# Multiscale viscoplastic behaviour of halite:

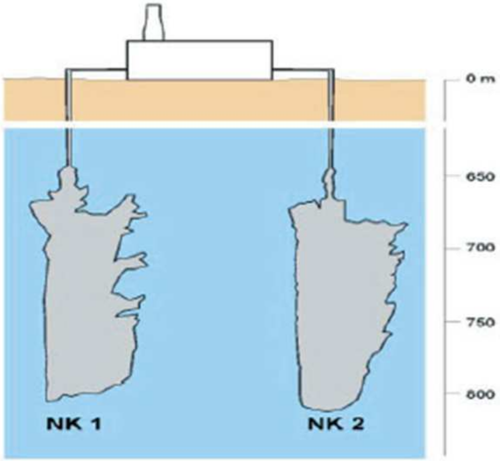
## micromechanical approaches by full field measurements


M. Bourcier[1], **A. Dimanov[1]**, M. Bornert[2], E. Héripré[1], B. Charrier[1], W. Ludwig[3] J. Raphanel[1]

*[1] Laboratoire de Mécanique des Solides, CNRS - Ecole Polytechnique, Palaiseau,*
*[2] Laboratoire Navier, CNRS - ENPC, Marne la Vallée,*
*[3] Mateis, CNRS - Insa, Lyon*

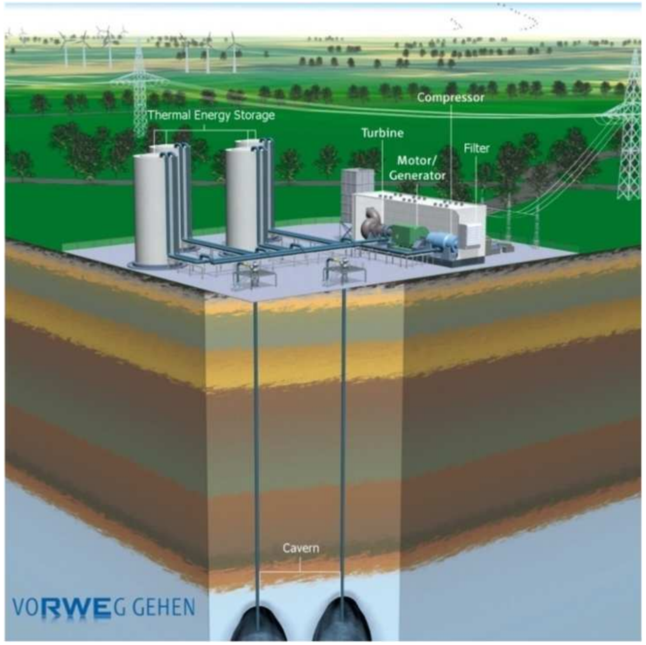
**Geotechnical applications**:
temporary storage of hydrocarbons, compressed air (halite deep caverns).
long term storage of nuclear wastes (halite mines).


Compressed Air Energy Storage (CAES)




Pilot Power Plant since 1978,
Huntorf, Germany
(E.N. Kraftwerke).

2 caverns, 300 000 m3
$P_{min}$ = 4 MPa,
$P_{max}$ = 7 MPa
(290MW)

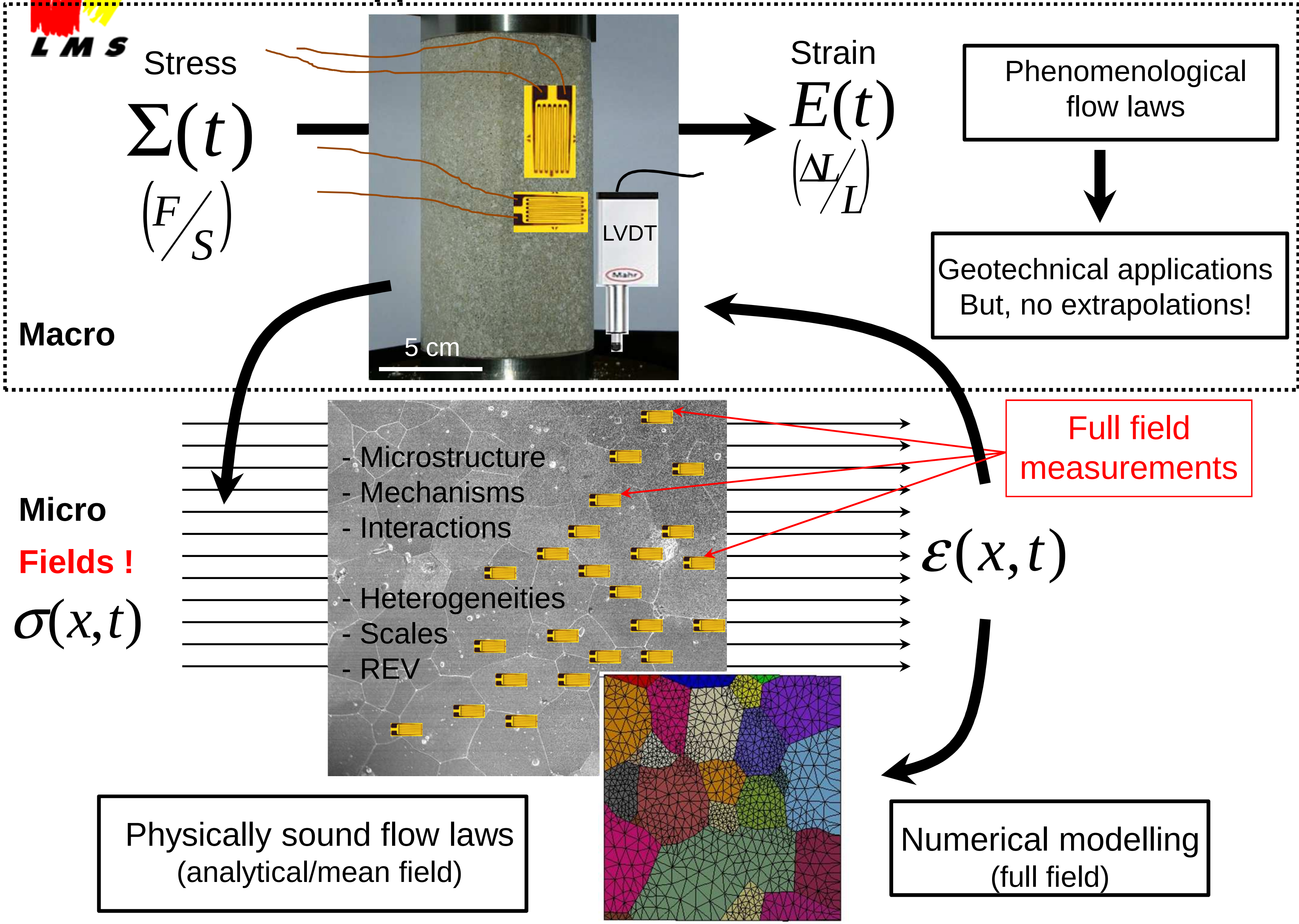



ADELE (Adiabatic CAES) :
heat-storage during air compression
(70% higher efficiency).

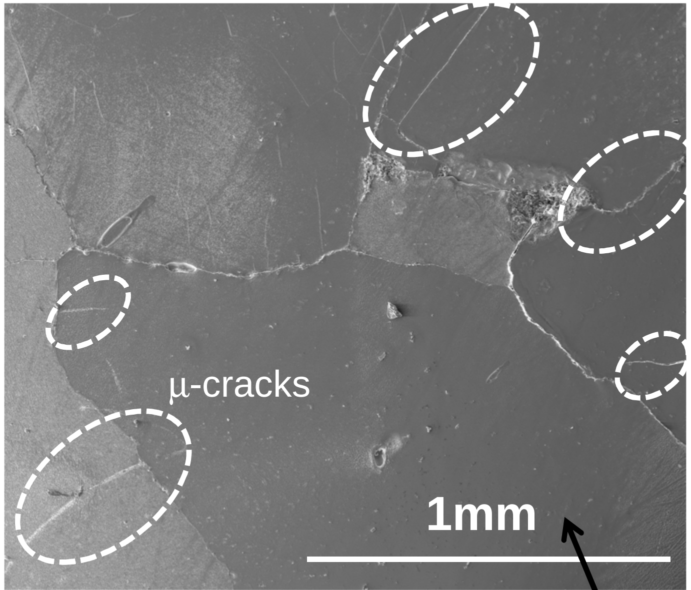
RWE Power project started in 2010
Possibly pilot power plant in Stassfurt (Germany)

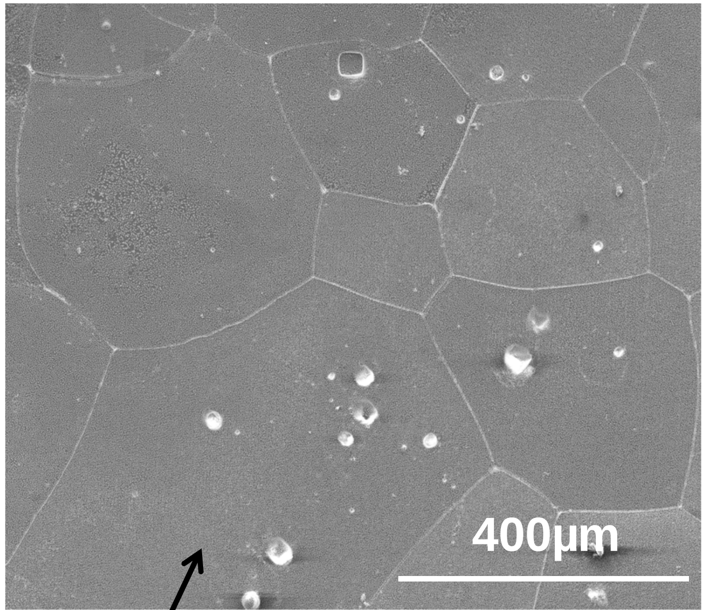
Storage capacity : 90 MW electric output.
Substitute for 50 wind turbines (4 hours).

# Multi scale approaches of mechanical behaviour



## Synthetic (unrealistic) versus natural (unreadable) materials

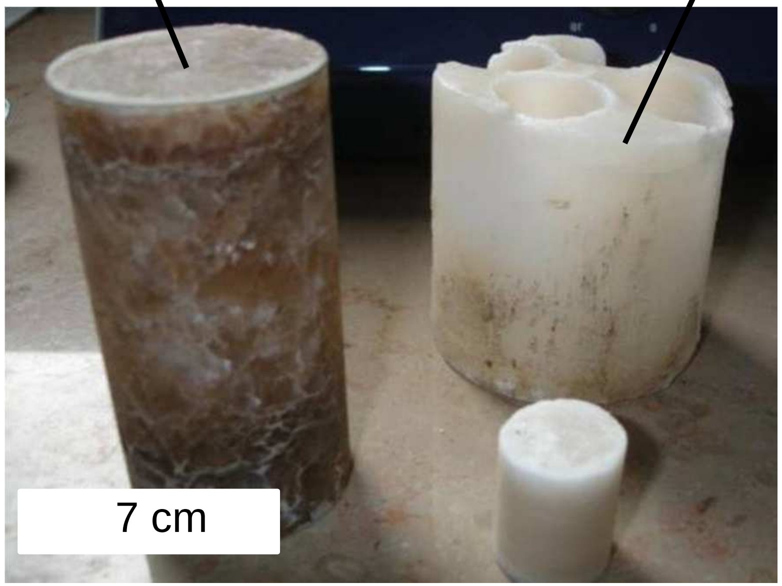



**Natural Halite samples :**

Heterogeneous,
second phases,
pre-strained,
pre-damaged…

How representative (REV)?

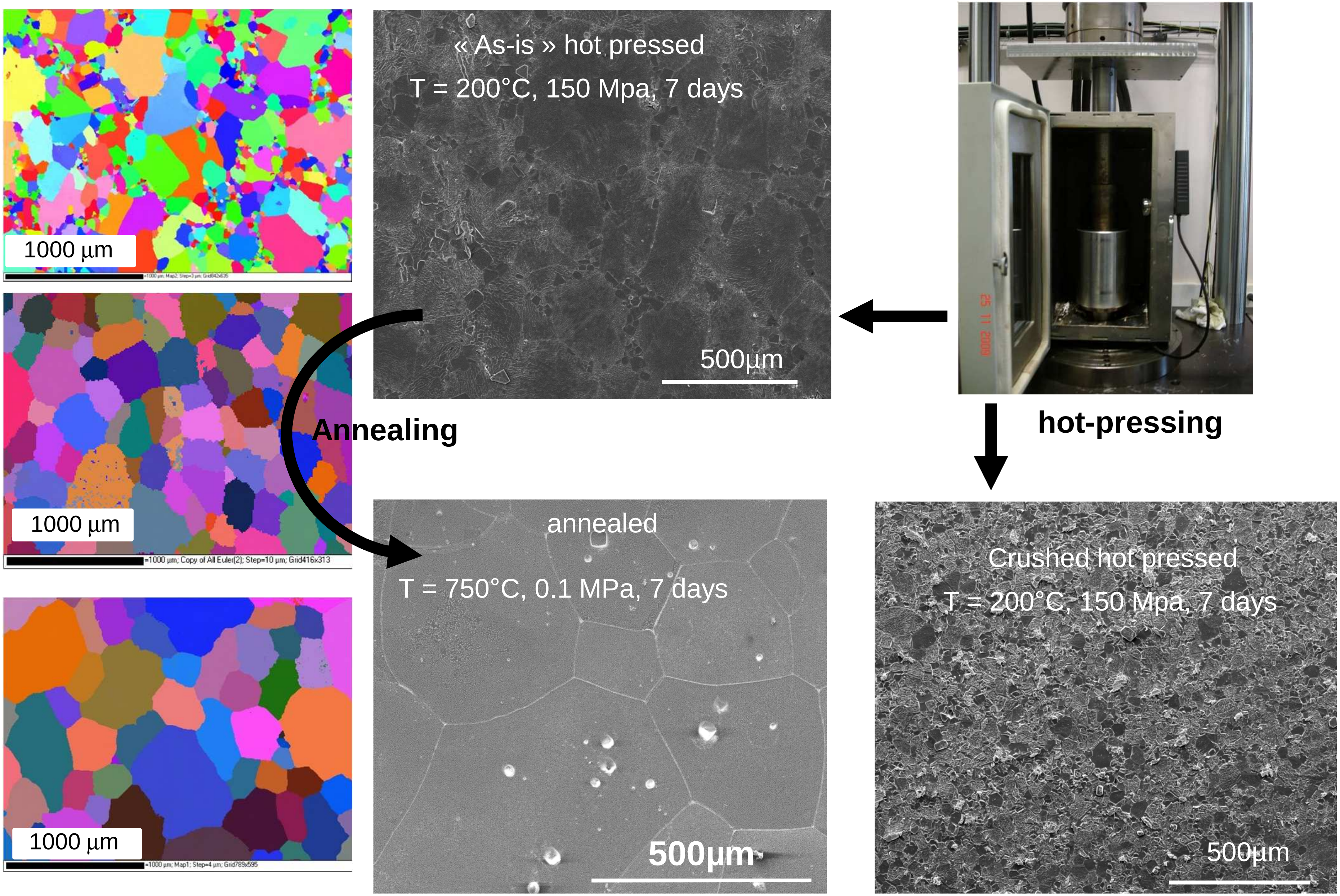



**Synthetic Halite samples :**

Homogeneous,
equilibrated/controlled
μ-structures,
strain/damage-free…

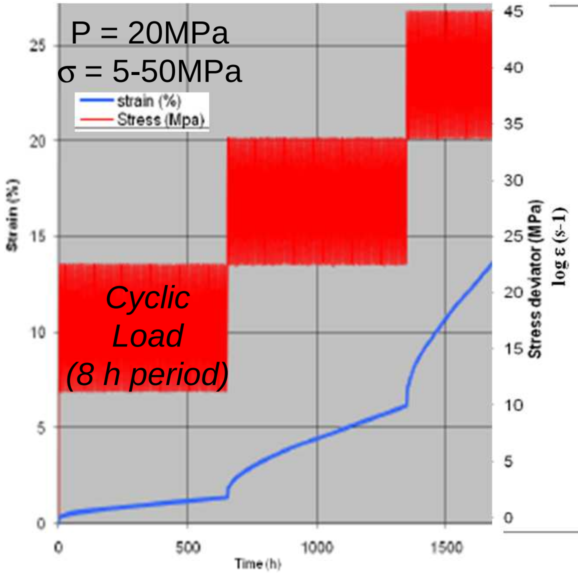
Best candidates for μ-mechanical testing.

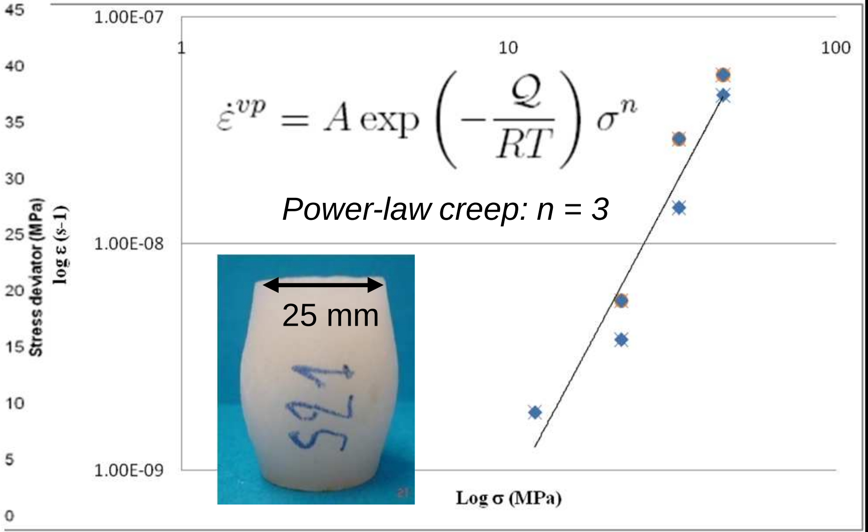
# Sample synthesis (from 99.9% NaCl powder)



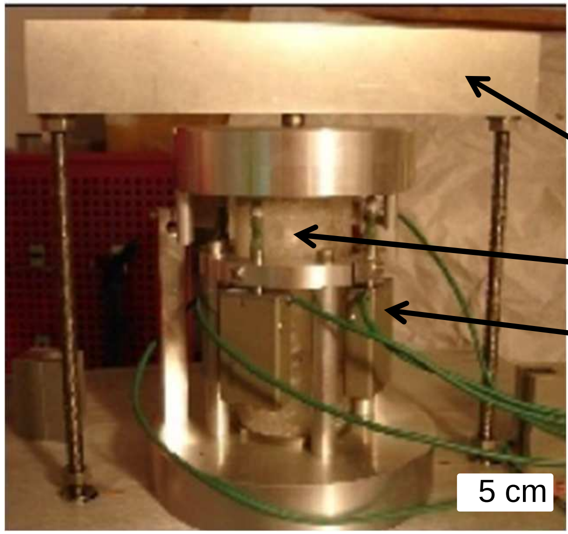
# Macroscopic constitutive relations

Classical creep tests (RT, triaxial)




Very slow creep tests (Varangéville mine)

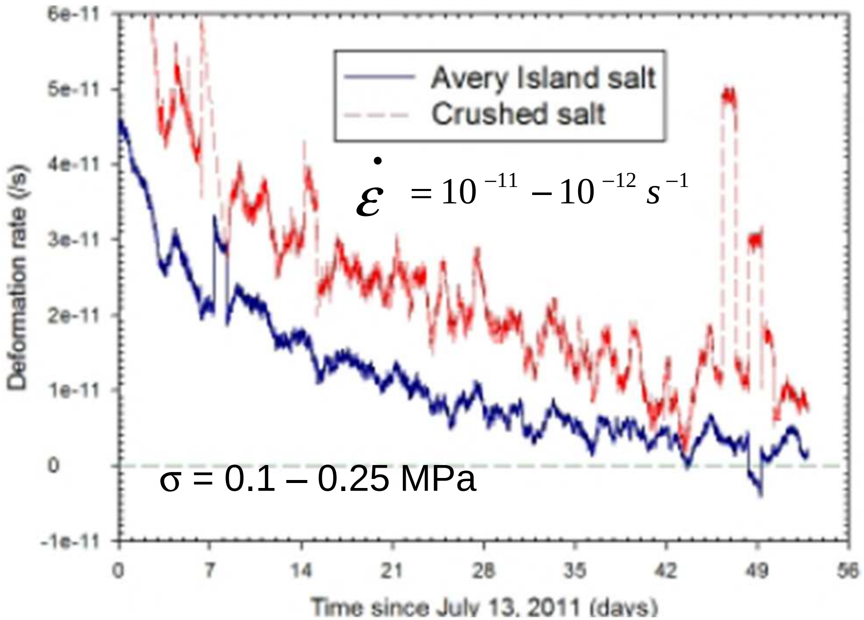



**Dead load rig**

Loading plate

Sample

4 LVDT

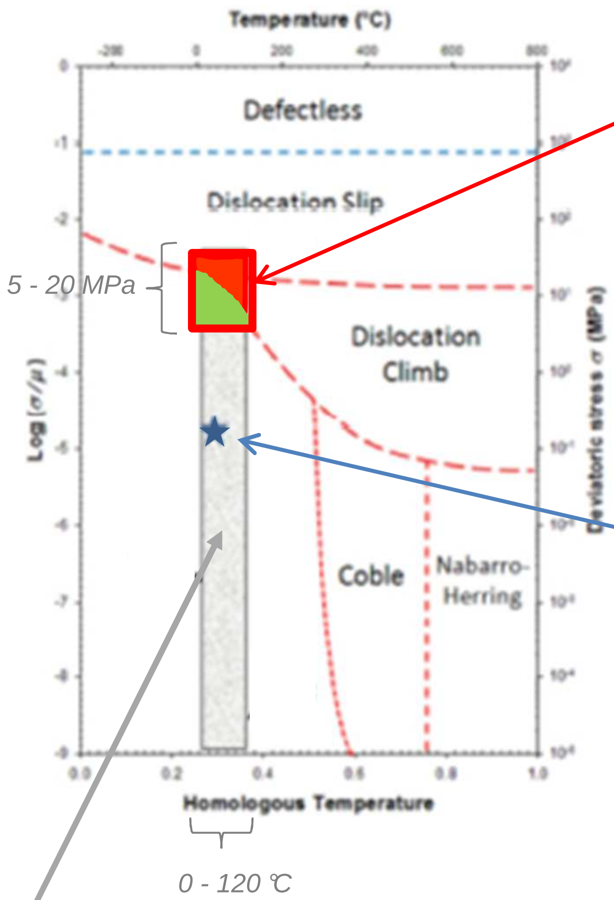


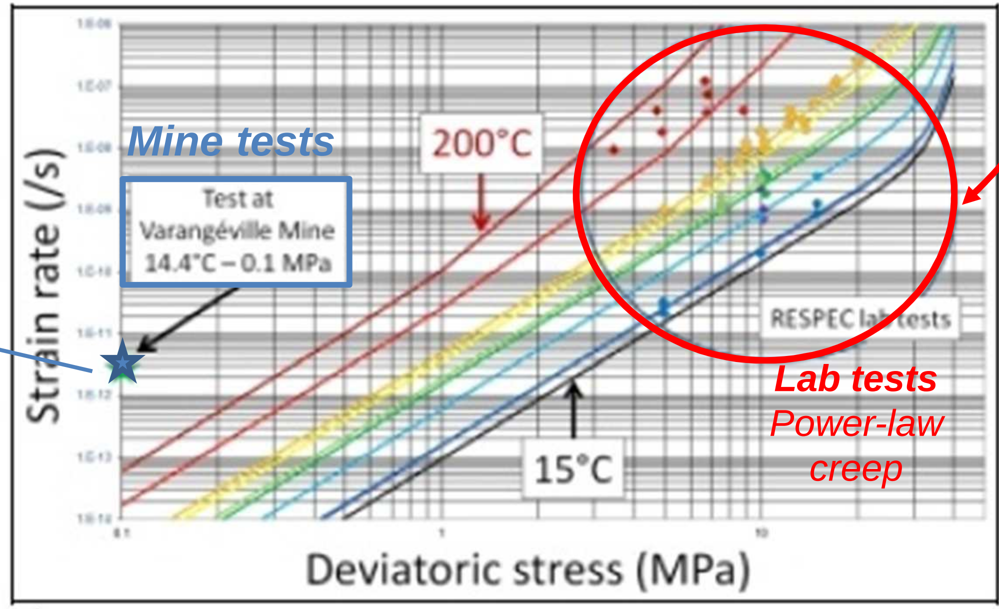
Macroscopic tests ⟶ phenomenological flow laws

*Lab tests*

$$\dot{\varepsilon}_{CL} = 8.1\times10^{-5}\exp\left(\frac{-51600\ \mathrm{J\,mol^{-1}}}{RT}\right)(\sigma_1-\sigma_3)^{3.4}$$

*Power-law creep (Munson, 1997)*

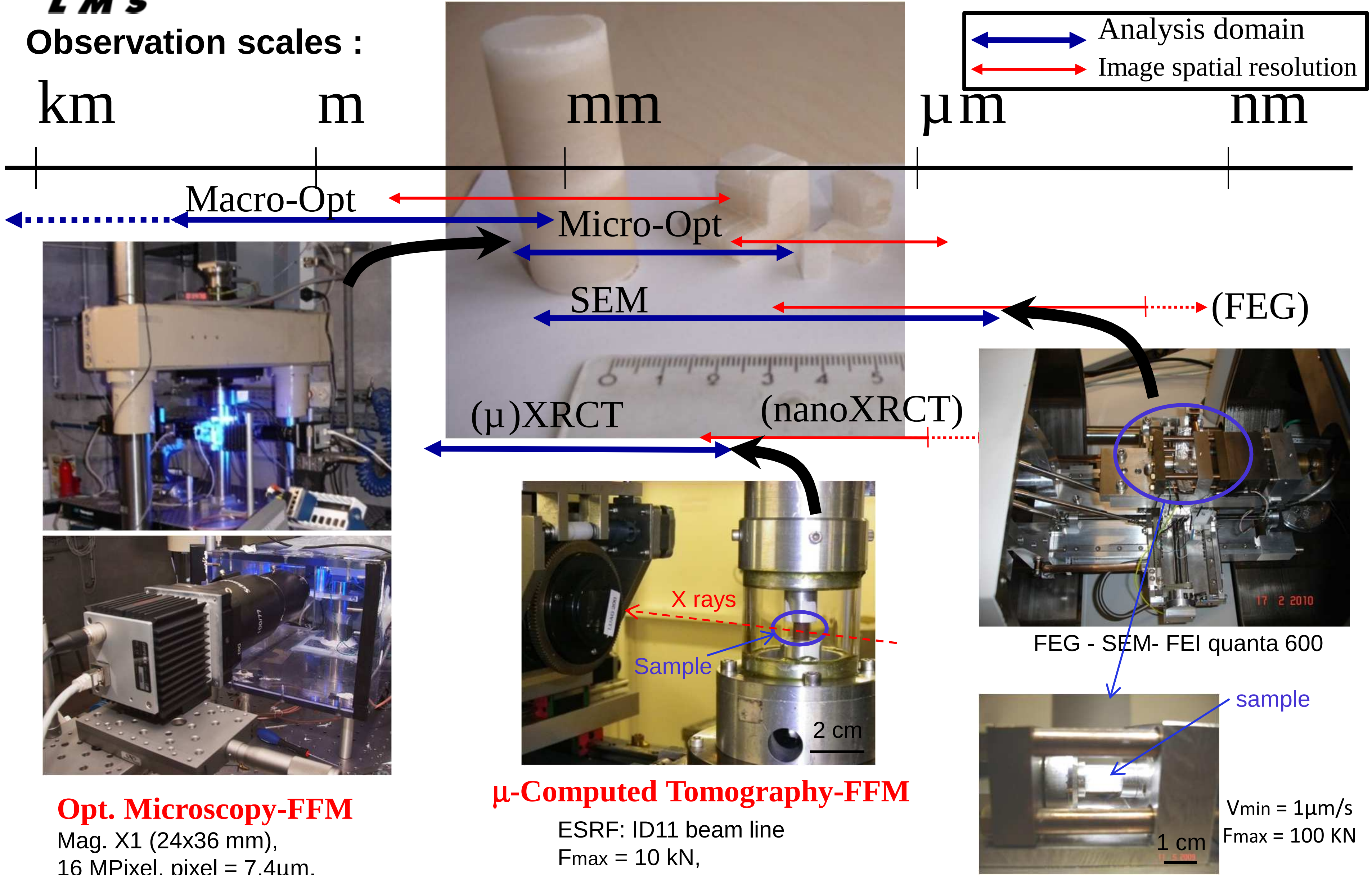



*Storage conditions*
*(undefined mechanism)*

⟶ missleading extrapolations!

# Multi-scale micro mechanical testing:
## Micro-extensometry, or Full (mechanical) Field Measurements (FFM)




**Opt. Microscopy-FFM**
Mag. X1 (24x36 mm),
16 MPixel, pixel = 7,4µm.
Vmin = 1µm/s, Fmax = 100 KN

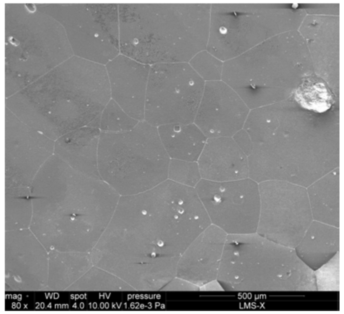
**µ-Computed Tomography-FFM**
ESRF: ID11 beam line
Fmax = 10 kN,
Vmin = 1µm/s (~$10^{-4}$-$10^{-5}s^{-1}$)

**SEM-FFM**

# Full field measurements by Digital Image Correlation (FFM-DIC)

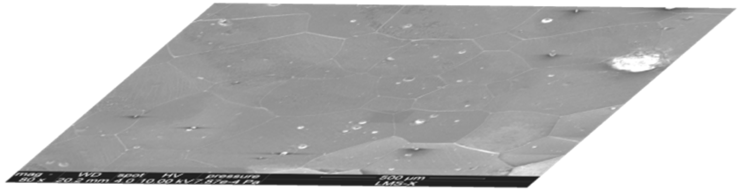
Images ⟶ displacements ⟶ deformations

**1- Image acquisition during loading**



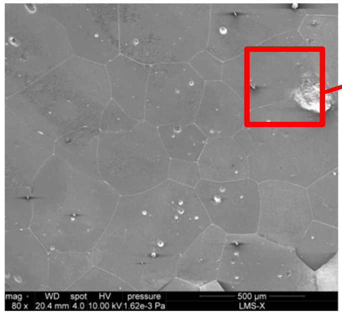
time $t_0$: Reference image

. . .



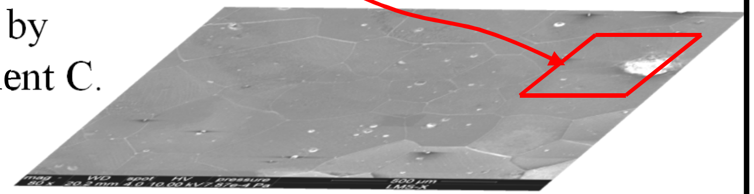
time t: image after deformation

**2 – Image Correlation**



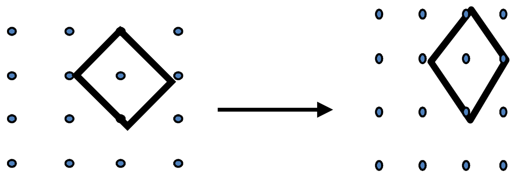
Equivalent domain search:
Comparison of grey scale levels by minimization of correlation coefficient C.

$$C(\Phi_0) = 1 - \frac{\sum_{i\in D} f(\underline{X_i})\cdot g(\underline{x_i})}{\sqrt{\sum_{i\in D}\left(f(\underline{X_i})\right)^2}\cdot\sqrt{\sum_{i\in D}\left(g(\underline{x_i})\right)^2}}$$

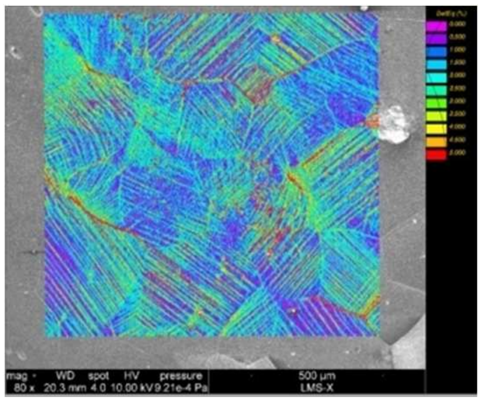


**Displacement field**

**3 – Calculation deformations**



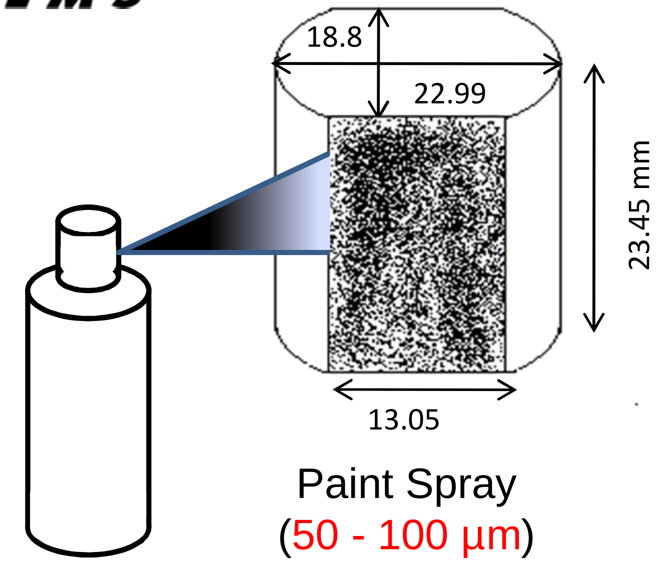
$\varepsilon_{11}$, $\varepsilon_{22}$, $\varepsilon_{12}$, $\varepsilon_{is}$, $\varepsilon_{eq}$ …



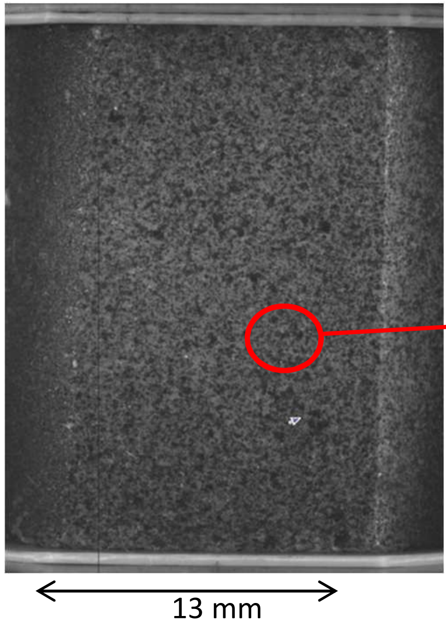
**Strain field**

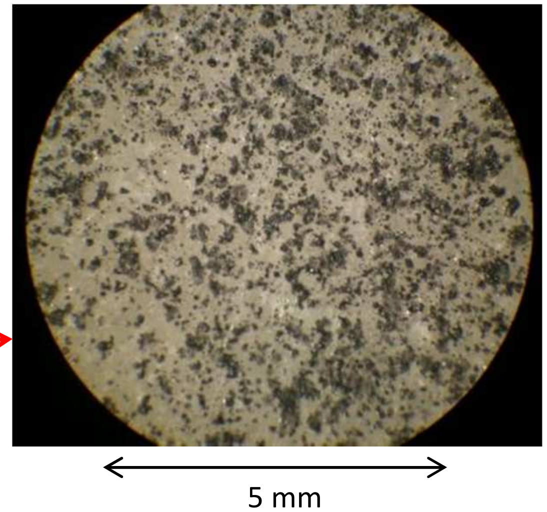
CorrelManuV© (M.Bornert)

# Displacement surface markers

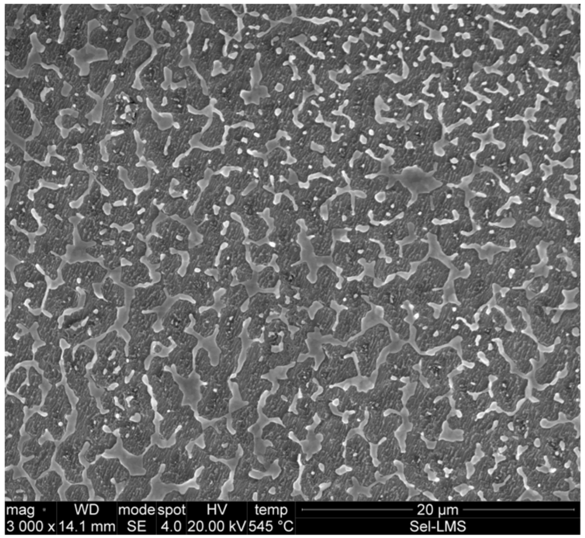


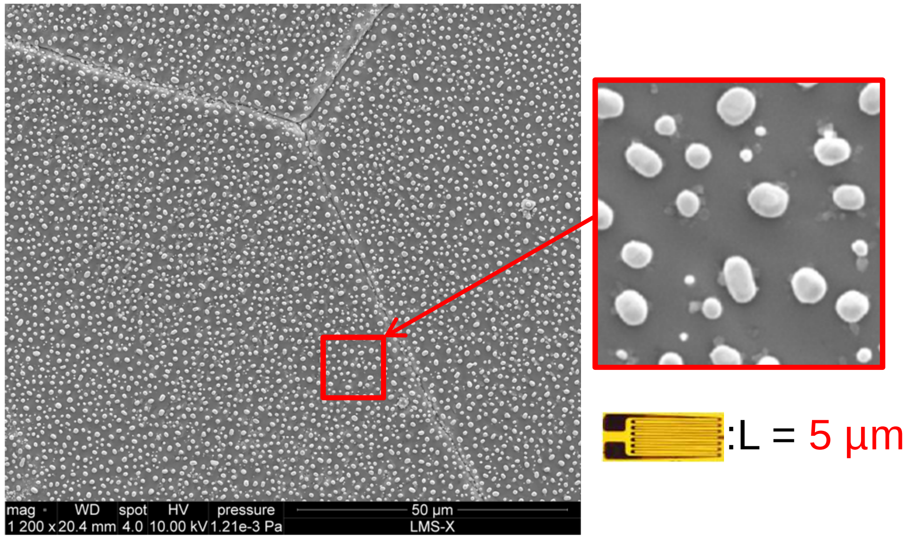

Paint Spray
(50 - 100 µm)




 :L = 250 µm



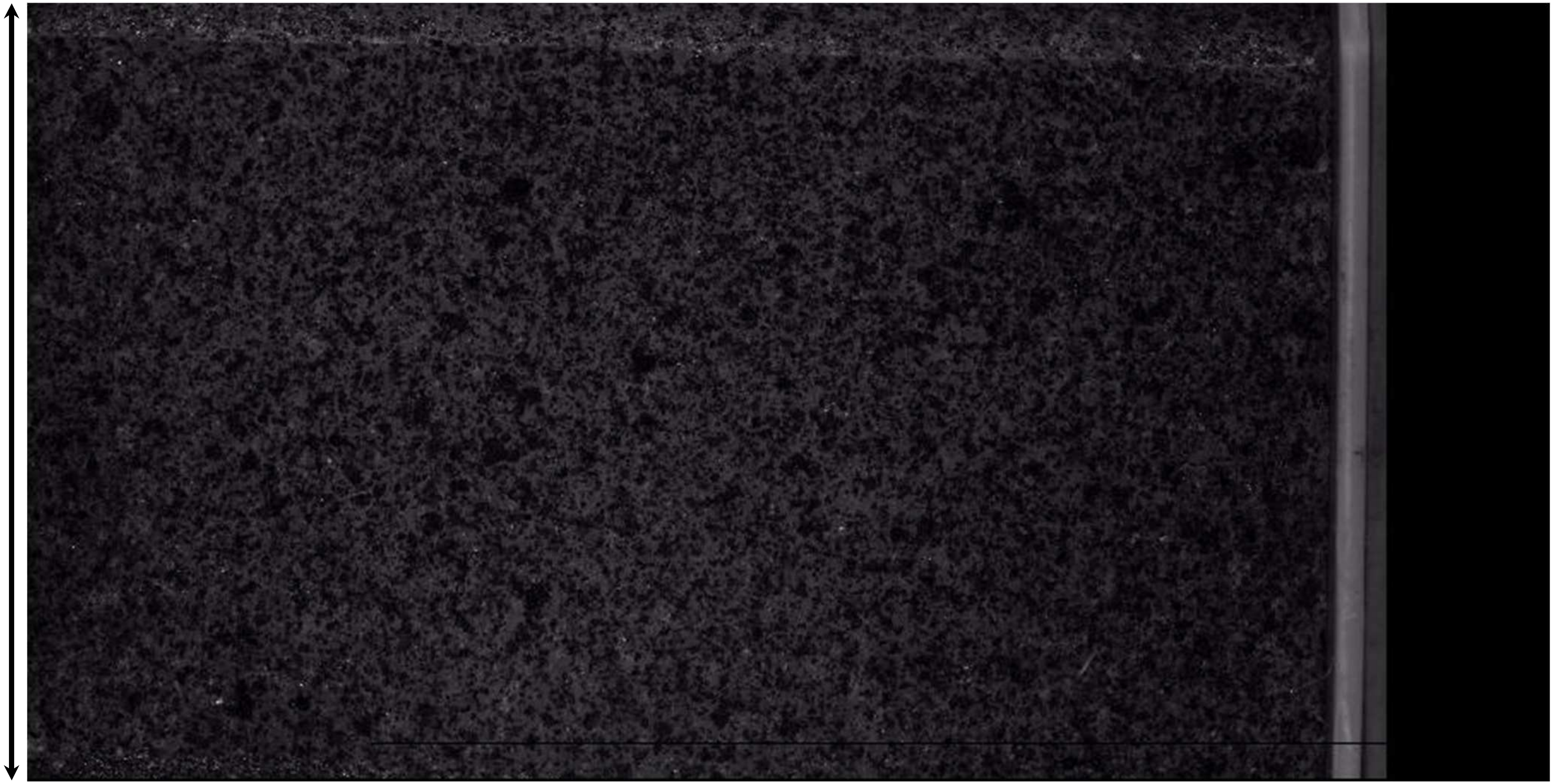




:L = 5 µm

Dewetting of thin gold film at 550°C: Gold spheres (1-2 µm)

## « In-situ » OM - FFM - DIC

Sample: flattened cylinder
(h=24mm, D=22 mm)
Measurement basis: L = 250 µm

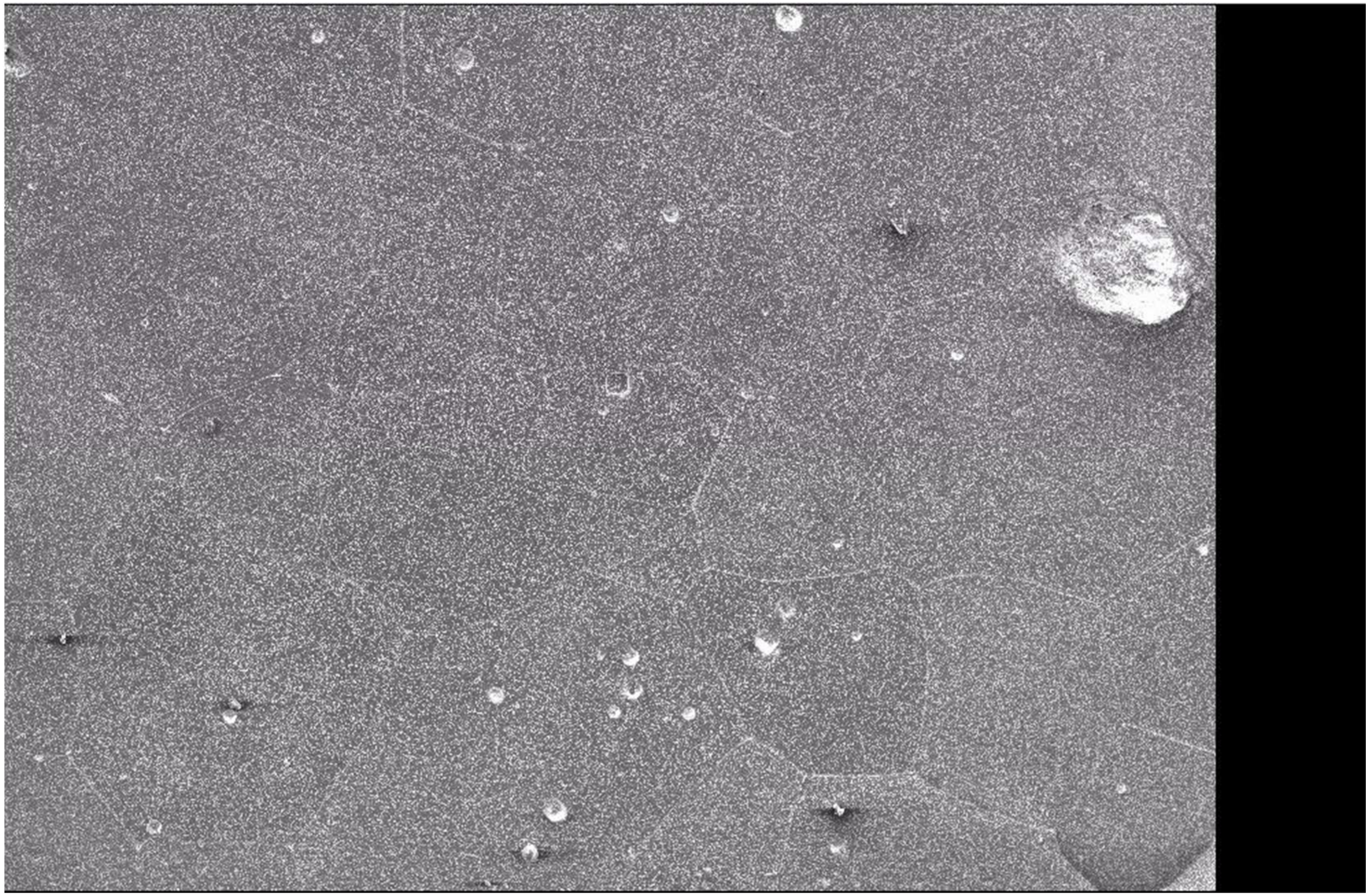


15mm

Macro field is heterogeneous:
1) Imperfect uniaxial loading: fretting effects.
2) Structure effects.

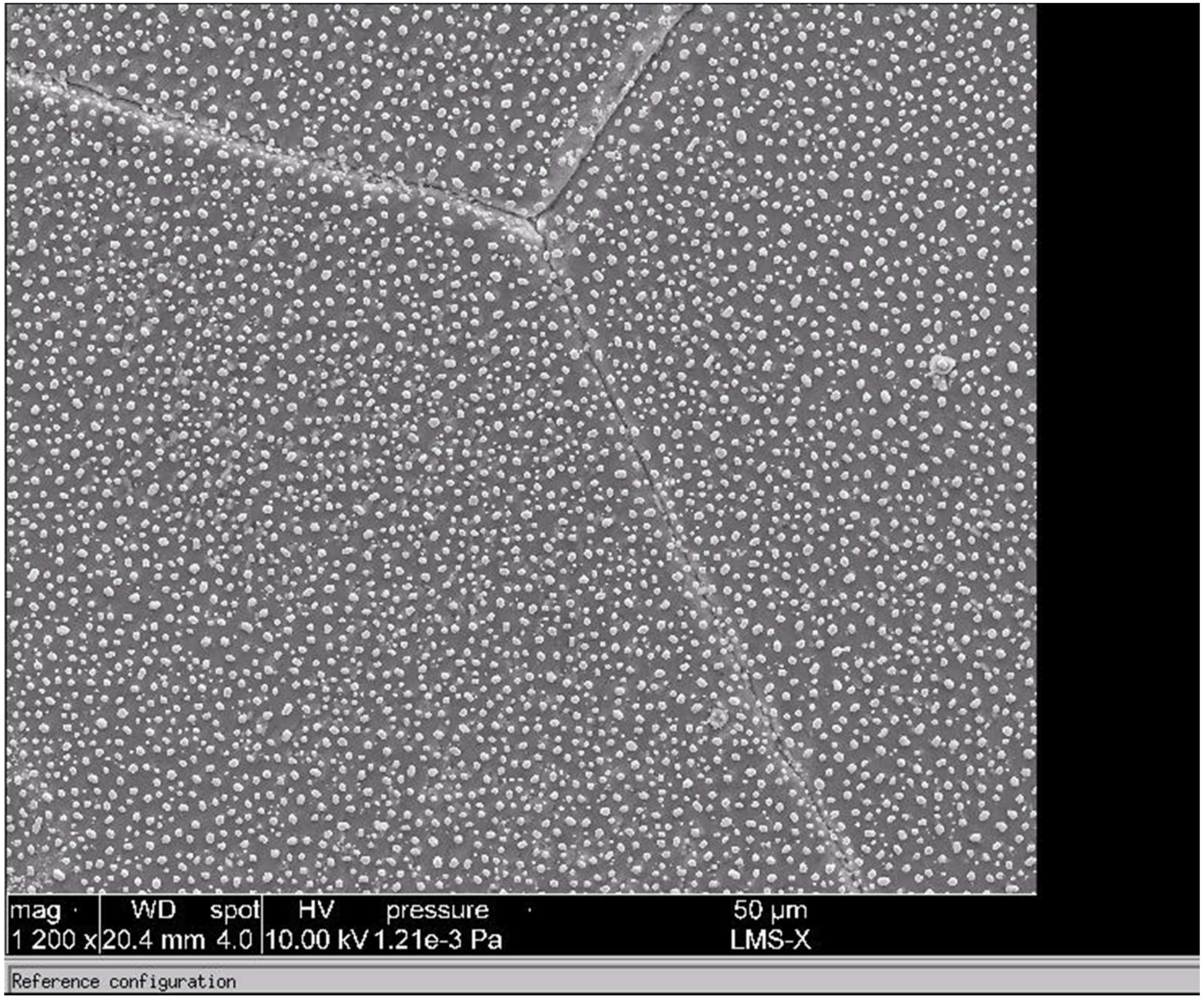
(final macro strain ~5%)

« In-situ » SEM - FFM - DIC Coarse grains (200 - 500 µm) Strain rate = ~ $10^{-4}s^{-1}$



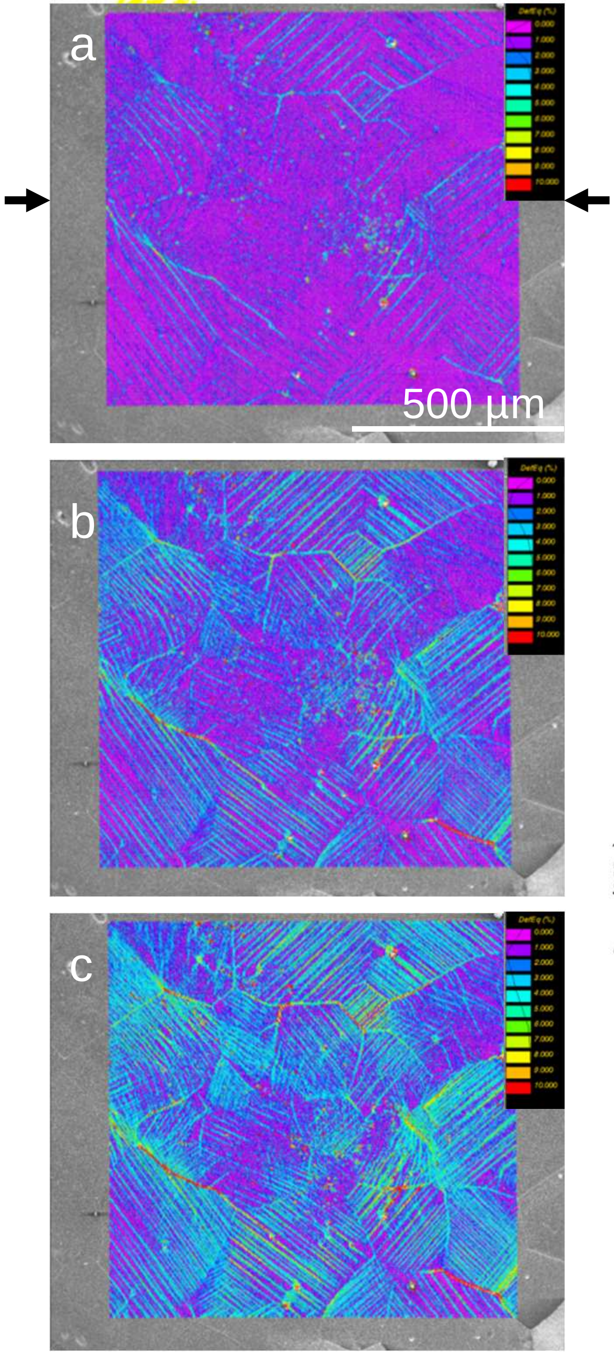
500µm

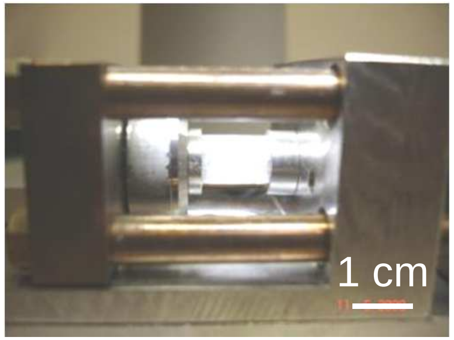
« In-situ » SEM - FFM - DIC Coarse grains (200 - 500 µm) Strain rate = ~ $10^{-4}s^{-1}$




50µm

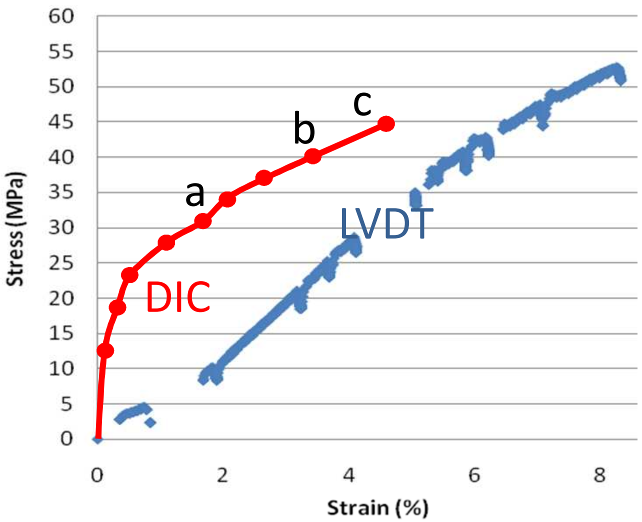
# « In-situ » SEM - FFM - DIC



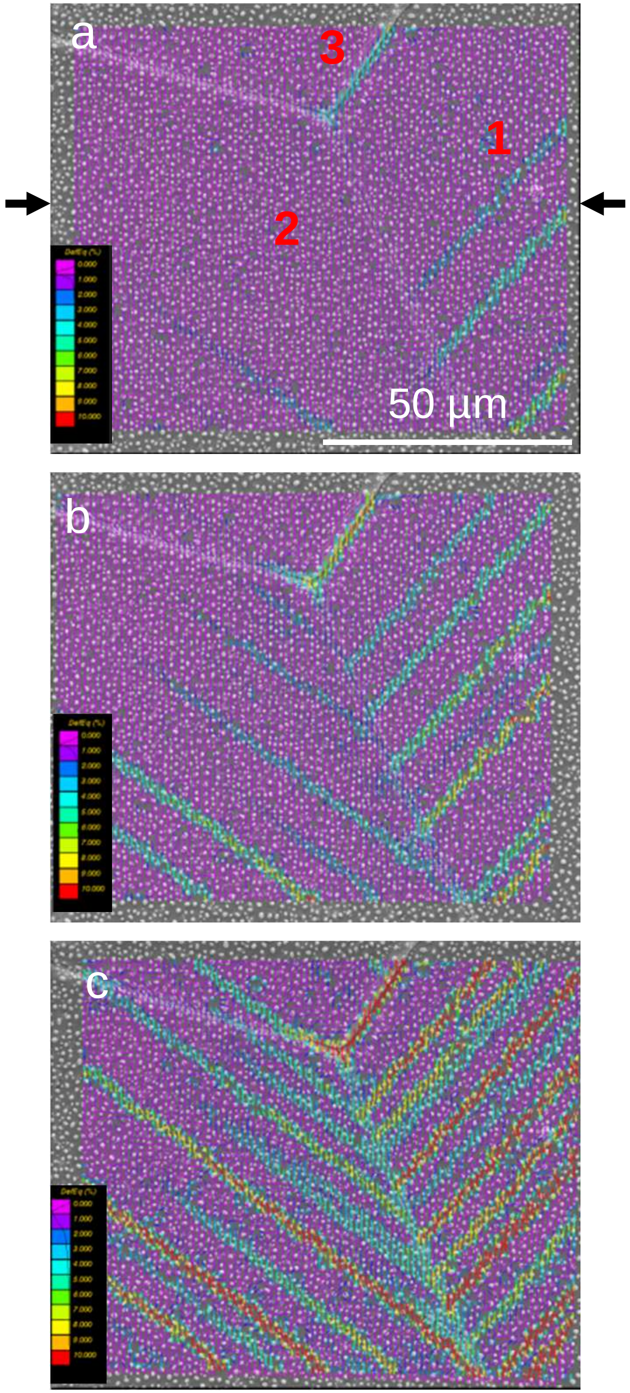



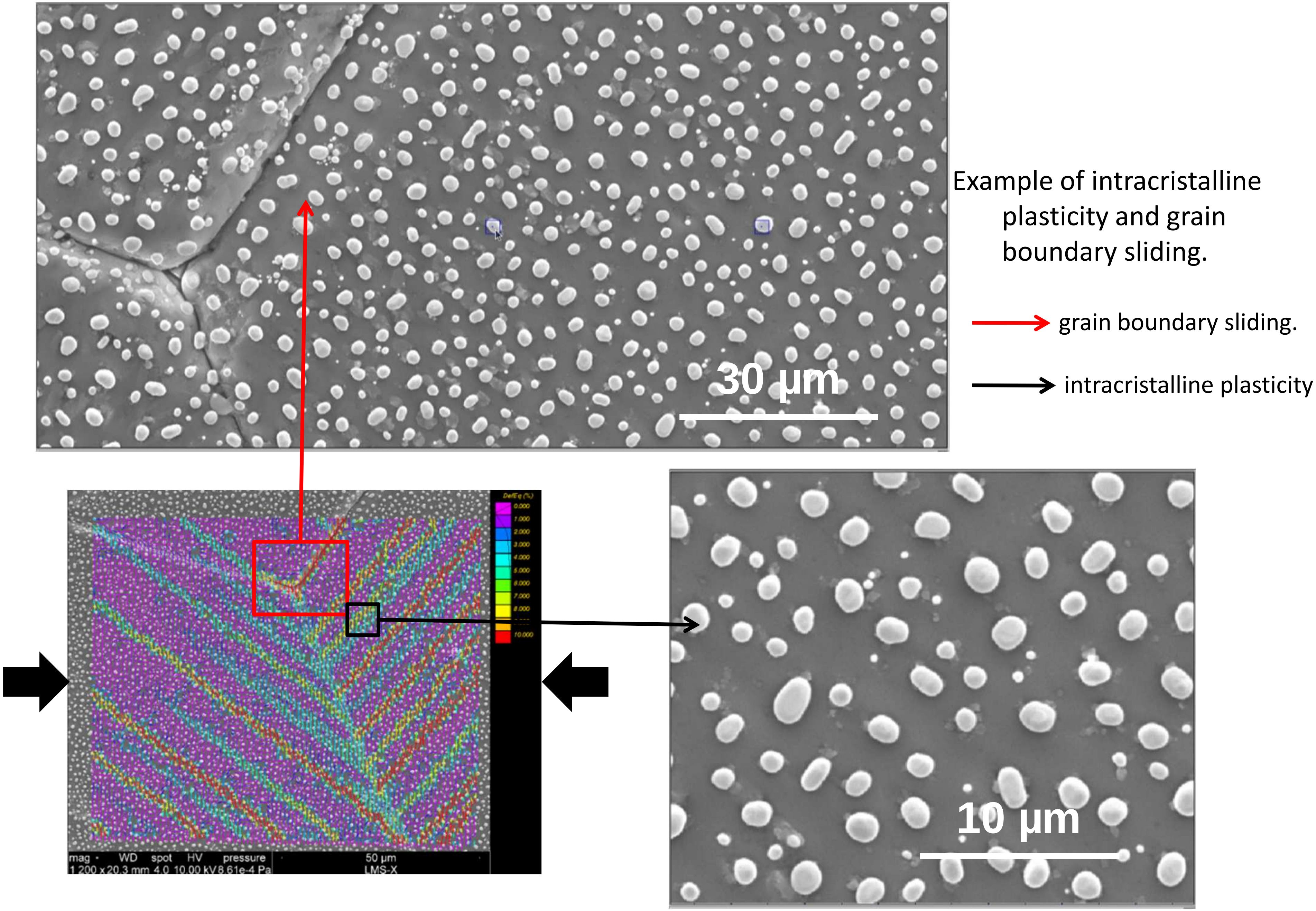

Large grains (200 - 500µm)

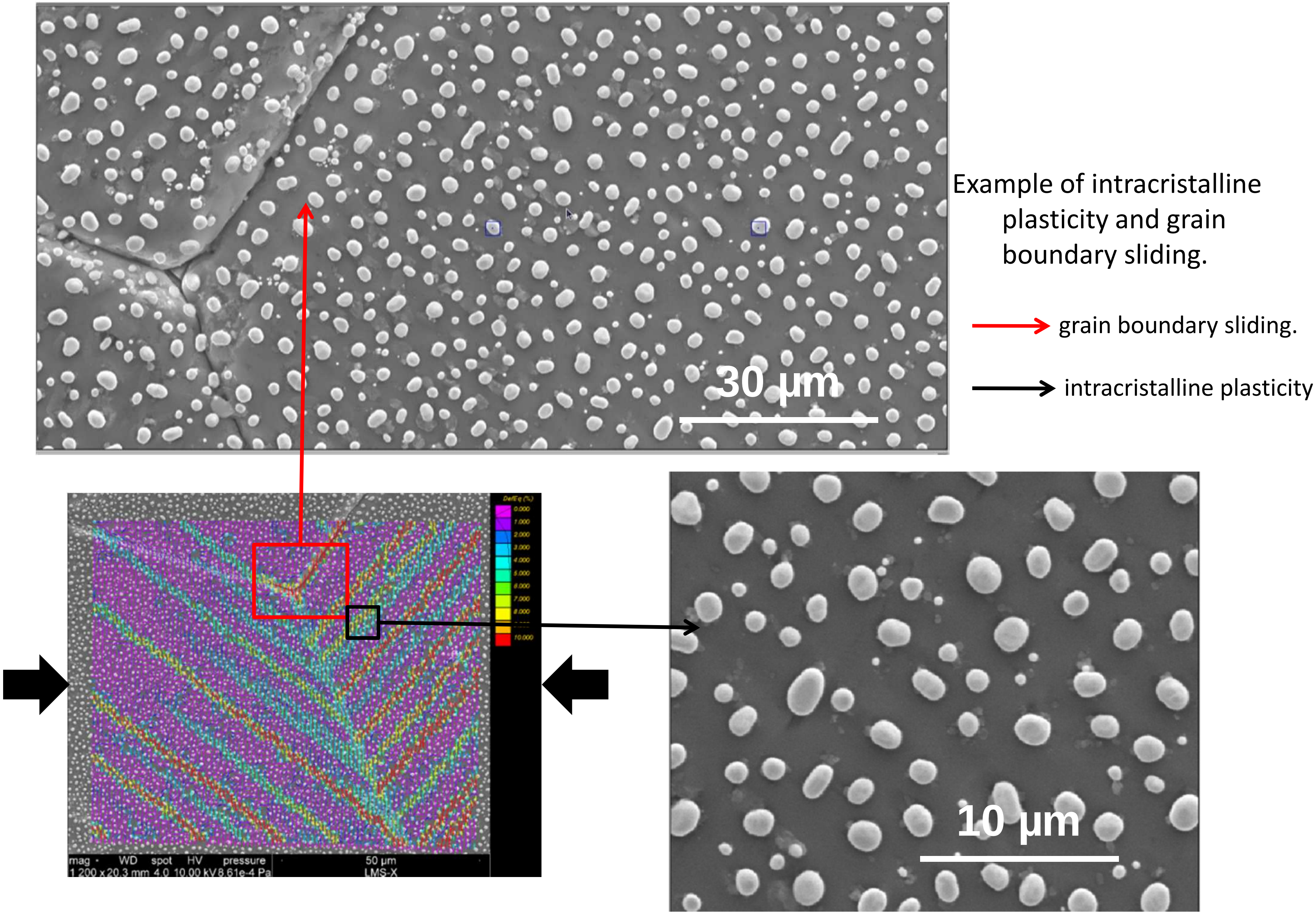
Strain rate = ~ $10^{-4}s^{-1}$




**10 % strain**
**locallizes at interfaces.**




30 µm
10 µm
Example of intracristalline plasticity and grain boundary sliding.
grain boundary sliding.
intracristalline plasticity
mag WD spot HV pressure
1 200 x 20.3 mm 4.0 10.00 kV 8.61e-4 Pa
50 µm
LMS-X

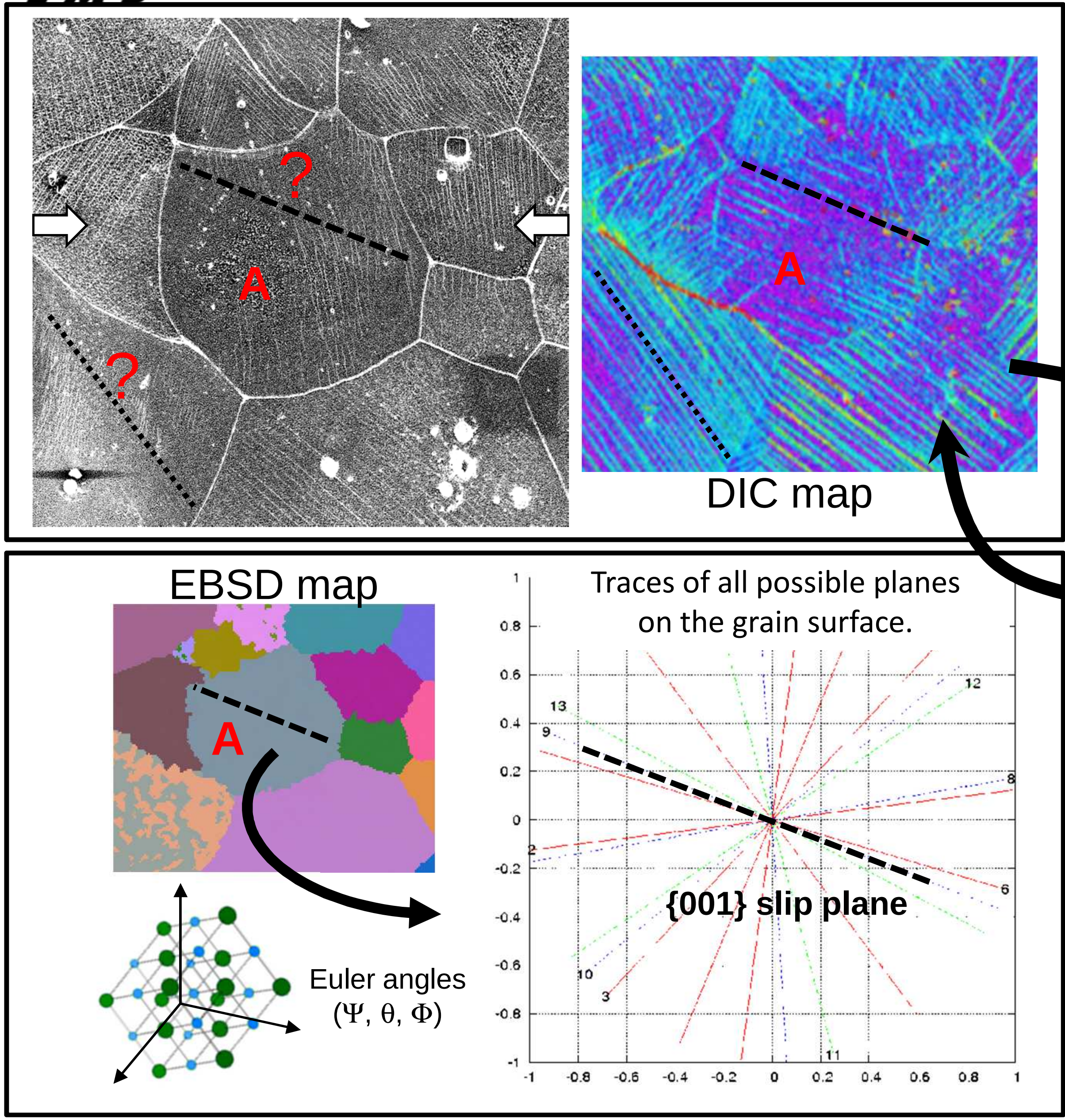


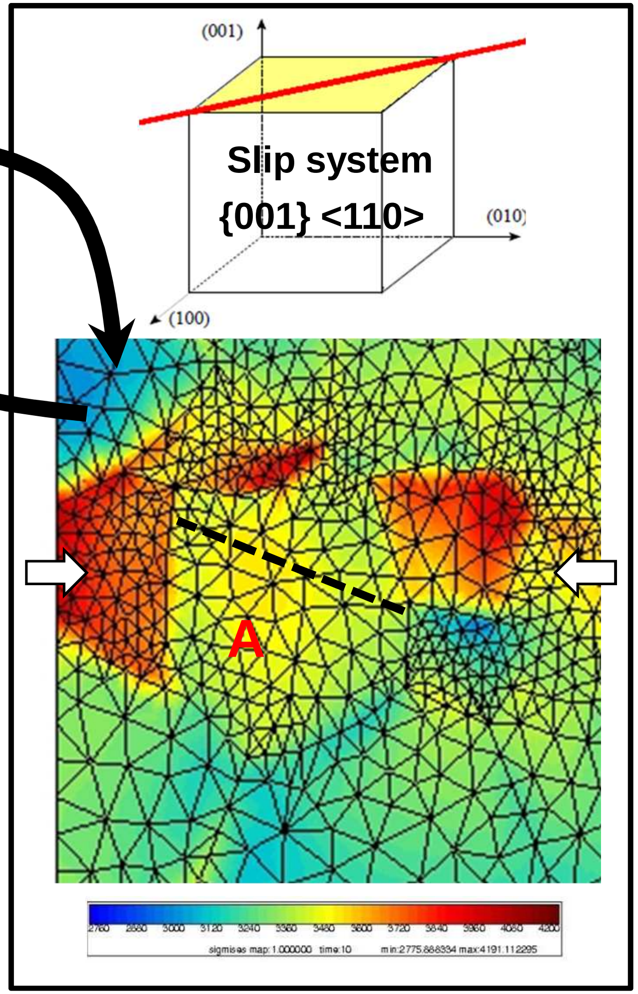

Example of intracristalline plasticity and grain boundary sliding.

# Identification of slip systems

$$\sigma_{ij}^{s} = \begin{pmatrix} \sigma_{xx} & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix} \qquad S_{ij}^{s} = \begin{pmatrix} S_{xx} & S_{xy} & S_{xz} \\ S_{yx} & S_{yy} & S_{yz} \\ S_{zx} & S_{zy} & S_{zz} \end{pmatrix}$$

**<110> slip direction**

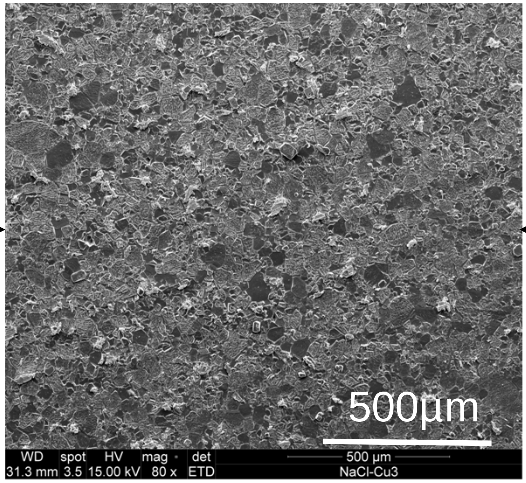


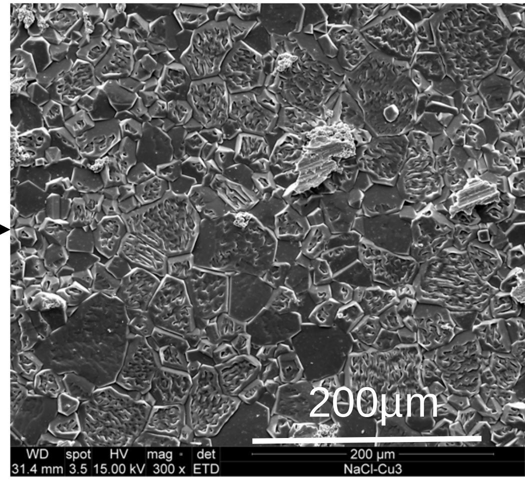



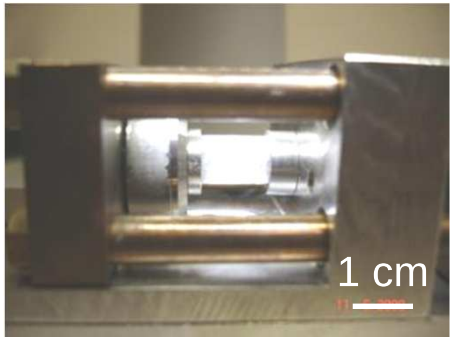
# « In-situ » SEM - FFM - DIC

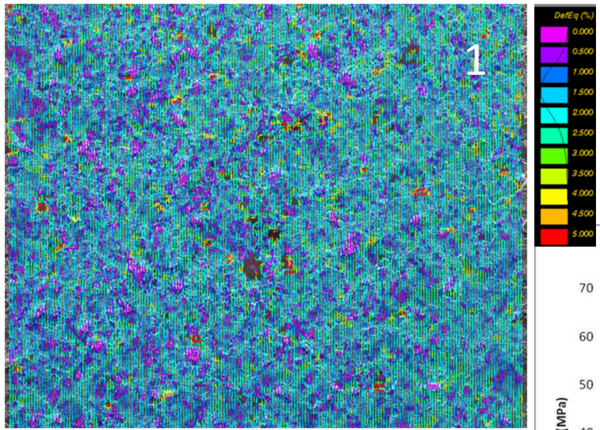
Fine grains (< 100 μm)

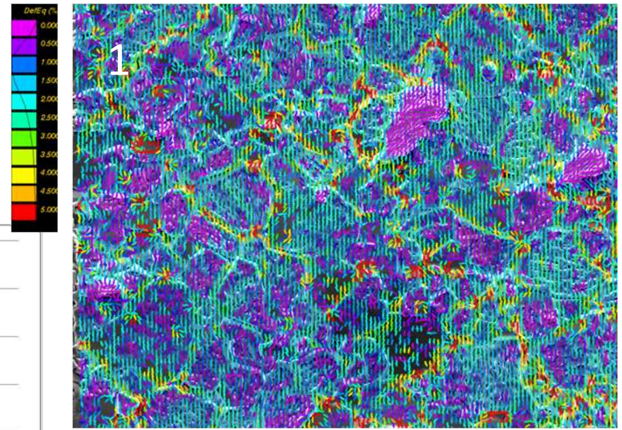
Strain rate = ~ $10^{-4} s^{-1}$

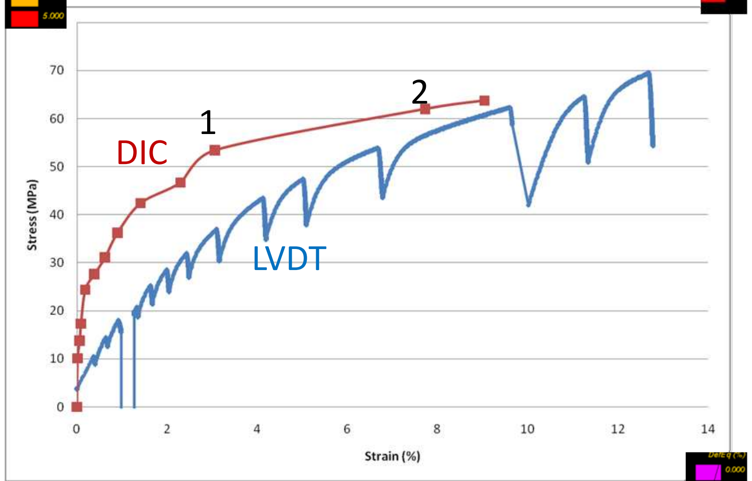


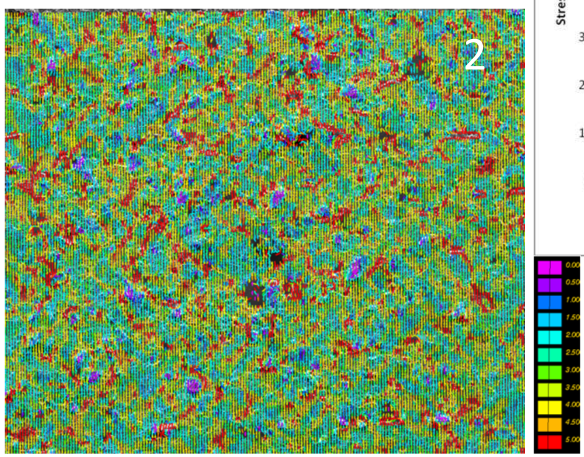



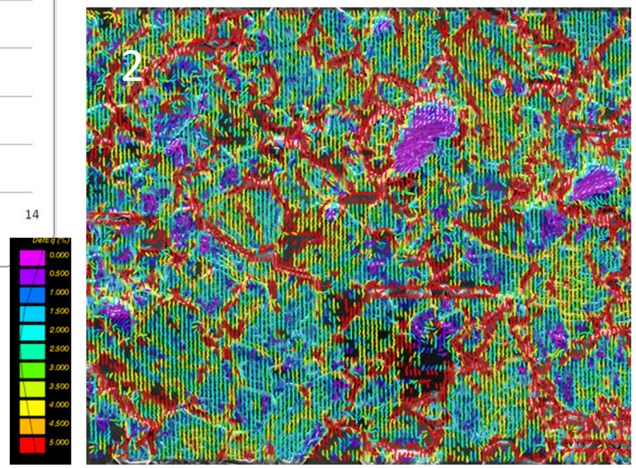



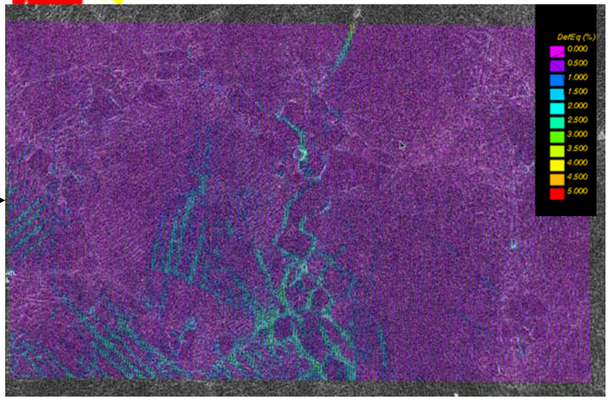



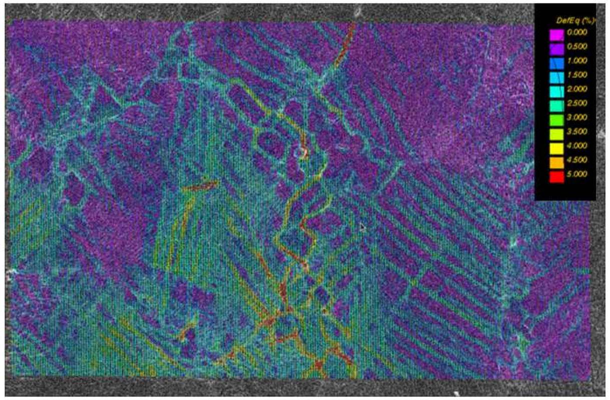



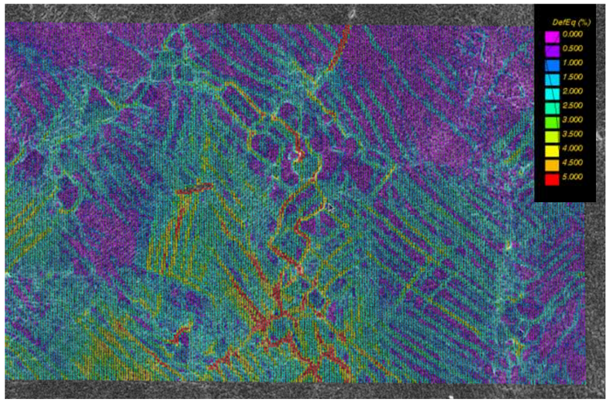




**50 % strain**
**locallizes at interfaces.**

# « In-situ » SEM - FFM - DIC

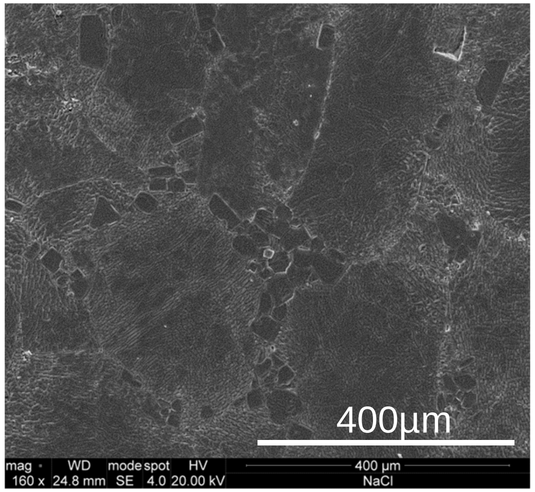


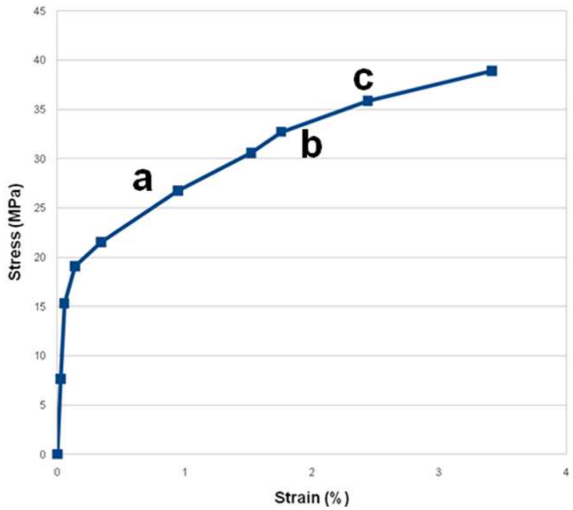


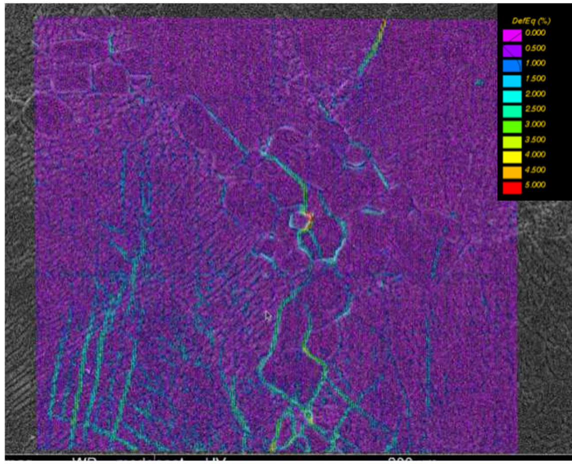


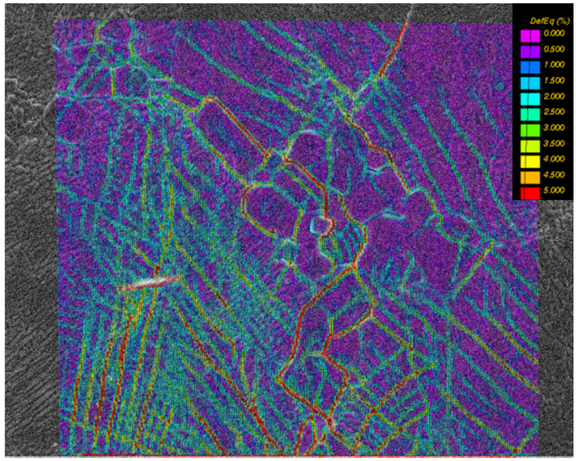
Bimodal grain-size distribution
(< 100 and 200-400 μm)

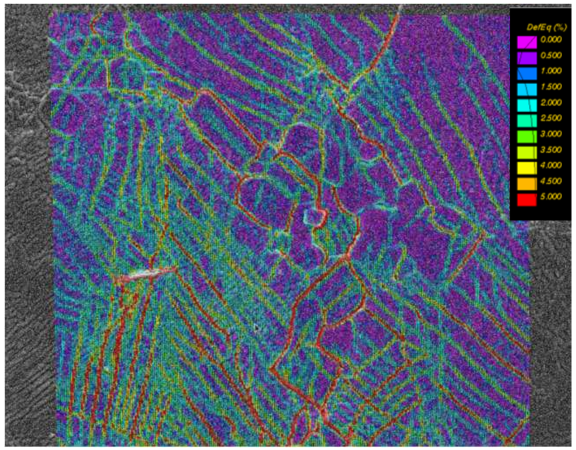
Strain rate = ~ $10^{-4}s^{-1}$



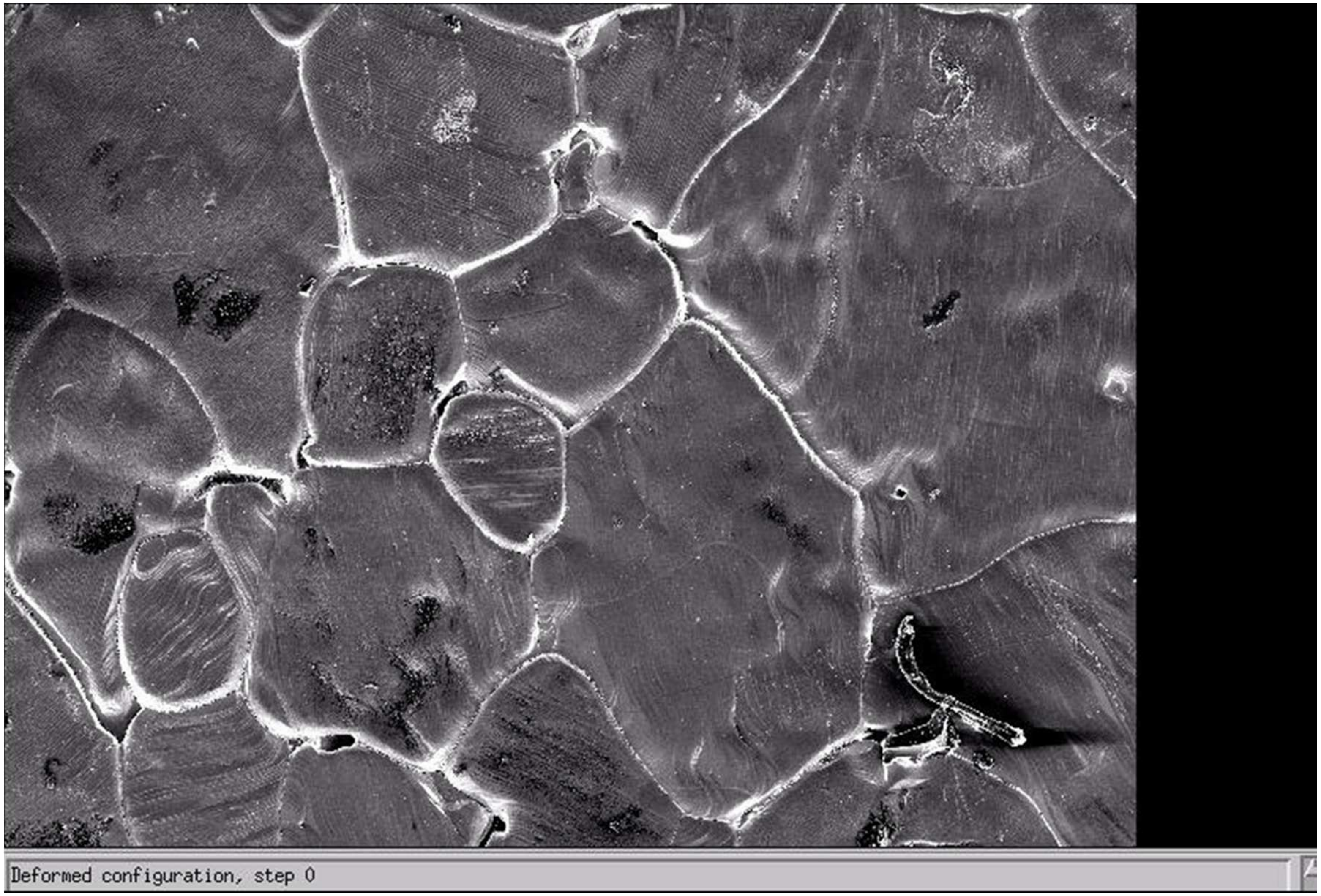



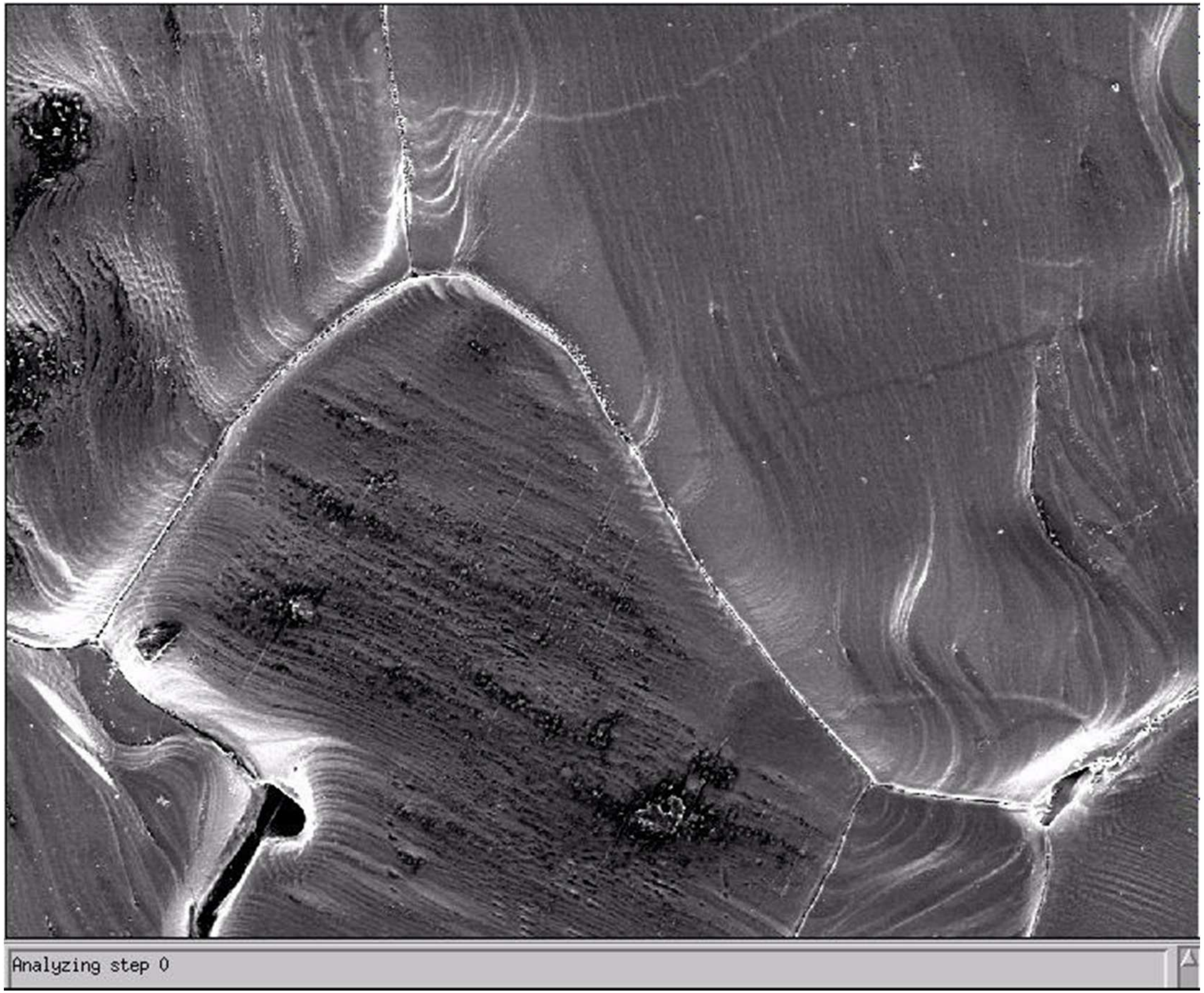




# Interfacial locallization, CSP - GBS interactions and damage



## Interfacial locallization, CSP - GBS interactions and damage



# *Conclusions*

**Micromechanical testing coupled with FFM-DIC allowed:**

→ **Identification of 2 mechanisms of viscoplastic deformation**
-CSP: Crystal slip plasticity (dislocation glide): main mechanism.
-GBS: Grain boundary sliding : secondary (but necessary) mechanism, accommodating for local incompatibilities of CSP.

→ **Quantification of their respective contributions to total strain,** which depend on grain size and its distribution.

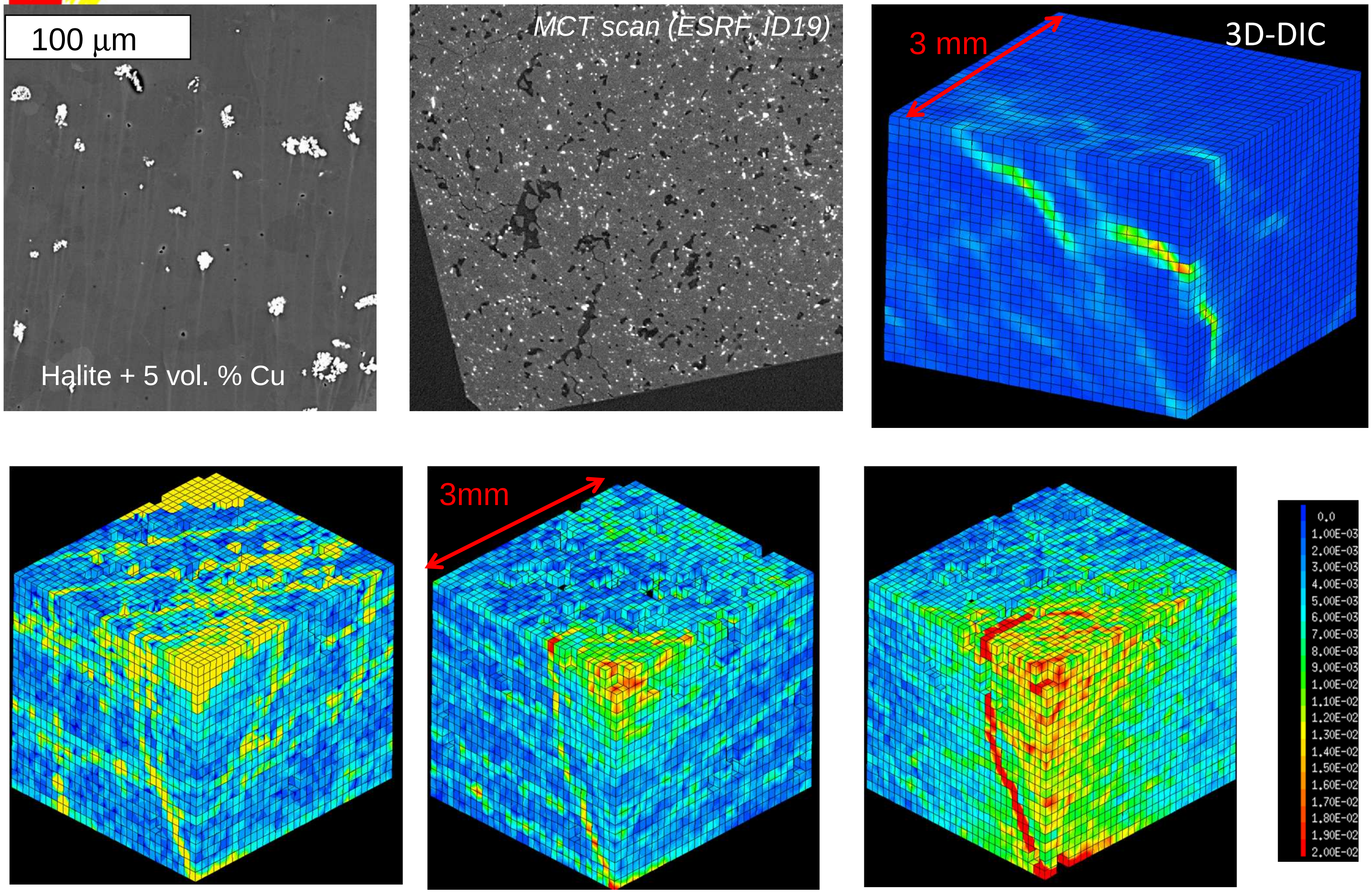
→ **Identification of the active slip systems,** which are not only the easiest ones: local stress states deviate from the macroscopic stress state.

→ **Stress and strain heterogeneities relate to microstructure,** viscoplastic anisotropy, interplay of co-operational mechanisms…

**Next steps:**

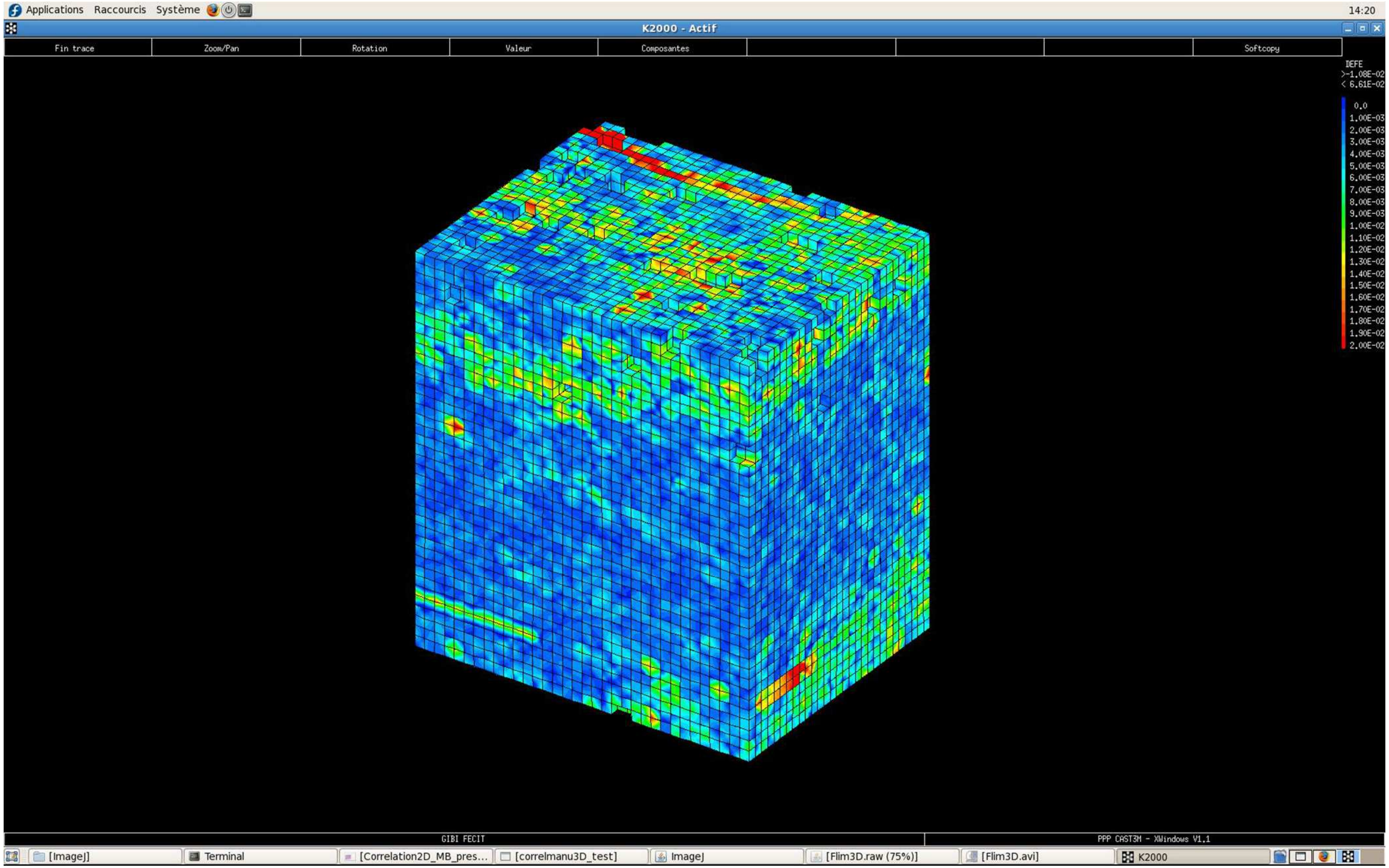
Implementation of GBS in polycrystal numerical modeling.
3D DIC: volume strain fields.

# 3D Full field measurement (preliminary results)




Voxel size 5 µm, Size of the correlation domain $20^3 \sim 100^3$µm

# 3D Full field measurement



# 3D Full field measurement