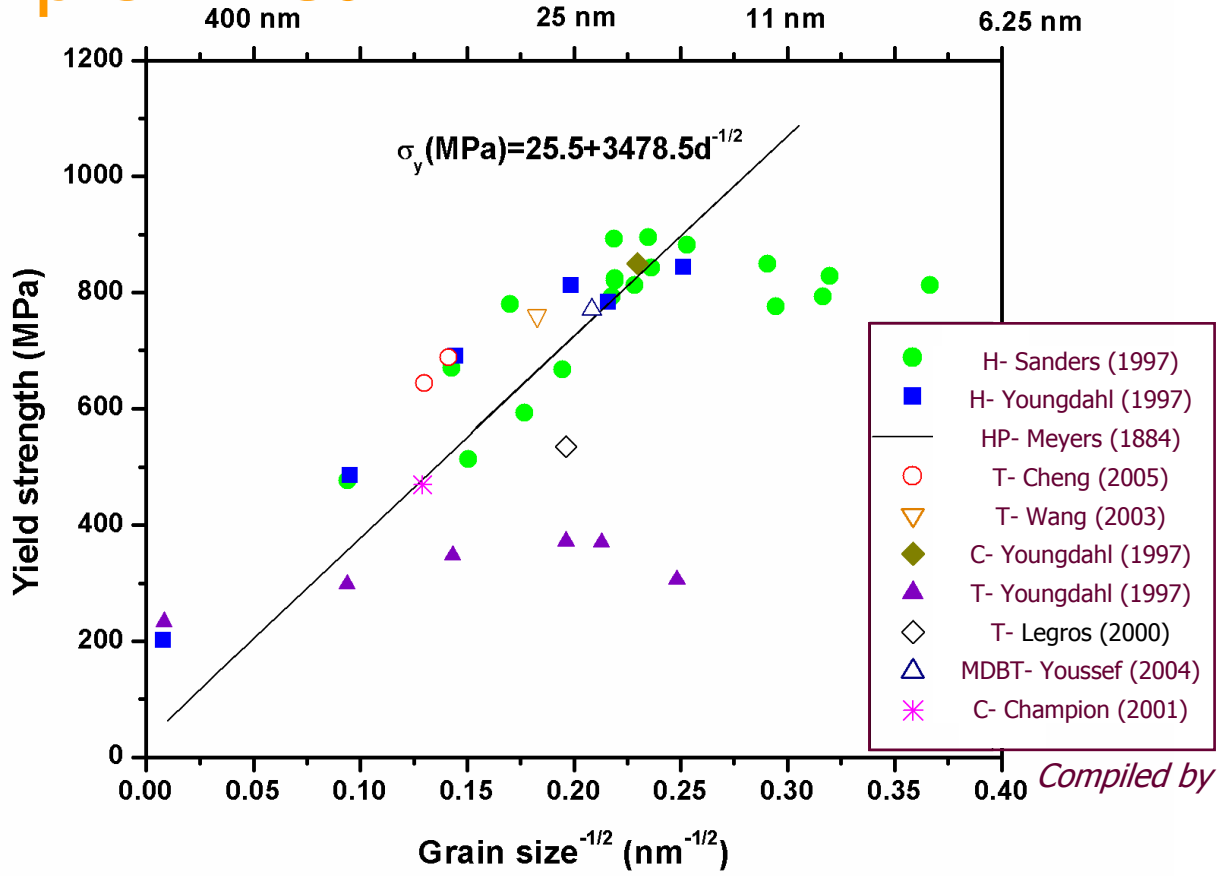
$$\sigma = \sigma_0 + \frac{k}{\sqrt{d}}$$

- This has stimulated the processing of polycrystals of smaller and smaller grain size, which have been classified, with respect to their grain size, as:
  - *microcrystalline* (*mc*) if d >1000nm,
  - *ultra fine crystalline* (*ufc*) if 100nm<d<1000nm,
  - and *nanocrystalline* (*nc*) if d <100nm
- Processing methods include: powder compaction, electro-deposition, SPD…

- Our present understanding of the deformation mechanisms of *standard polycrystals* depends on:
  - the production of specimens with well designed and characterized microstructures.
  - the gathering of consistent and systematic data through mechanical tests over a wide range of strain rates and in a large temperature interval.
  - A thorough observation of the evolution of the microstructure during mechanical testing.
  - Establishing a consistent correlation of the mechanical behavior with the microstructure by a relevant theoretical analysis.

- As far as *nano grained polycrystals* are concerned, today's situation is characterized by:
  - *Limited control and description of the as prepared microstructure*
  - *Incomplete information on the evolution of the microstructure*
  - *Inaccuracy of the measurement of the relevant mechanical parameters*
  - *Inadequate theoretical interpretation*

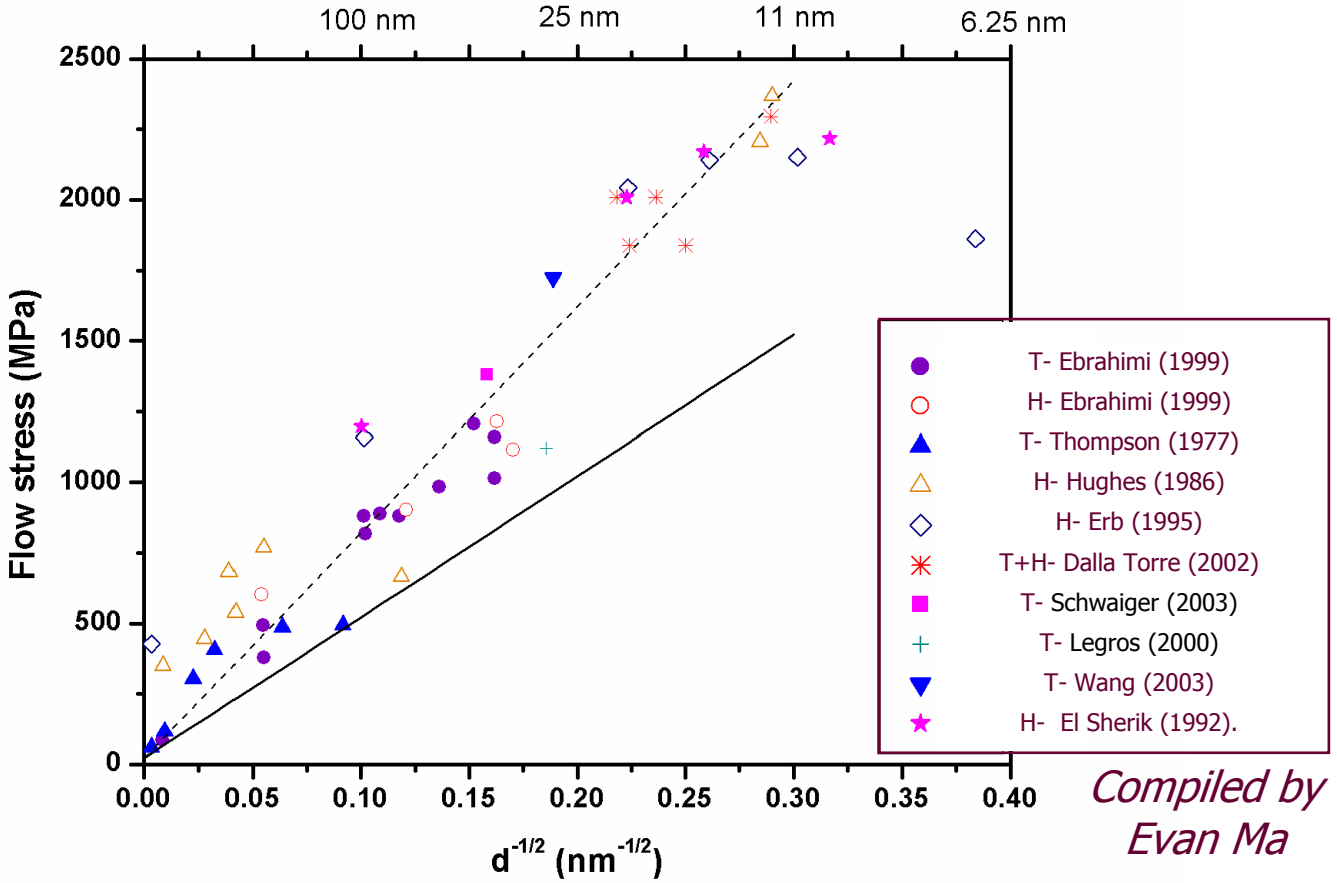
Example 1: Cu



Compiled by Evan Ma

In the case of *sp*, there is a general, and justified agreement on the relevance of the identification of the 0.2% proof strain flow stress σ_{02} to the yield stress σ_y, as well as the latter to a fraction, generally 1/3, of the Vickers hardness H_V.

Example 2: Ni



Compiled by Evan Ma

Various mechanical tests on these products have revealed a remarkable increase of strength. But the measured values of the yield stress significantly differ from those obtained by extrapolating the HPL formula

Example 3

The smallness of the grain size of *ngp* imposes very strong constraints. This imposes to revisit the meaning of concepts such as elastic-plastic transition, macroscopic and microscopic yield stress,…

$$\varepsilon_M > \frac{b}{d}$$

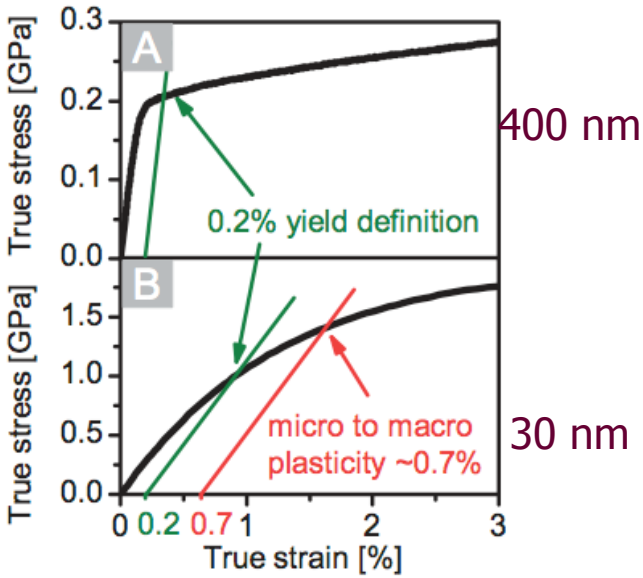
At ε_M = 0.2% ➔ d > 500b ≈125nm (b≈0.25nm)

Moreover at 0.2% strain, the fraction of deforming grains is:

$$\frac{n(\varepsilon_M)}{N} \approx \varepsilon_M \frac{d}{b} = 2\times10^{-3}\frac{d}{b}$$

$$d \le 20b \Rightarrow \frac{n}{N} \approx 4\%$$

Saada, Mater. Sci. Eng., 400 (2005) 146-149



Brandstetter et al. Adv. Mater., 16 (2006), 1545-1548

Example 4: deformation mechanisms

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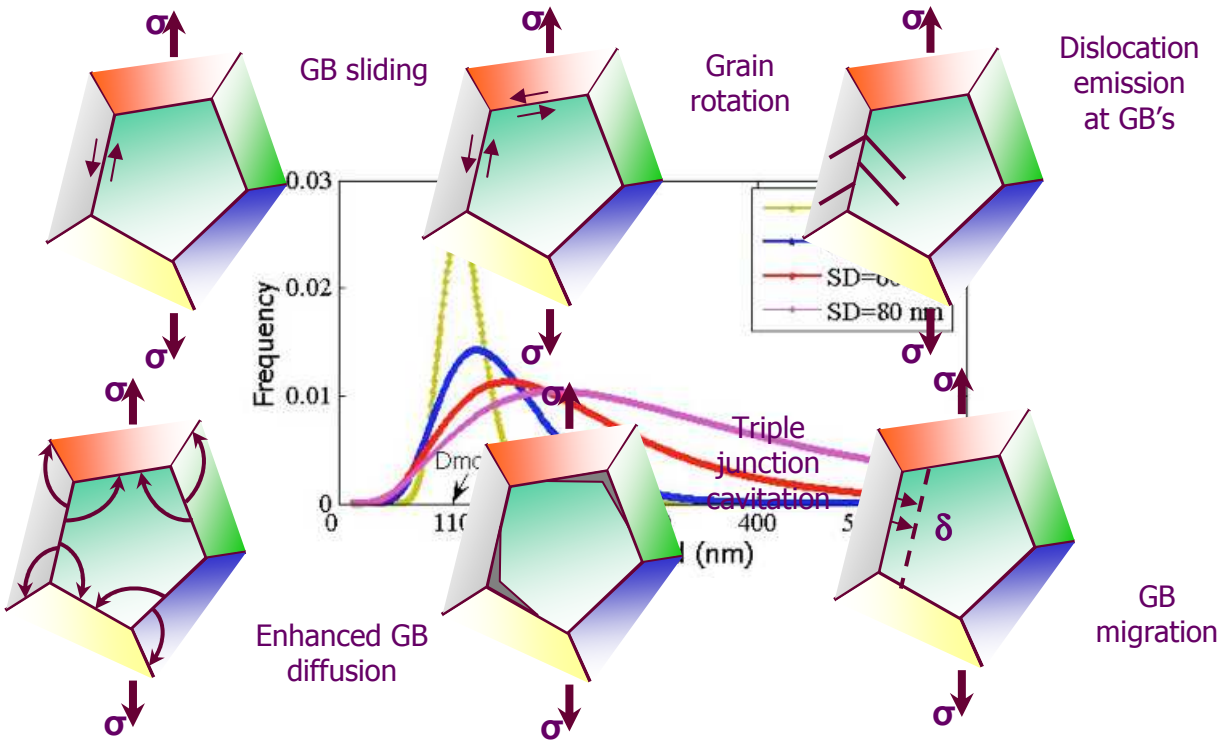
- Samples usually exhibit a Log normal grain sizes distribution
- The observed mechanical behavior is a complex interplay of different processes

Proposed nc-deformation mechanisms



D. Gianola, T. Rupert, K. Hemker "Experiments in grain boundary dominated materials". Braunwald, Switzerland, September 2007.

Since the grain size alone neither defines the microstructure nor characterizes the mechanical behavior of these very small-grained polycrystals, we shall distinguish simply:

- *micro-grained polycrystals* (*mc*) whose grain size is larger than one or a few microns,

- *ultrafine-grained polycrystals* (*ufg*) whose grain size is smaller than one micron

- *HIP processing of ultrafine grained Iron: Mechanical properties

* RNMP AGUF (with ARCELOR)

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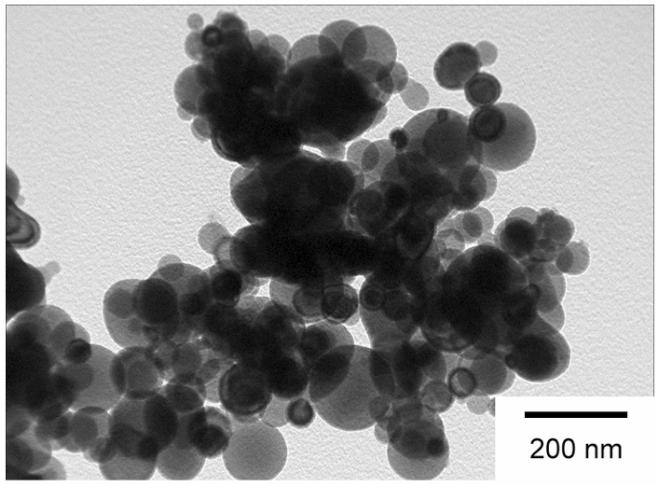
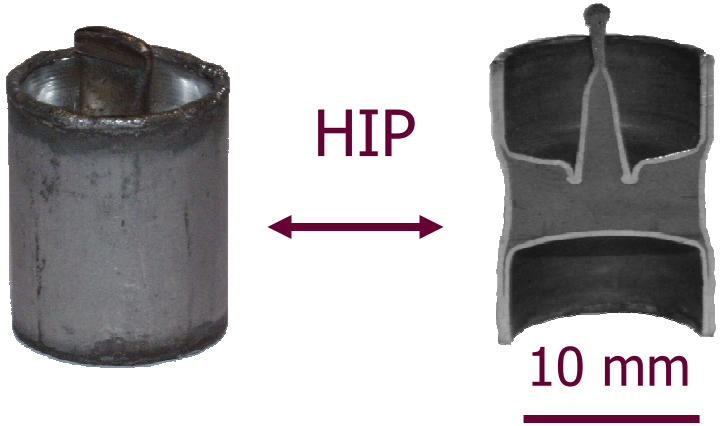
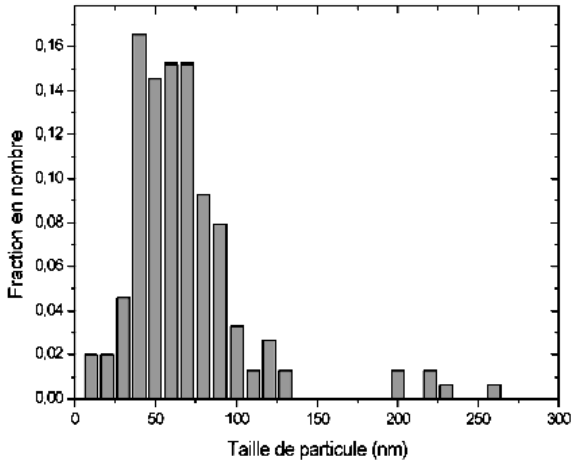
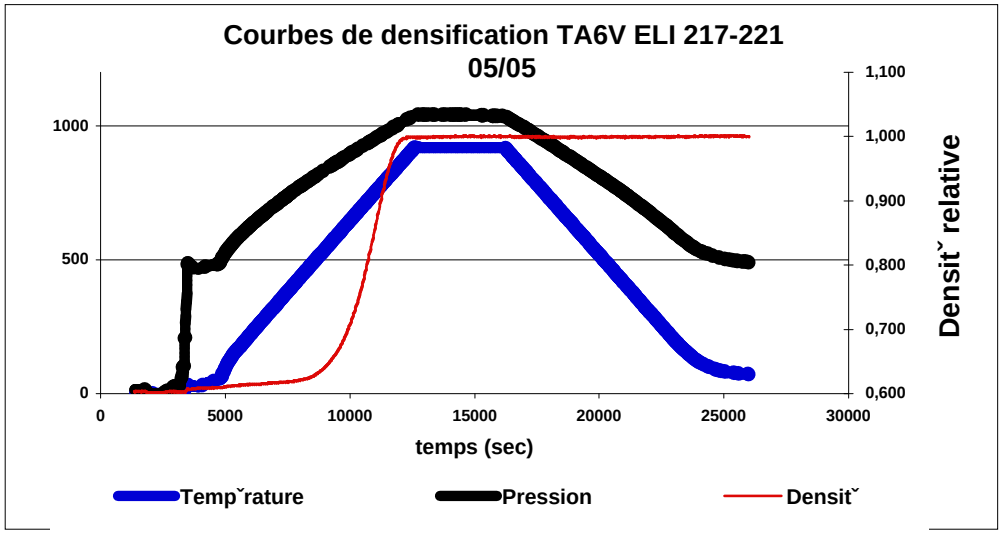
Starting materials and the HIP process









Commercial purity Iron powders

Obtained by EEW from *Argonide Corp.*, USA

Fe, Fe_3O_4...

Stainless container

Pressure applied through *Ar* gas

Optimized HIP parameters for the Fe powder

	Sample ID	Fe200	Fe201	Fe203	Fe205
Oxide reduction at 450°C	Time (h)	3	3+4*0.5	6*0.5	8*0.5
	# steps	-	4	6	8
HIP	**Holding Press. (MPa)**	**120**	**110**	**155**	**200**
	Holding T° (°C)	**800**	**800**	**720**	**620**
	Holding Time (h)	**2.5**	**14**	**1.5**	**2.5**

Compact density > 99

As-processed microstructures

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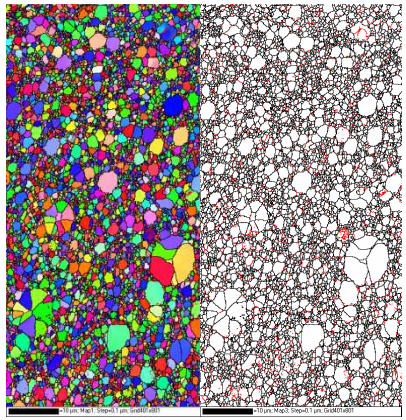
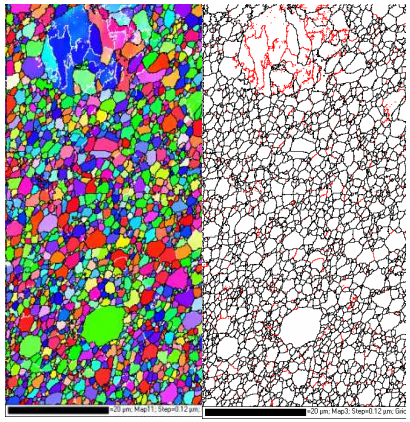
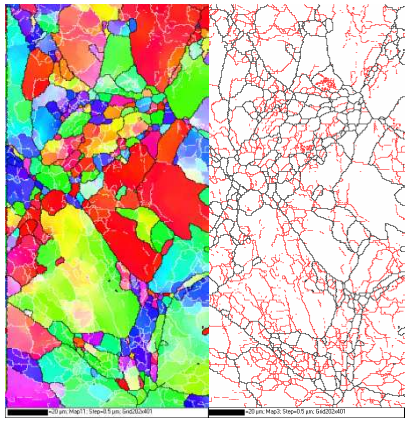
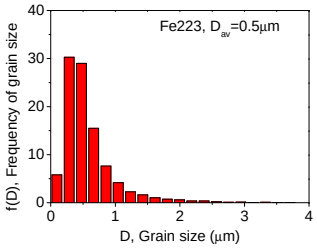
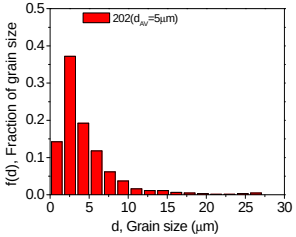


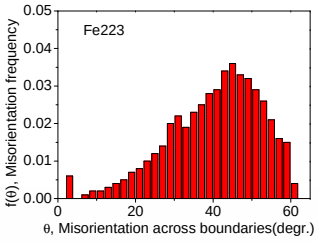


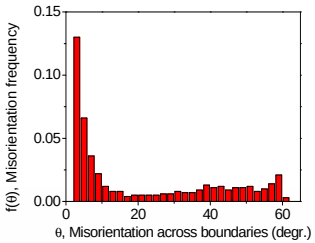


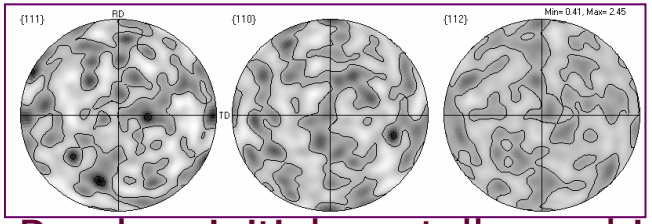
0.5µm 1.0µm 5.0µm



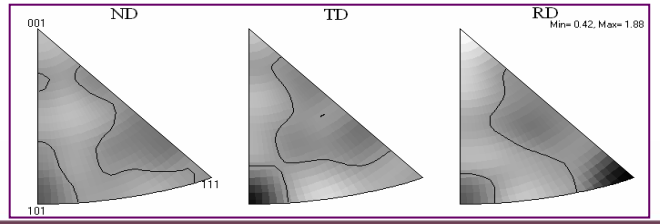




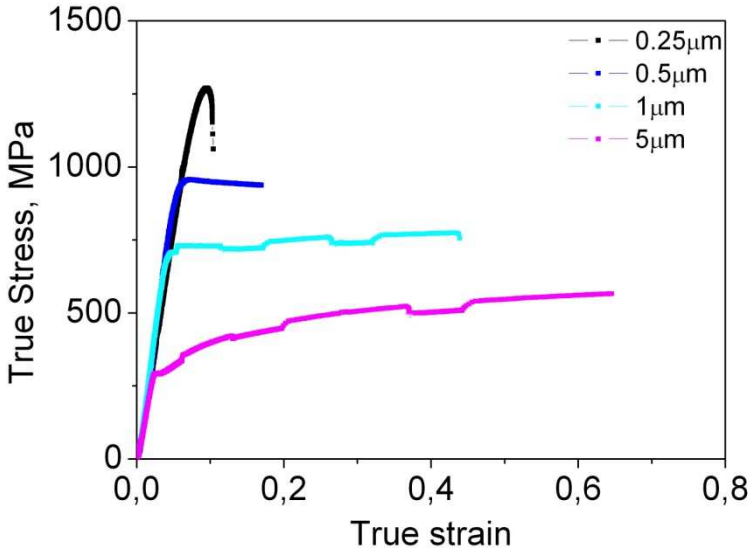




Random initial crystallographic texture



Mechanical behavior



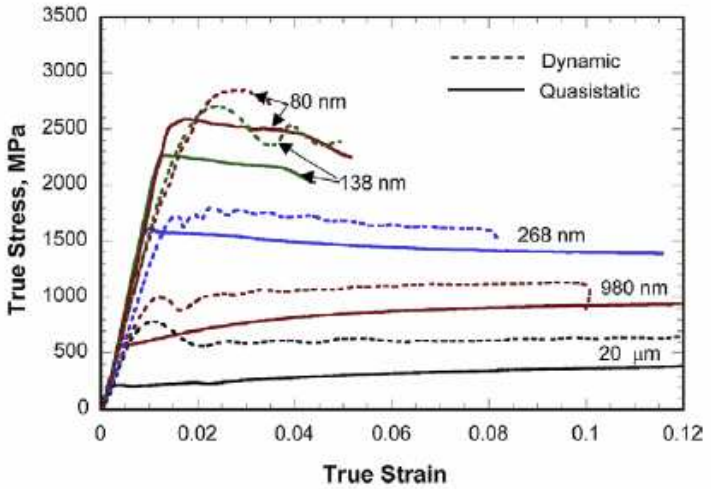


Fig. 2. Typical stress-strain curves obtained for the consolidated iron under quasistatic ($1–4 \times 10^{-4}$ s^{-1}) and high-strain-rate ($3–6 \times 10^{+3}$ s^{-1}) uniaxial compression for all of the grain sizes.

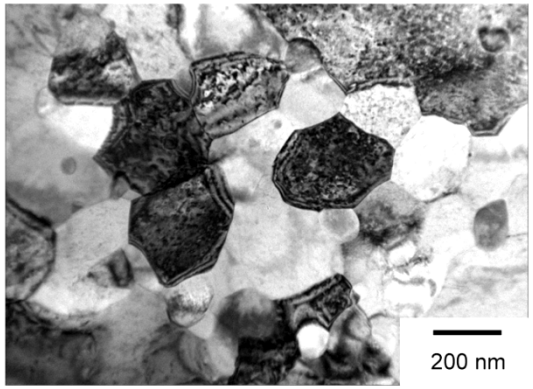
Quasistatic compression test at RT:

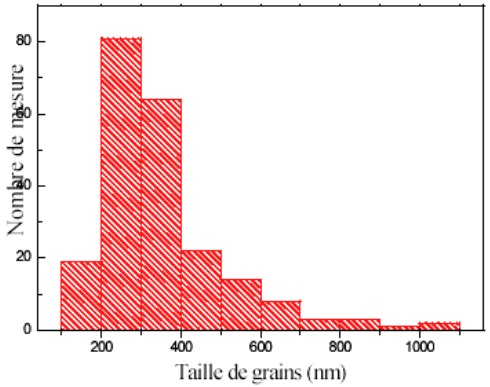
D. Jia et *al.* Acta Mater. 51 (2003) 3495

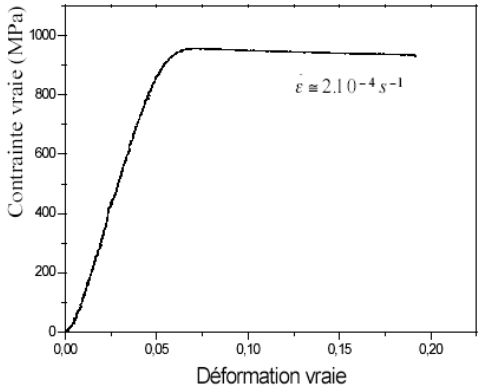
- Increase in strength

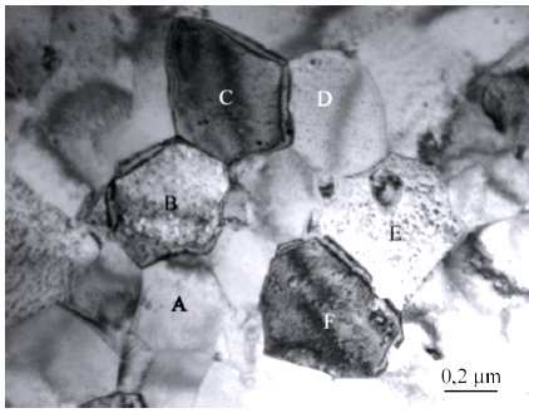
- Transition of the deformation mode from hardening to softening

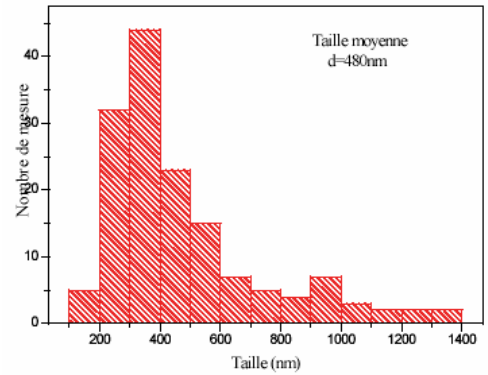
Note: this behavior depends also on the grain size distribution

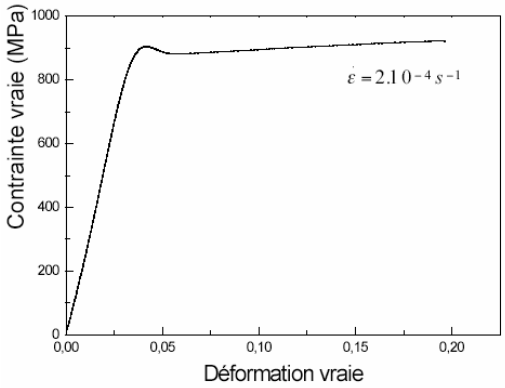






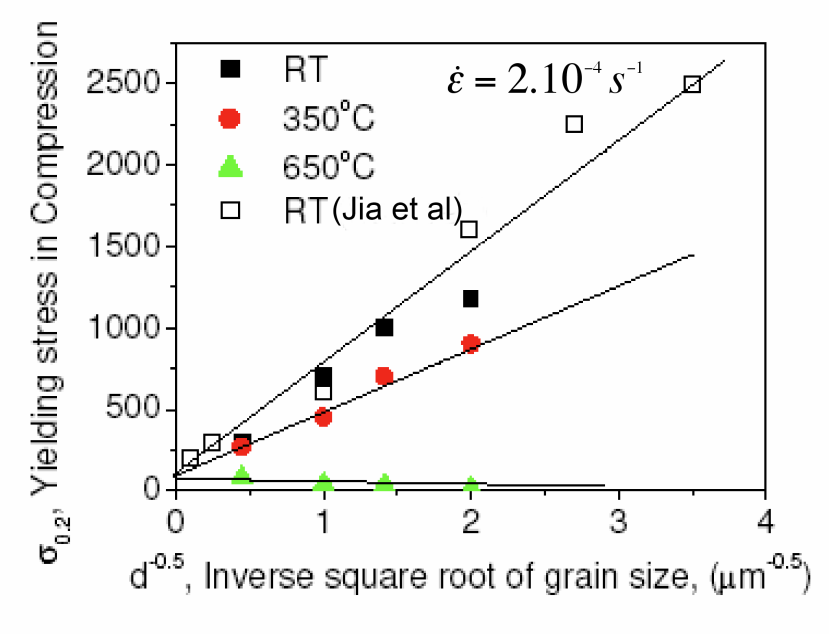


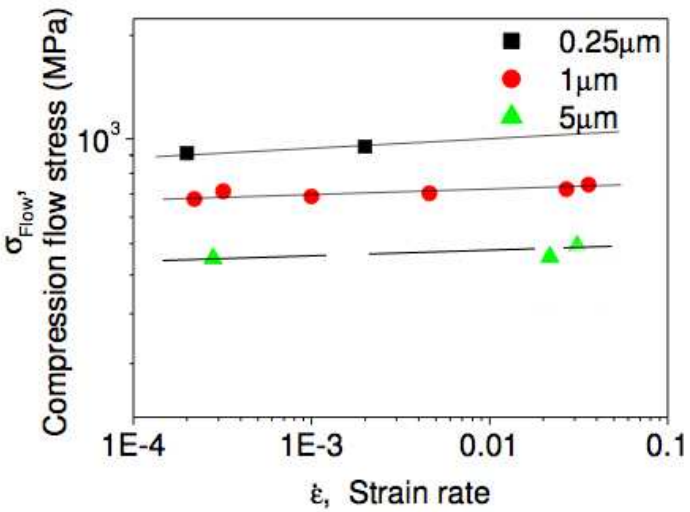




Grain size distribution effect

Evolution of the mechanical properties

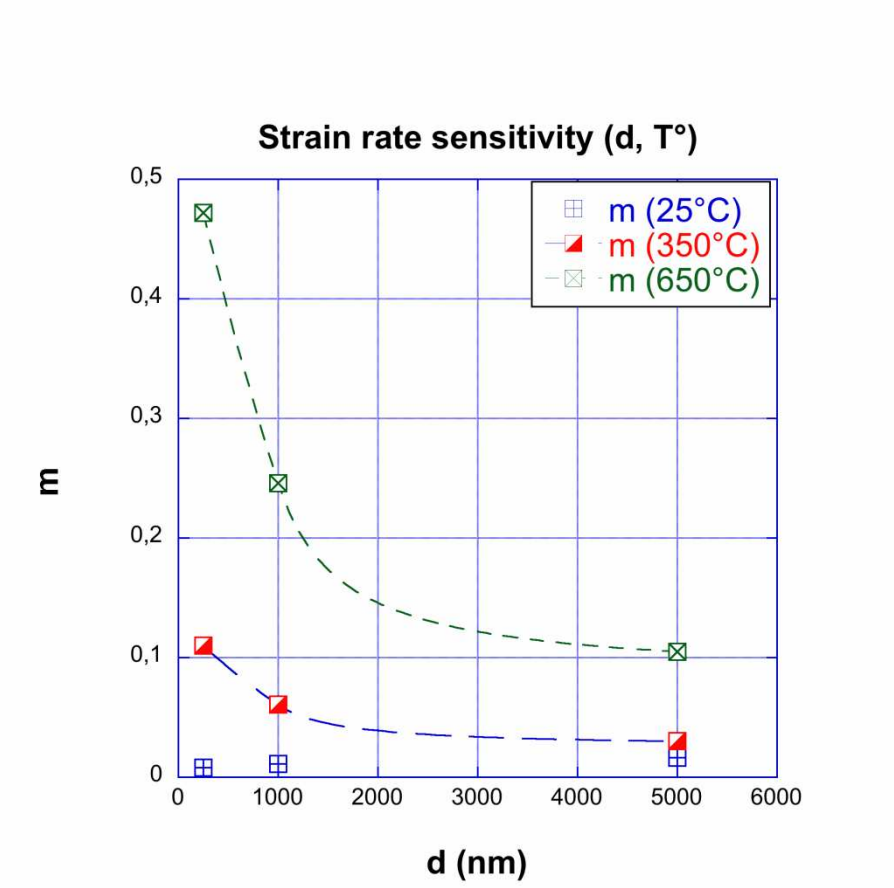




$$\sigma = \sigma_0 + \frac{K_{H-P}}{\sqrt{d}}$$

Low strain rate effect on the flow stress in the range 10^{-4} ~$10^{-1}s^{-1}$ at RT

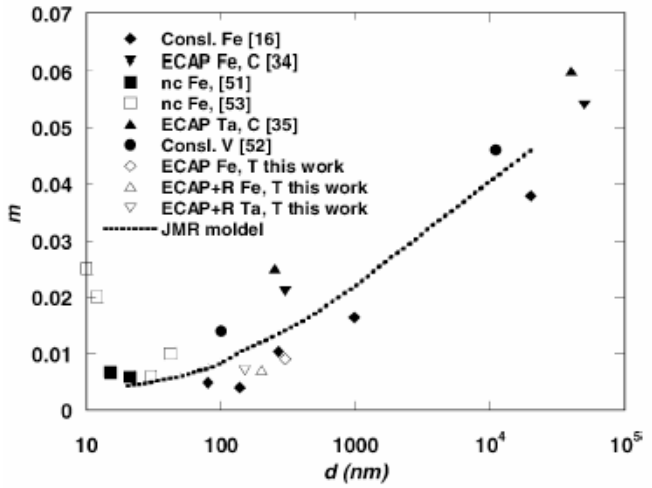
Strain rate sensitivity dependence on *d* and *T*

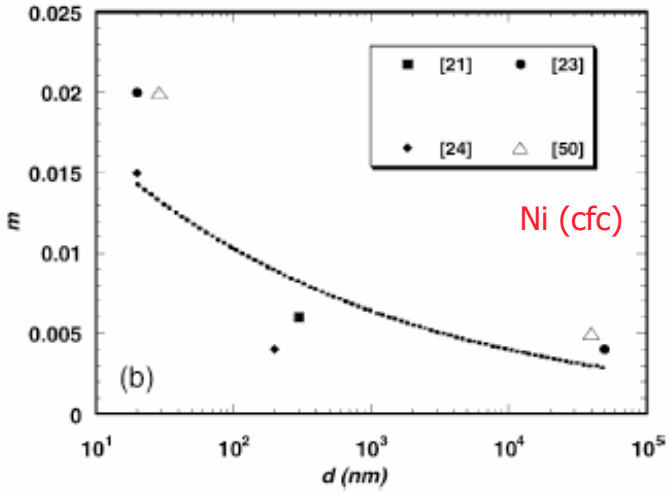


Activation volume at RT

v_{ufg}*: 20-50b^3

$V_{conv.}$* ≈10b^3





Wei et al., Mat. Sci. and Eng. A (2004)

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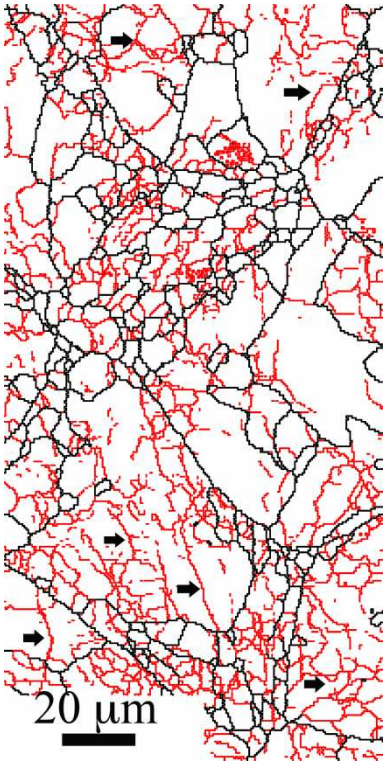
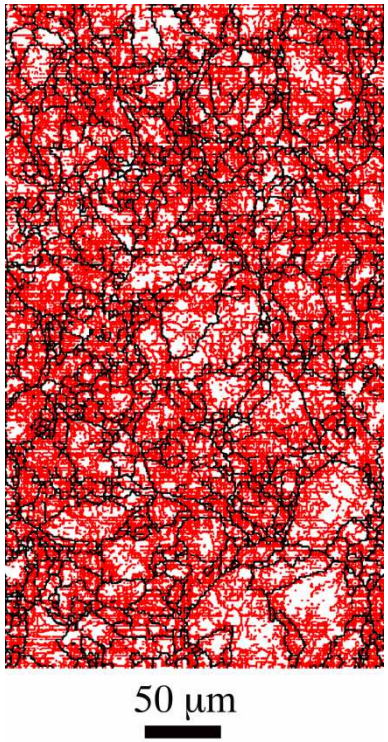
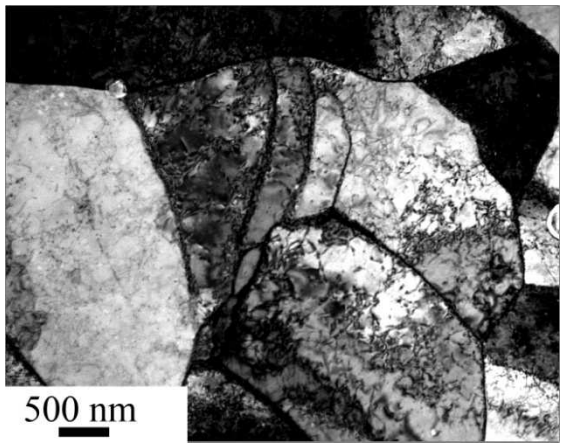
Microstructure evolution: *mc* iron, RT

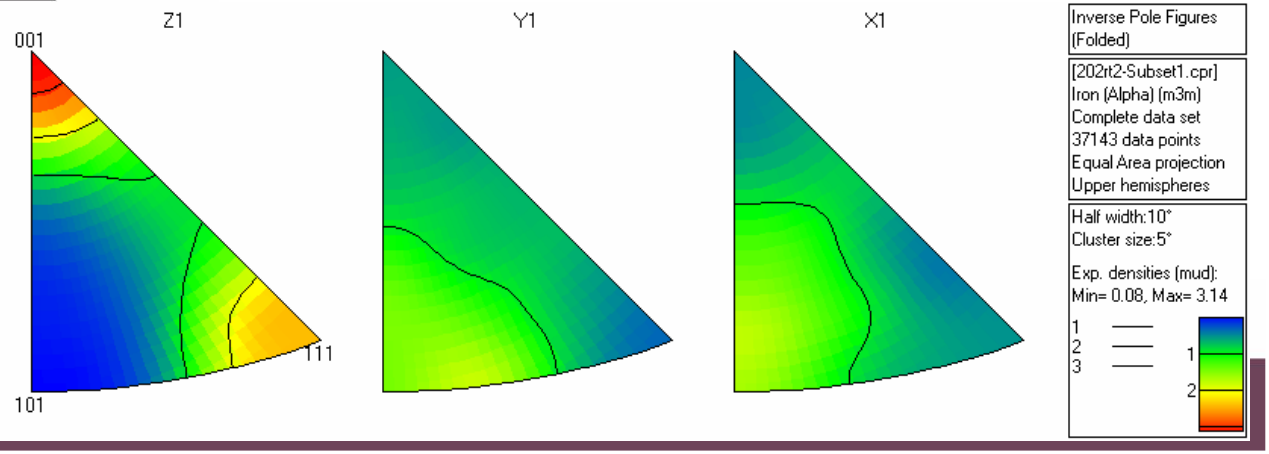




High density of SAGBs

Two texture fibers // compression axis (ND)





Microstructure evolution: *ufg* iron, RT

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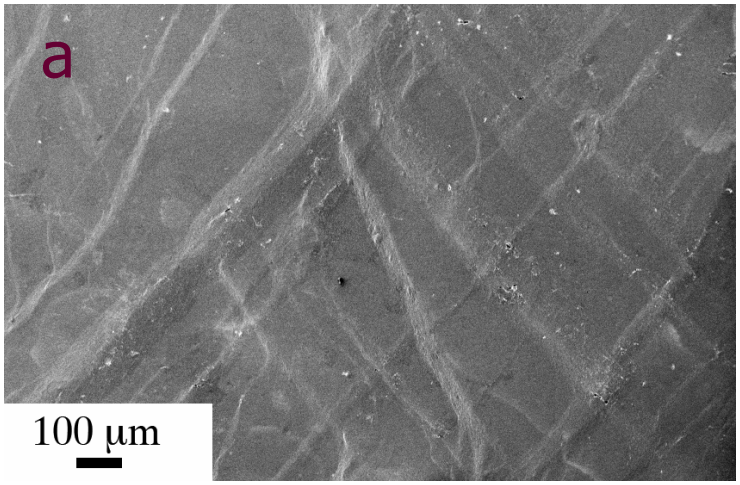

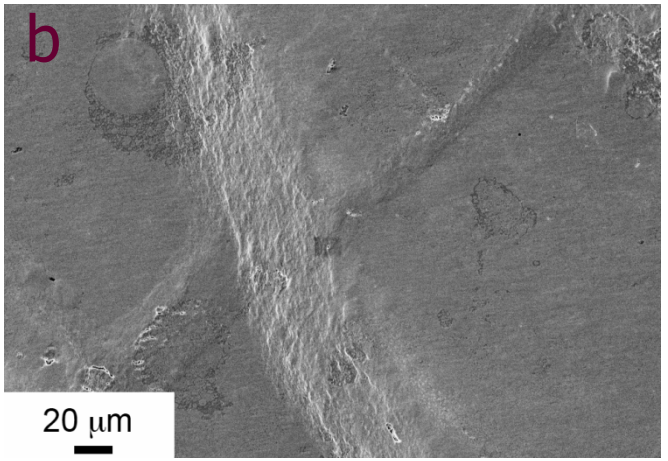

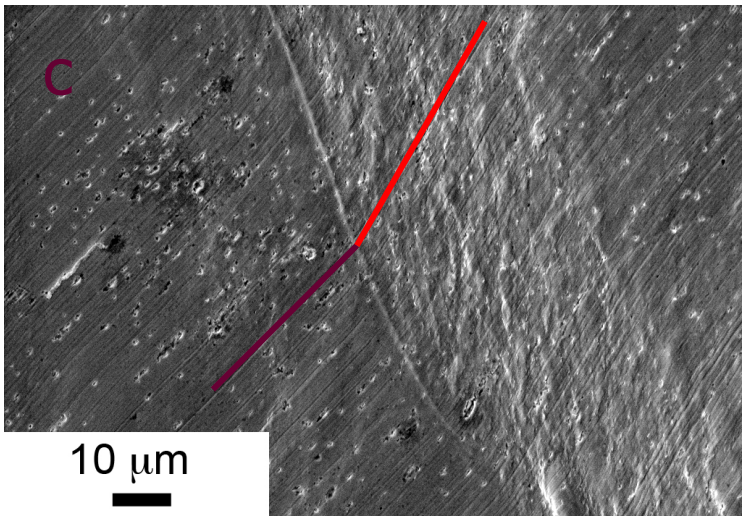

Deformation localizes within shear bands (a, b, c)

Deformation is compatible (a)

Intense shear (c)

ε = 15%; RT;

Grain size = 0.5 μm

EBSD analysis (*ufg* materials, RT)

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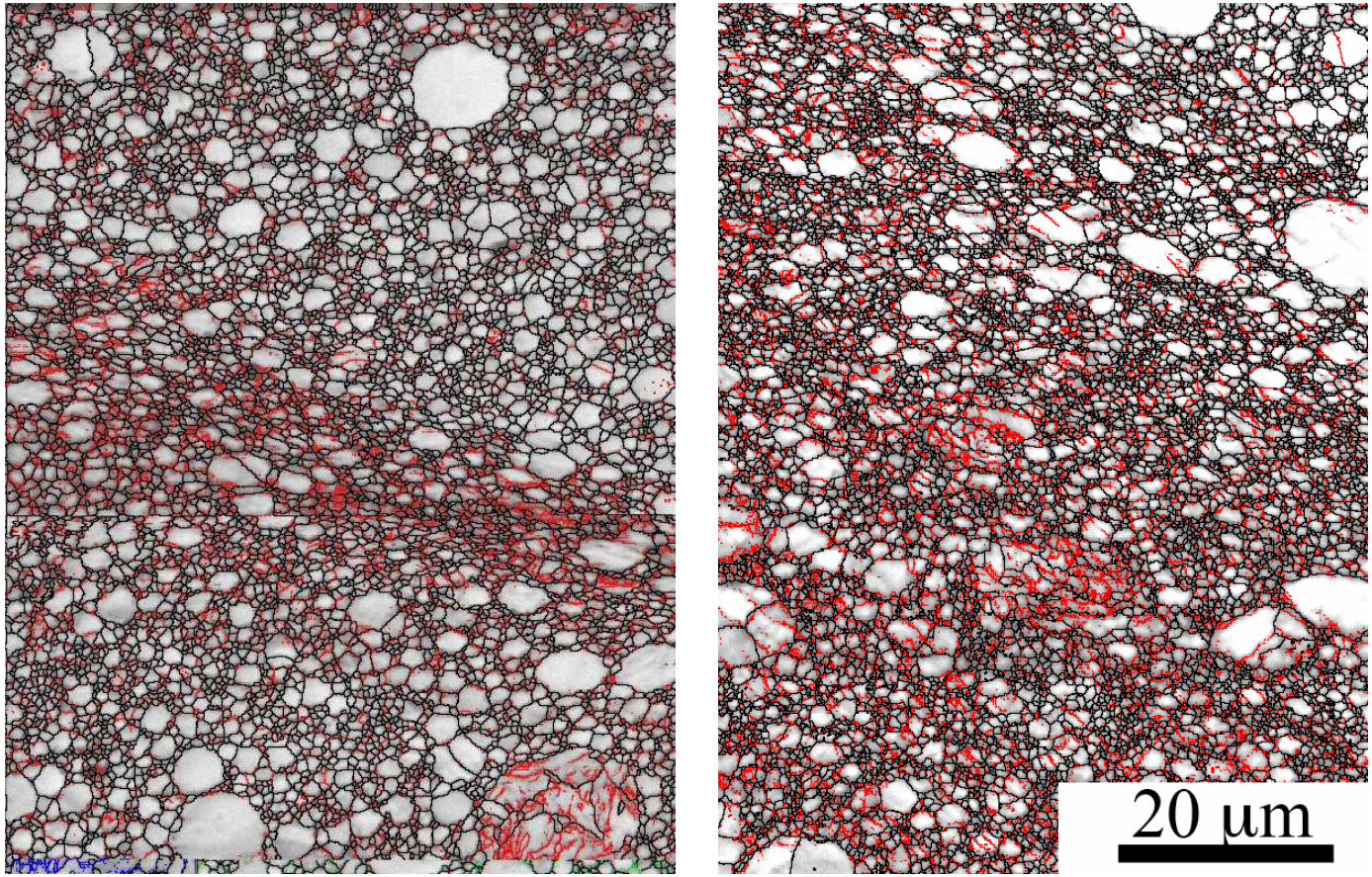
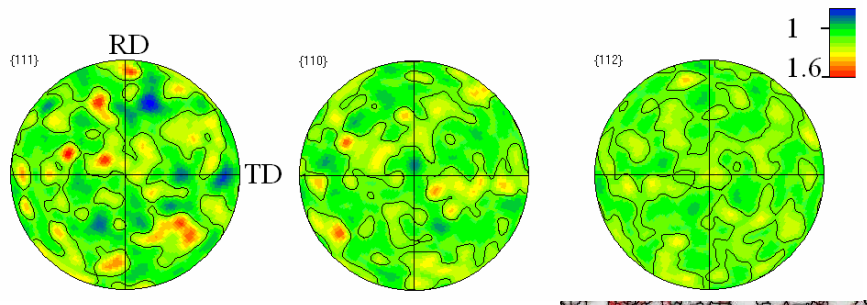
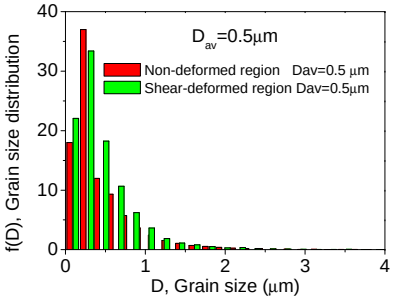


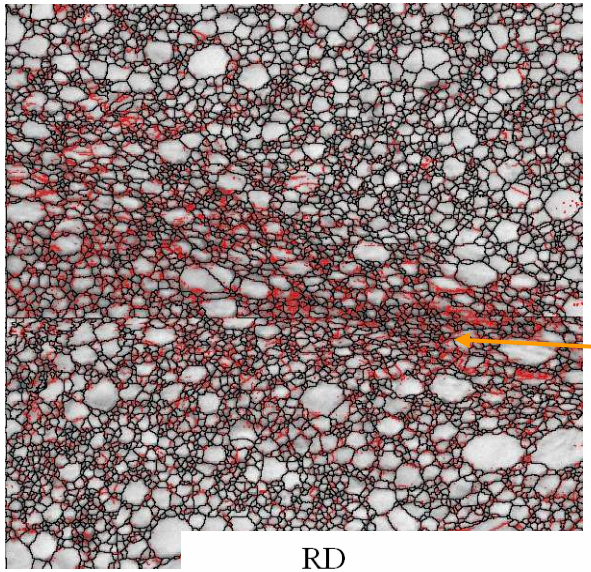
single SB wide SB

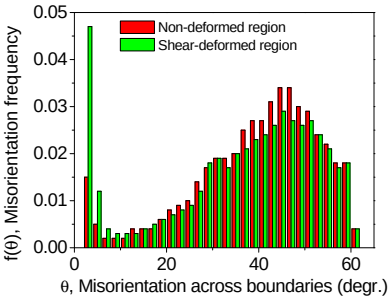
High density of SAGBs within de SB: the deformation apparently by motion of lattice dislocations

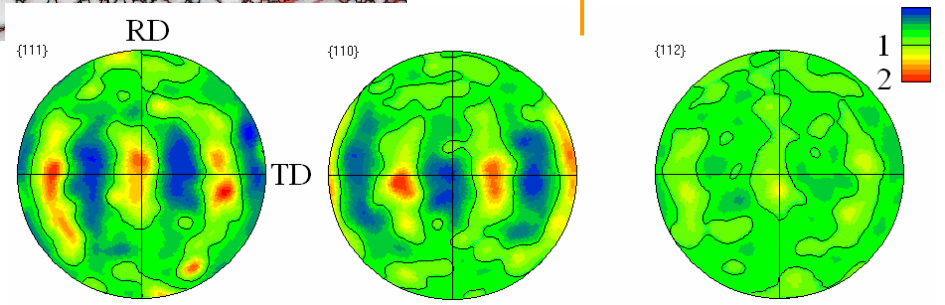


(a)



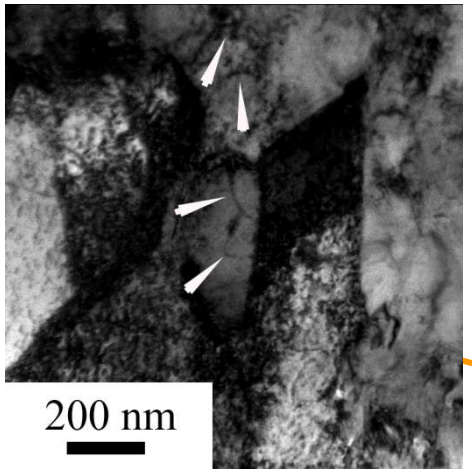


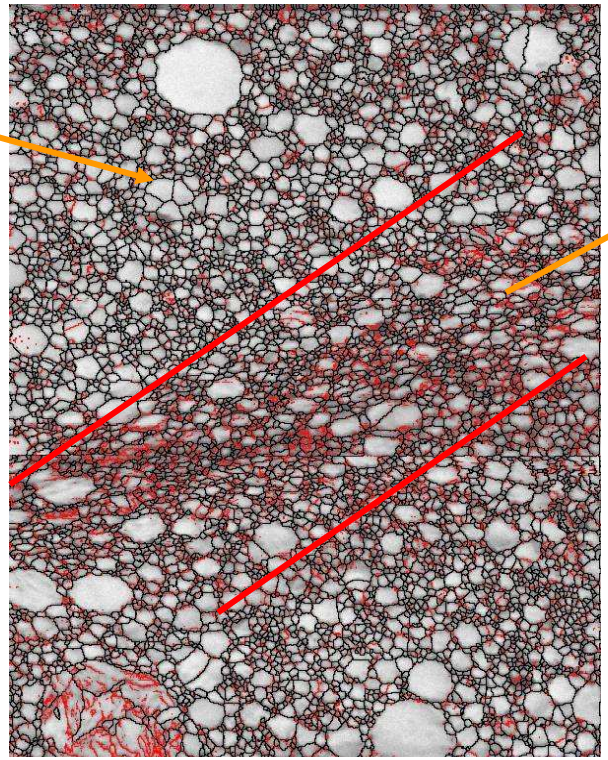


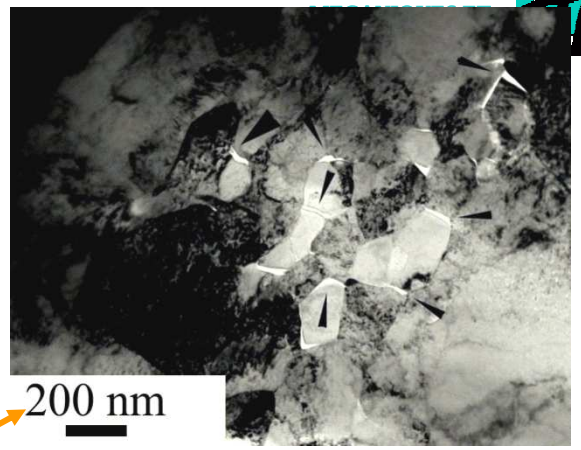


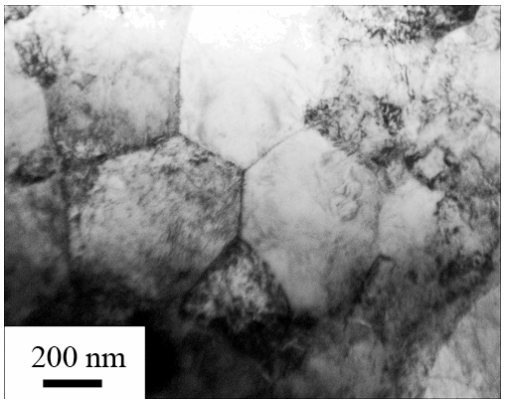
(b)

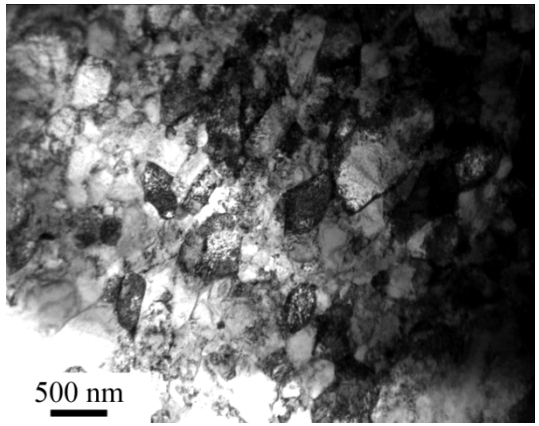
Heterogeneous deformation mode. (a): random texture outside the SB; (b) building of a [111] texture inside the SB region.







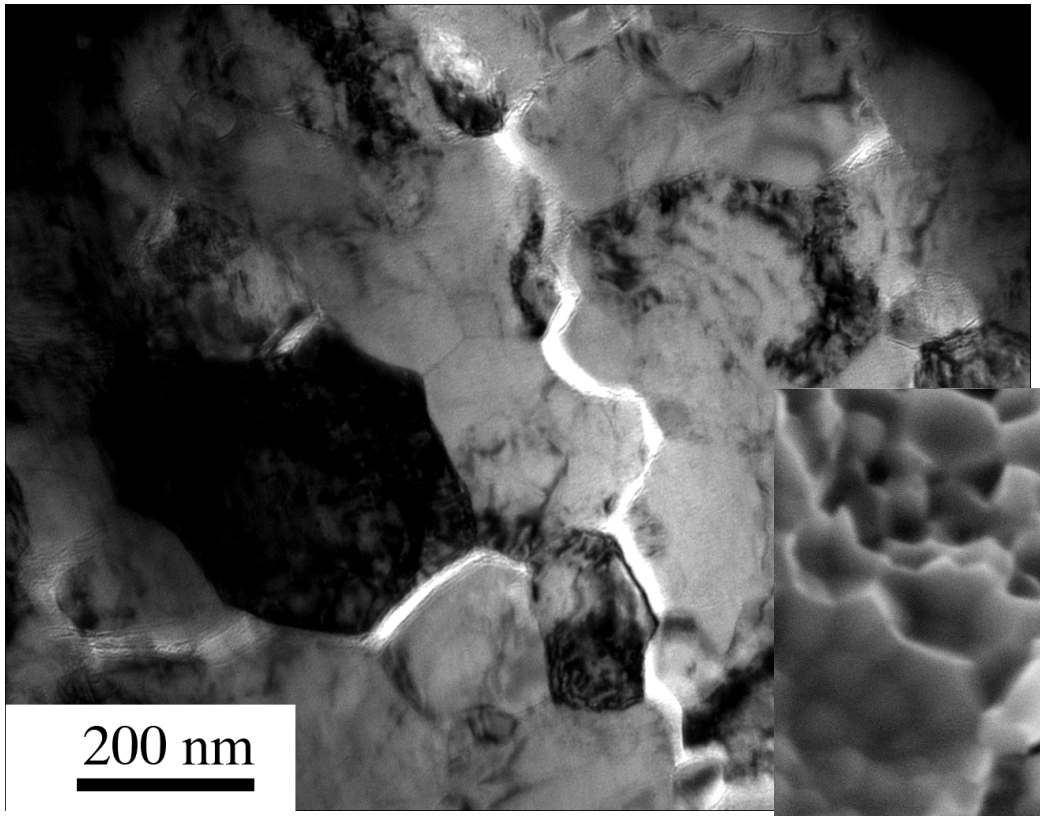


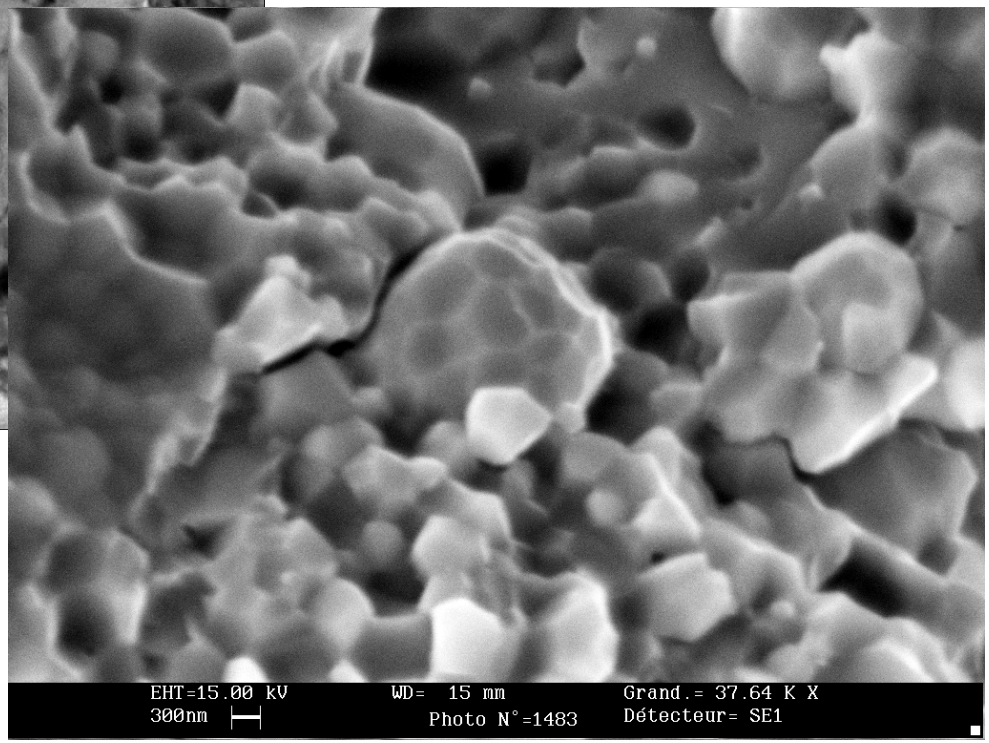


Details of the microstructure by combined TEM + EBSD experiments

Inter granular fracture mode (at RT)

LABORATOIRE DES
PROPRIETES
MECANIQUES ET
THERMODYNAMIQUES
DES MATERIAUX



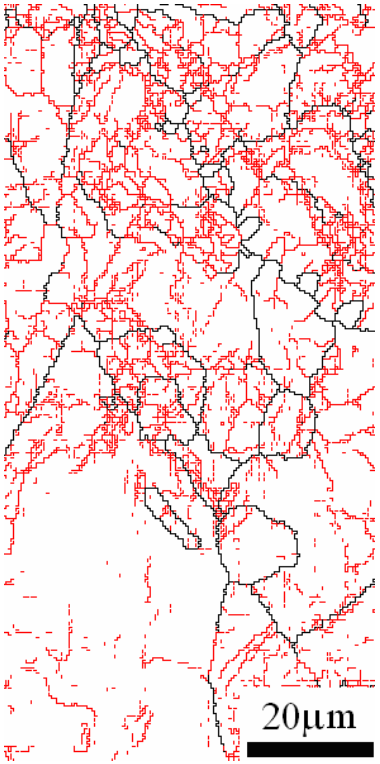


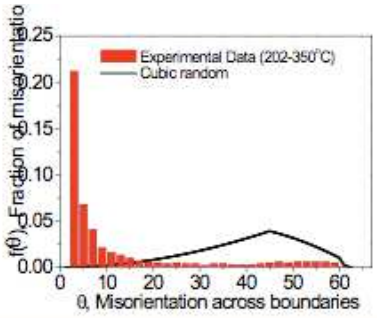
- Temperature effect on the microstructure

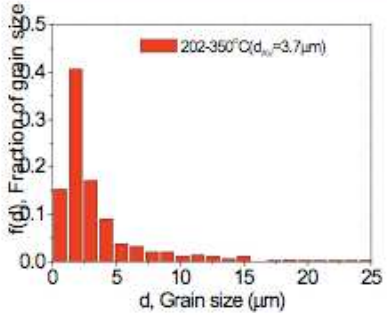
mc materials

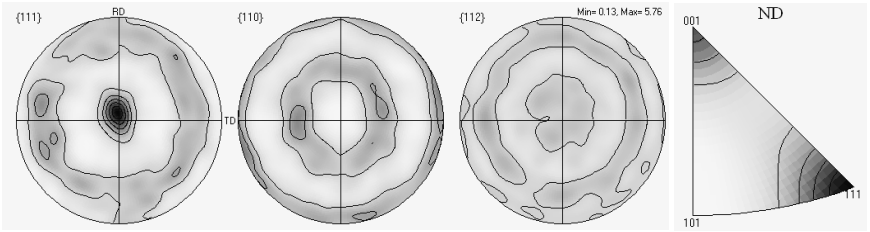
LABORATOIRE DES PROPRIETES MECANIQUES ET THERMODYNAMIQUES DES MATERIAUX

350°C



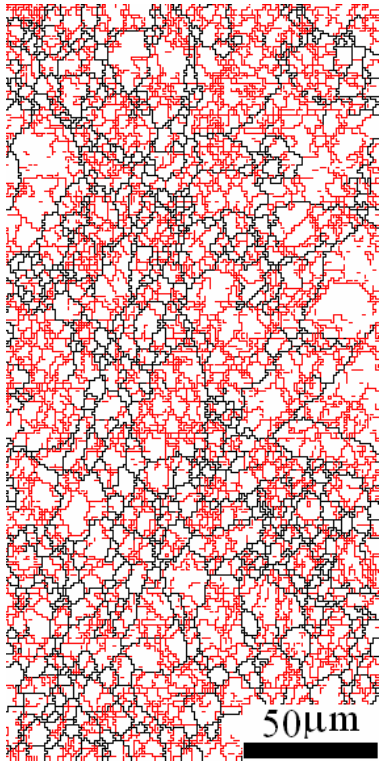


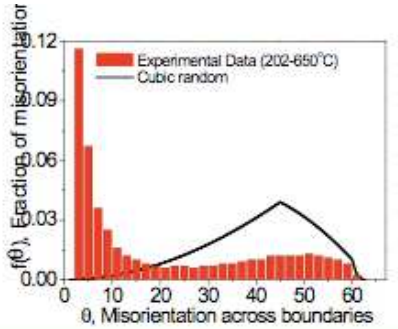


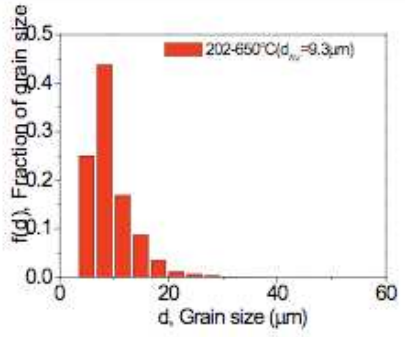


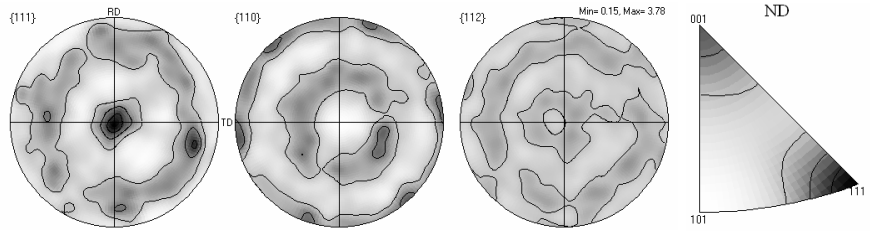
High density of SAGBs

650°C









Two Strong texture fibers

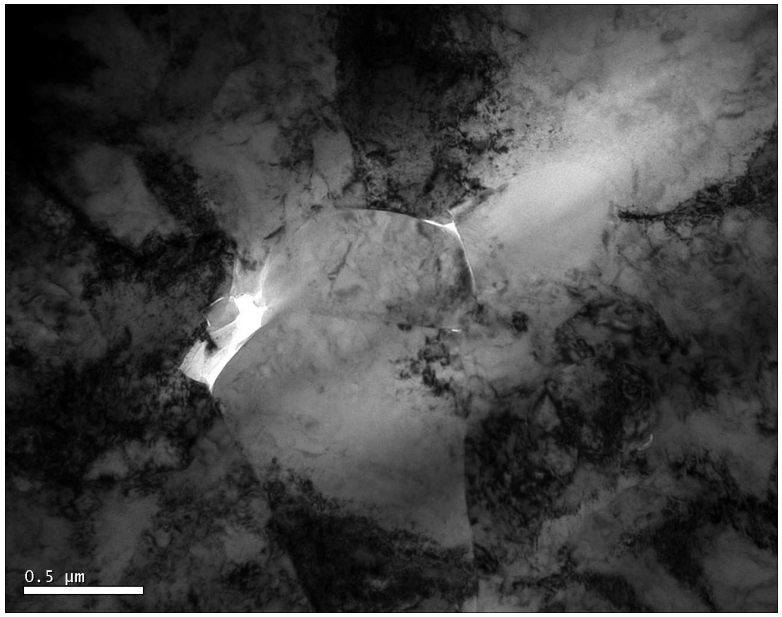
No shear banding

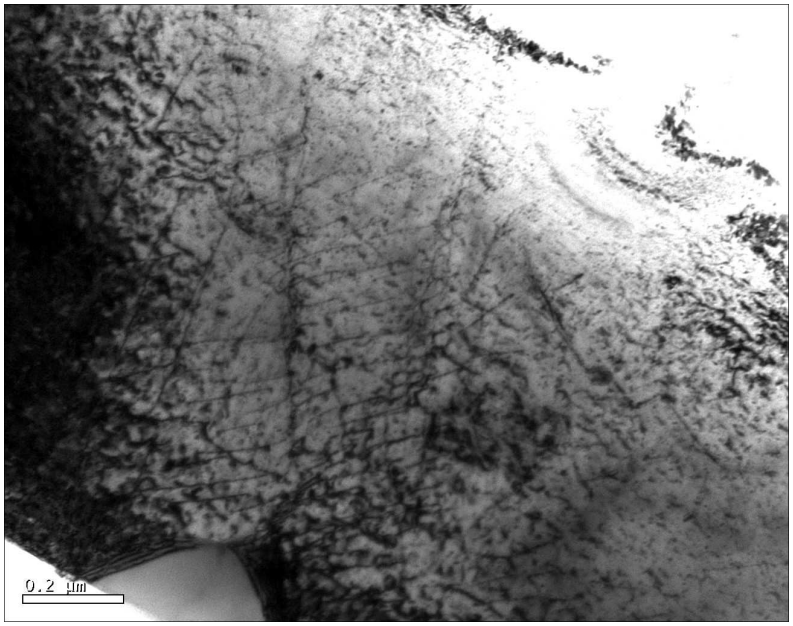
mc materials: TEM

LABORATOIRE DES
PROPRIETES
MECANIQUES ET
THERMODYNAMIQUES
DES MATERIAUX

350°C

650°C



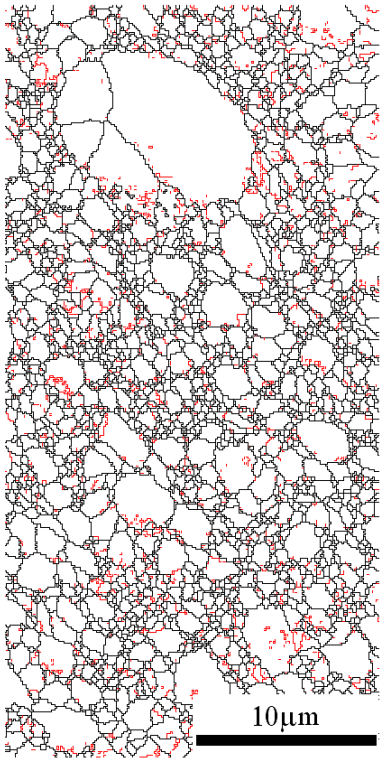


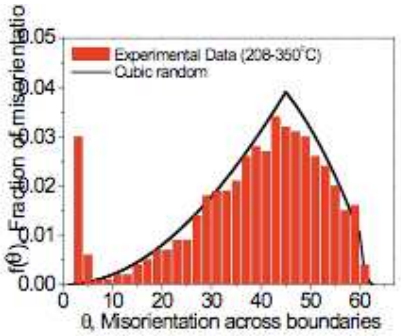
Plastic deformation by lattice dislocations, triple junction cavitation

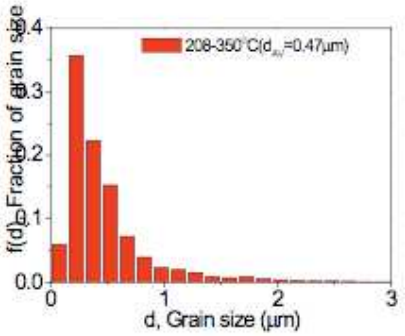
LABORATOIRE DES
PROPRIETES
MECANIQUES ET
THERMODYNAMIQUES
DES MATERIAUX

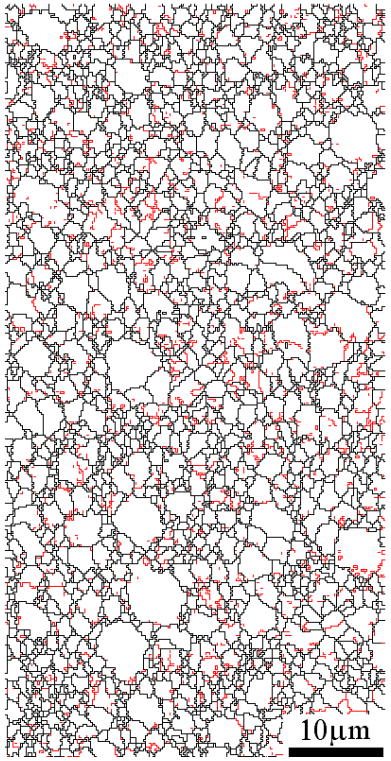
Ufg materials

350°C

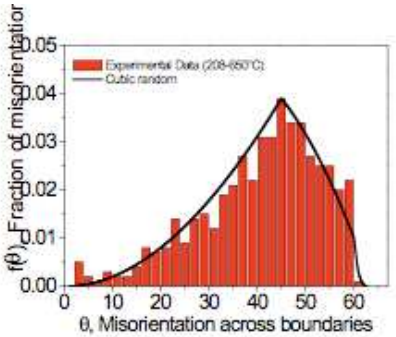


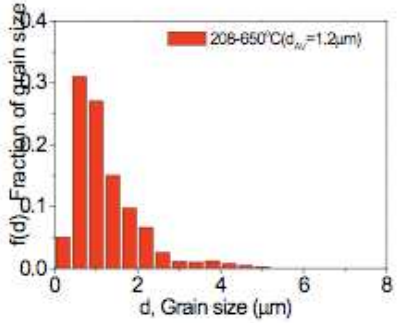


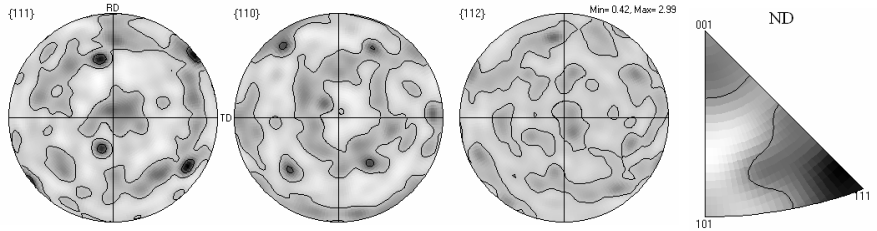


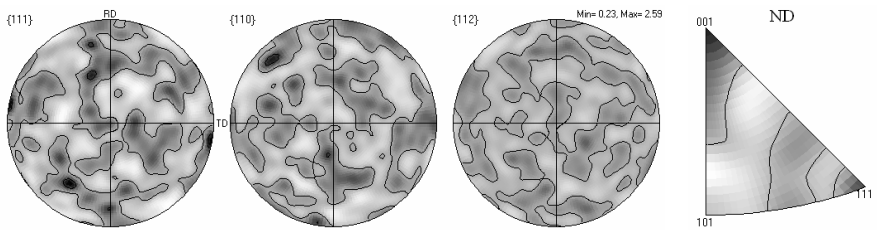


650°C









weak density of SAGBs

Random crystallographic texture

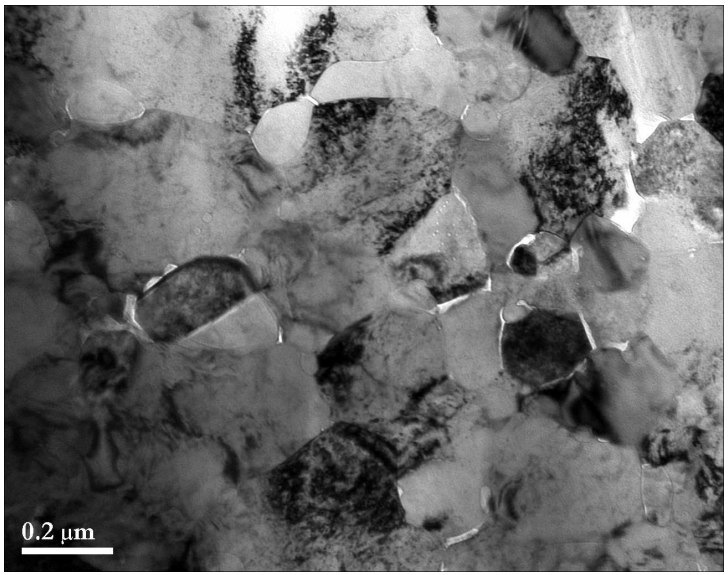
No shear banding

350°C

Ufg materials: TEM

650°C

LABORATOIRE DES PROPRIETES MECANIQUES ET THERMODYNAMIQUES DES MATERIAUX



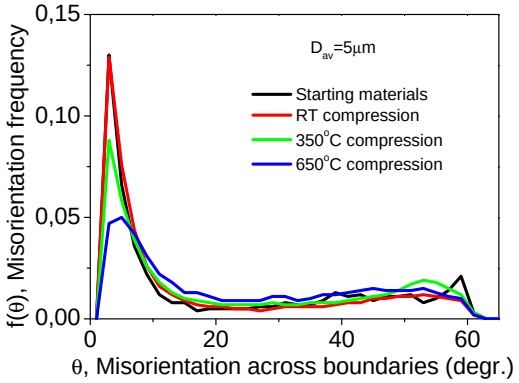
weak density of SAGBs

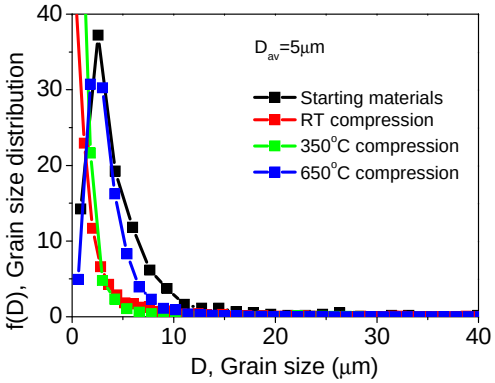
equiaxed grains

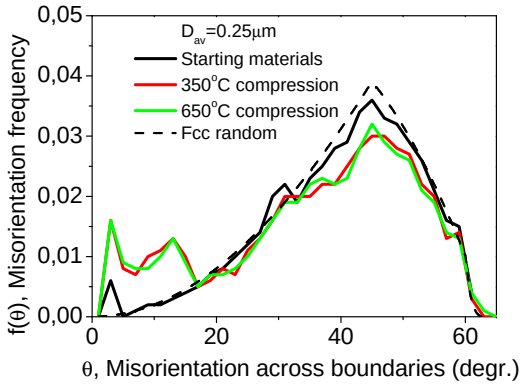
No shear banding

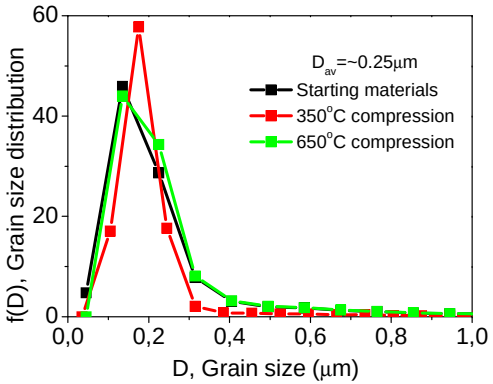
The deformation parameters

LABORATOIRE DES
PROPRIETES
MECANIQUES ET
THERMODYNAMIQUES
DES MATERIAUX









Conclusions

LABORATOIRE DES
PROPRIETES
MECANIQUES ET
THERMODYNAMIQUES
DES MATERIAUX

• At RT, a transition in the deformation mode occurs with grain size decreasing.

• Below at about 1 μm the deformation proceeds via SBs generation and widening

• The deformation within the SB is a complex interplay of lattice dislocations movement and grain boundary-related mechanisms (rotation, migration, sliding, damage…)

• At higher temperature, the *ufg* materials deform mostly via boundary mechanisms ($m \approx 0.5$); while the *mc* counterpart still deforms by lattice dislocations movement.