MECANISMES ACTIFS DANS LE SEL GEMME : QUANTIFICATIONS EXPERIMENTALES POUR UNE MODELISATION PERTINENTE

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dépasser les frontières
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IFSTTAR
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Why do we want to study the plastic behavior of rock salt ?

Importance of NaCl

From

Geology and in-situ problems

Creep of salt Glacier

Landsat image of two salt glaciers that formed when salt domes erupted from the flanks of mountains in the Zagros fold belt of Iran. The salt glacier on the left is flowing south. The one on the right is flowing north. Each glacier is about four miles long from head to toe.

Stability of salt cavities

Sinkhole linked to solution related subsidence in salt

To

Fundamental studies

Salt as an ionic crystal

- *Plasticity of Polycrystalline Ionic Solids (Skrotzki et al., 1981, phys. stat. sol.)*
- *Dislocations in Ionic Crystals (Castaing, 1980, j. phys.)*
- *Dissociation of Dislocations and Plasticity of Ionic Crystals (Haasen, 1974, j. phys.)*

Salt as laboratory analog material

- *Temperature dependent grain boundary migration in deformed-then-annealed material: Observations from experimentally deformed synthetic rocksalt (Piazolo et al., 2006, tectonophys.)*
- *Influence of crystal plastic deformation on dilatancy and permeability development in synthetic salt rock (Peach & Spiers, 1995, tectonophys.)*

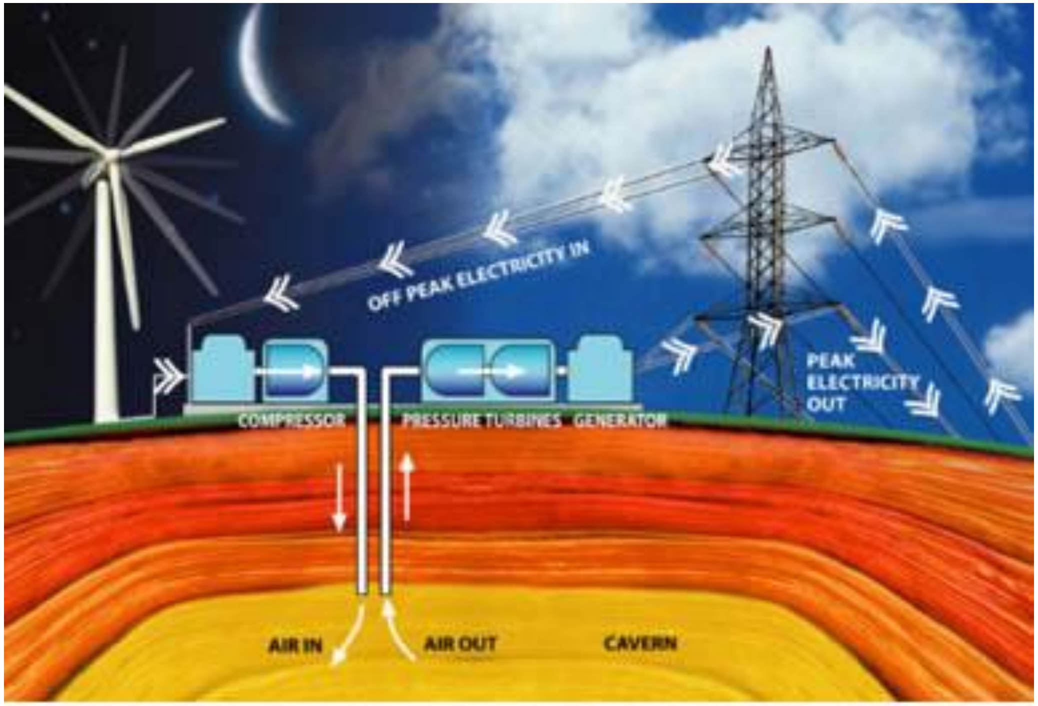
Salt as a multipurpose storage medium

- Cyclic loading, small loads, temperature effects, creep, plasticity, damage

New importance of Salt for storage in natural or man-made cavities

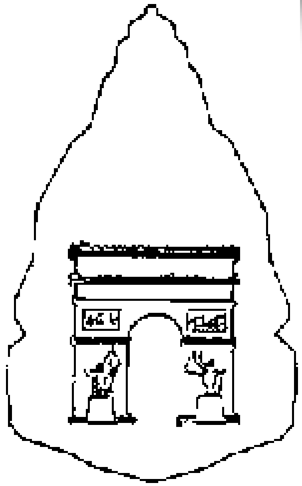
Nuclear Waste (in salt mine)

Energy storage (compressed air)



From www.reuk.co.uk/Storing-Wind-Power-with-Compressed-Air.htm

Size example of man-made cavity



What new developments have taken place since the plasticity studies of the 1980's ?

- Multiscale mechanical testing with continuous observations
  - ➢ Optical or Scanning Electron Microscopy
- 3D imaging techniques
  - ➢ absorption or diffraction,
  - ➢ synchrotron or lab tomograph
- Finite element computations accounting for 3D structures
  - ➢ Crystal plasticity (CPFEM) and more

Digital Image Correlation Techniques

Is salt a "good material" ?

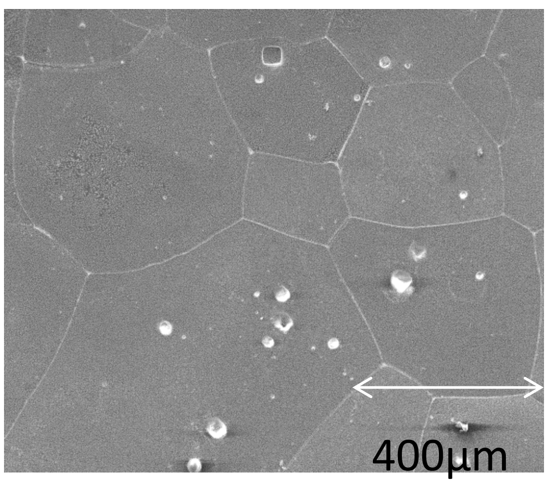
Yes

- Available, and cheap;
- Relatively easy to synthetize and prepare;
- Control of microstructures (grain sizes, texture);
- Crystalline material (cubic symmetry) : electron and Xray diffraction;
- Mechanical properties : ultimate strain, ultimate strength, strain rates, temperatures;

But nothing is perfect …

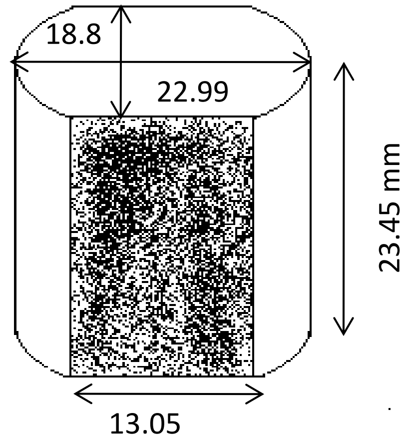
The material

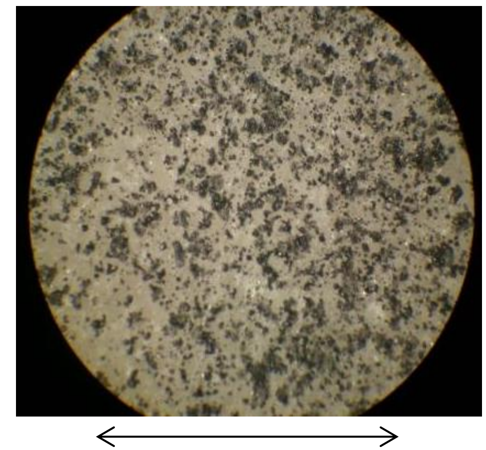
Synthetized by HIP + Annealing, starting with a pure and fine NaCl powder



Equilibrated structure,
almost straight GBs

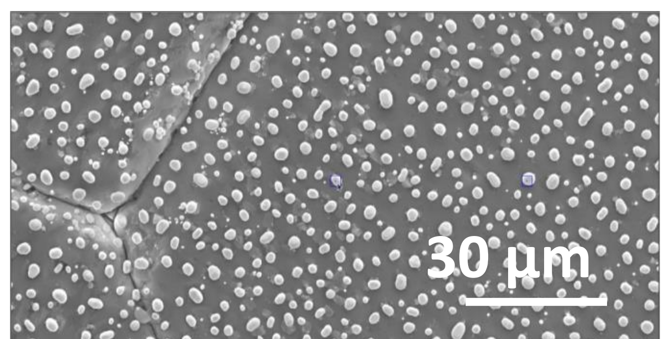
Surface marking by paint droplets *mm scale*

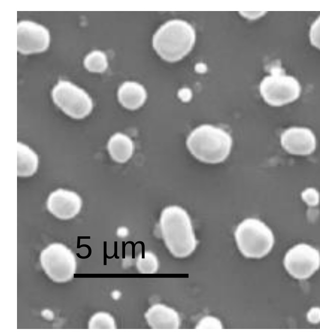




5 mm

or gold particles *µm scale*

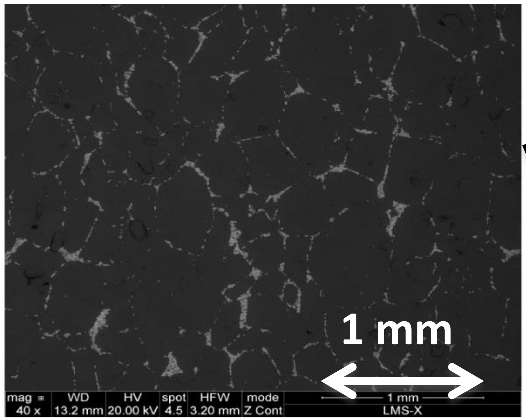


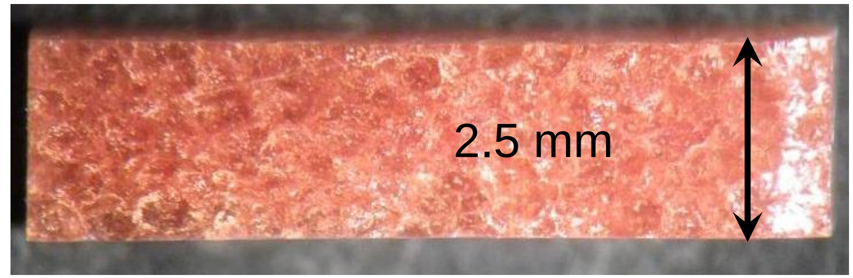


For 3D studies :

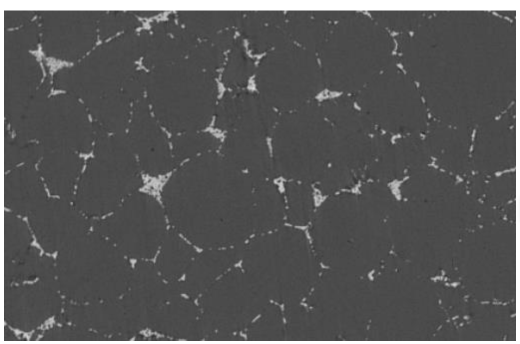
same HIP technique + mixing of 2% fine copper powder

SEM 2D images





µCT 3D images

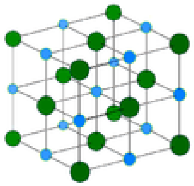


Synthetic halite (50-300µm) + <u>copper particles</u> (5 to 20µm) at grain boundaries

For small grained material (50 to 80 µM), copper particles maybe inside the grains or at GBs

Annealing to grow large gains => Cu particles tend to migrate to GBs

NaCl Single Crystal



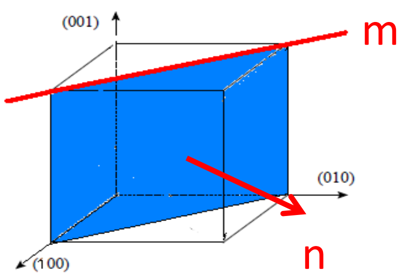
NaCl is a Ionic Crystal made of the two Cubic Lattices of Na^+ and Cl^-

Instead of a single family of glide systems like fcc metals {111} <110>

3 families have been identified

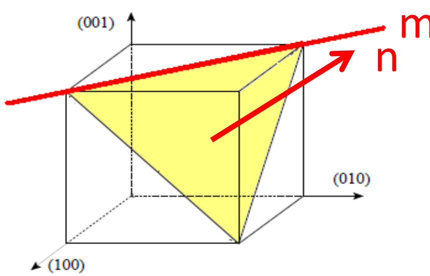
- {110} <110> 6 systems but only 3 independent ones (dodecahedric)
- {111} <110> 12 systems (octahedric)
- {001} <110> 6 systems (cubic)

Total : 6 directions 13 planes



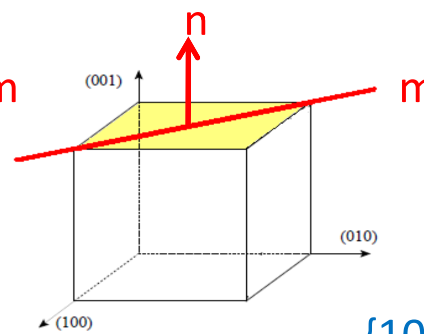
{110} <110>

Dodecahedric



{111} <110>

Octahedric



{001} <110>

Cubic

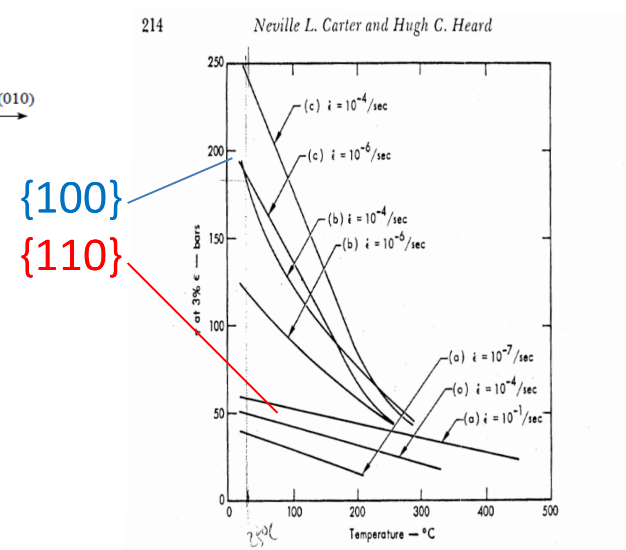
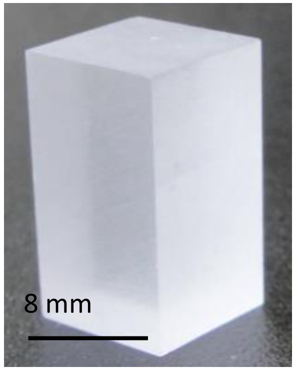


Fig 7. Resolved shear stress (3 percent strain) for observed slip systems versus temperature. (a) {110} <1$\bar{1}$0>, (b) combined {111} <1$\bar{1}$0> and {110} <1$\bar{1}$0>, and (c) {100} <110>.

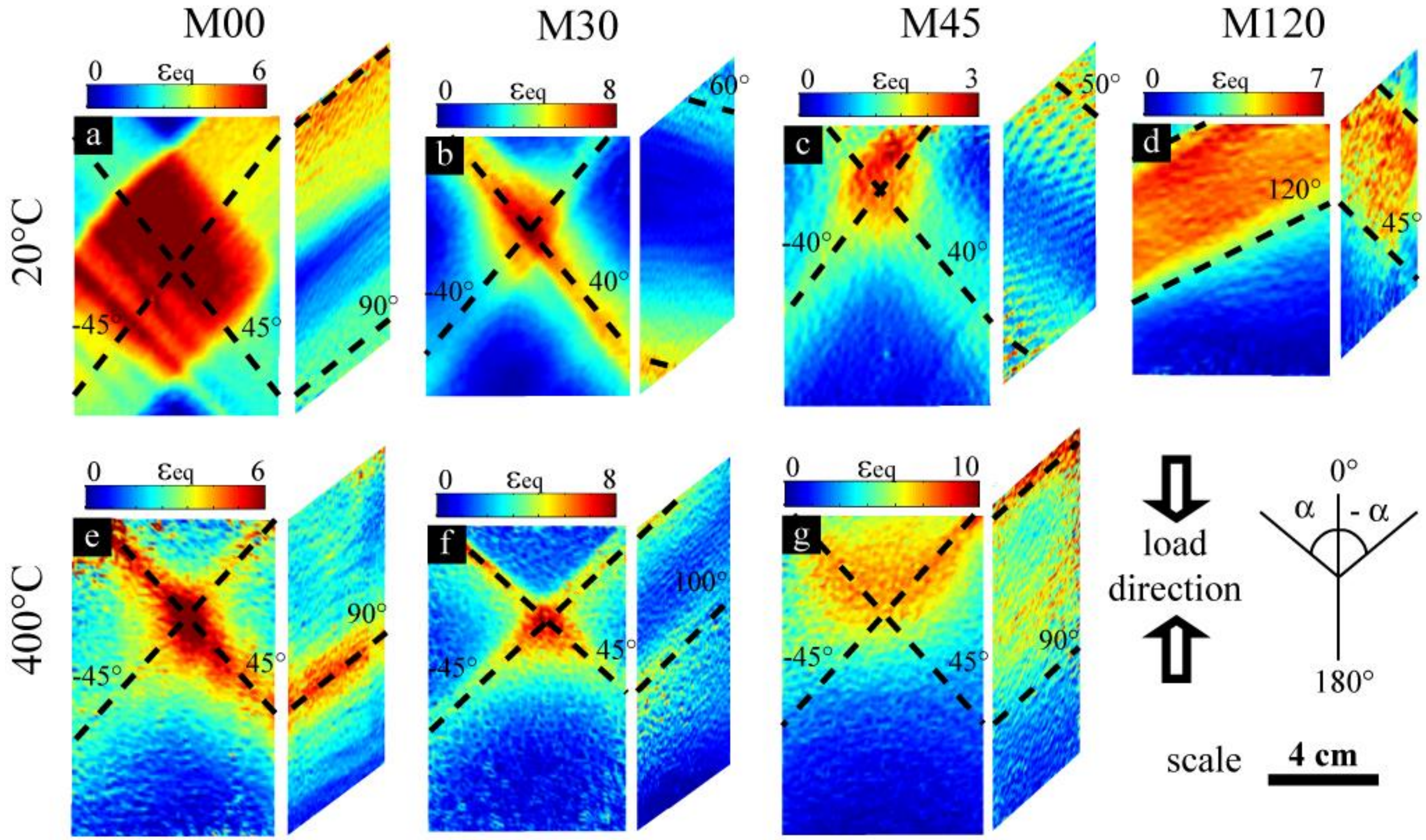
Each family has a different initial critical shear with a strong temperature dependence

Check the properties by doing our own study on single crystals deformed in uniaxial compression at different T

Test of 4 orientations in uniaxial compression

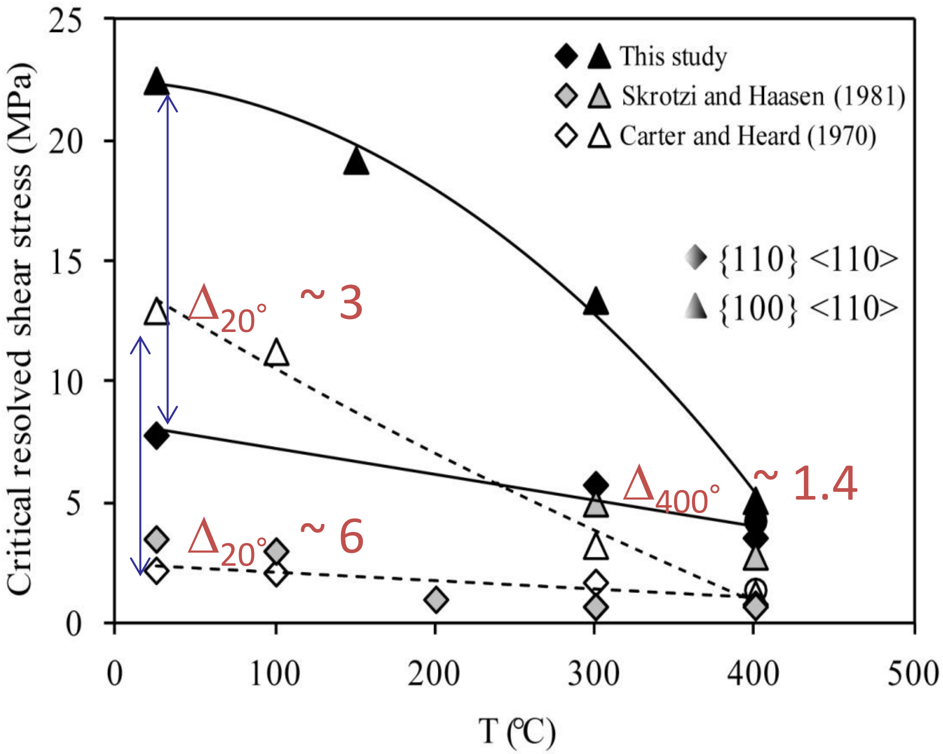


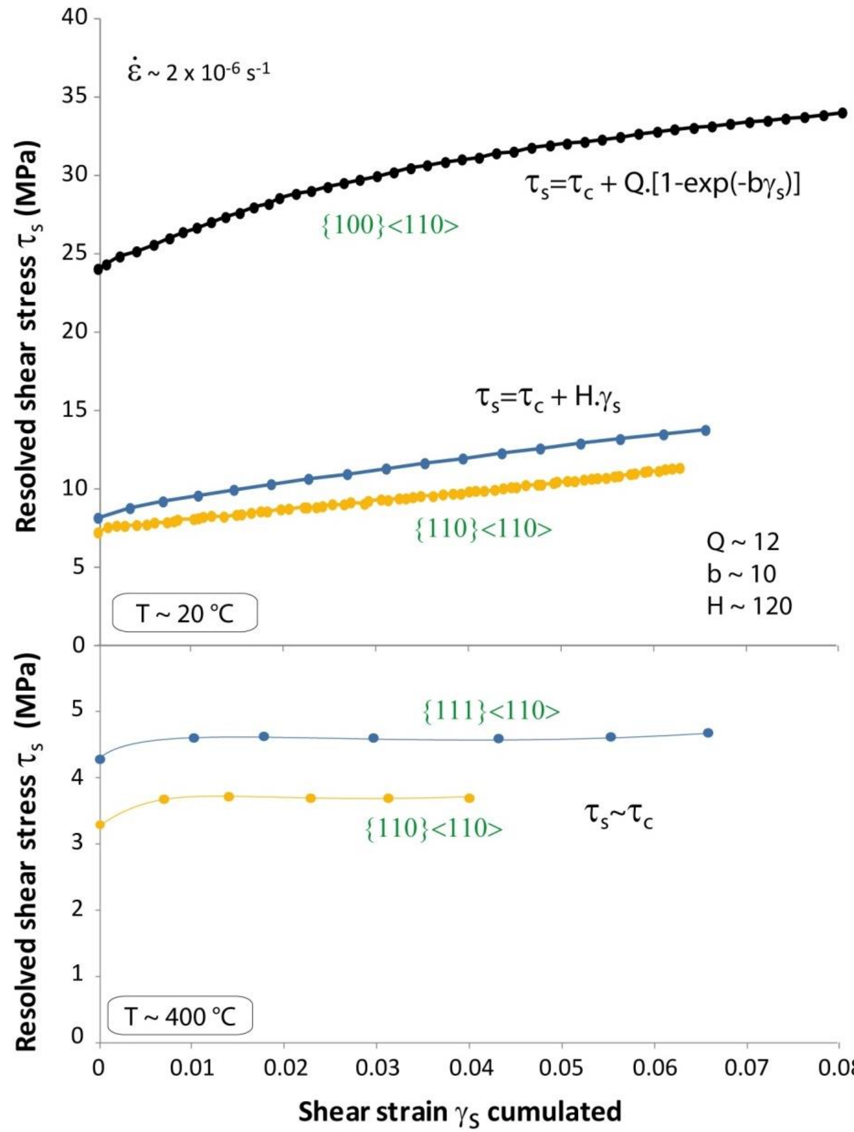
Simultaneous observations of 2 sides
+
Use of Digital Image Correlation to compute equivalent strain fields



M00	M30	M45	M120
{110}<110> {110}<110> Sf=0.5	{110}<110> Sf=0.37 {111}<110> Sf=0.48	{110}<110> Sf=0.25 {111}<110> Sf=0.41	{100}<110> Sf=0.48

NaCl Single Crystal



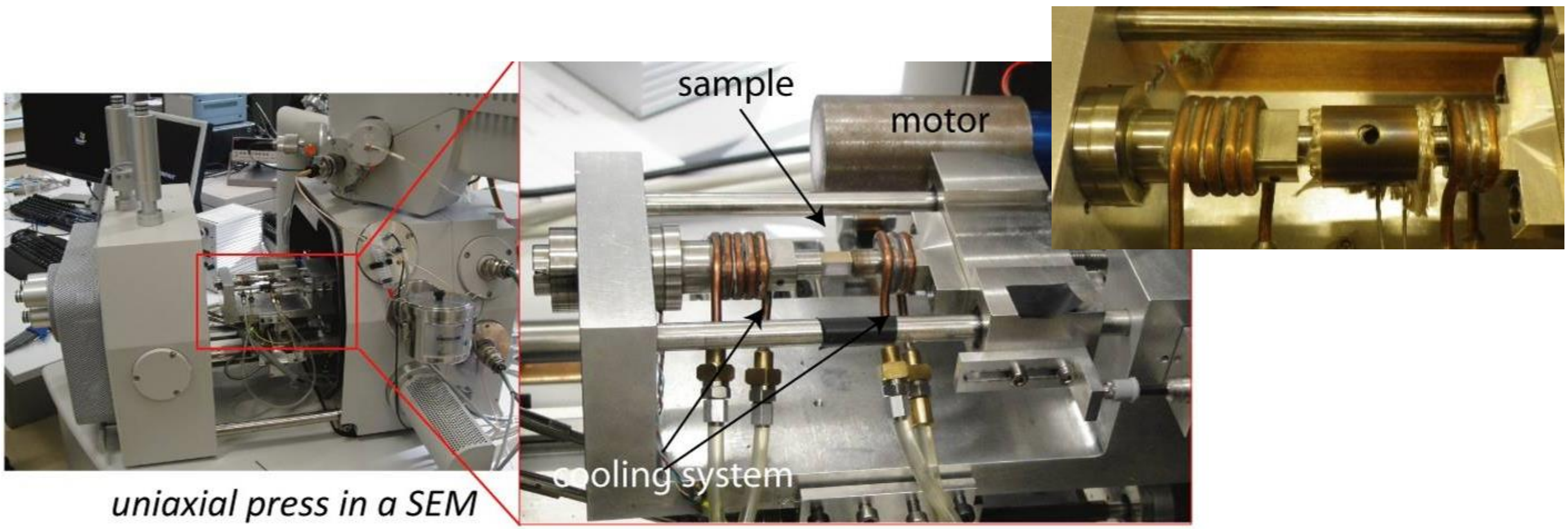


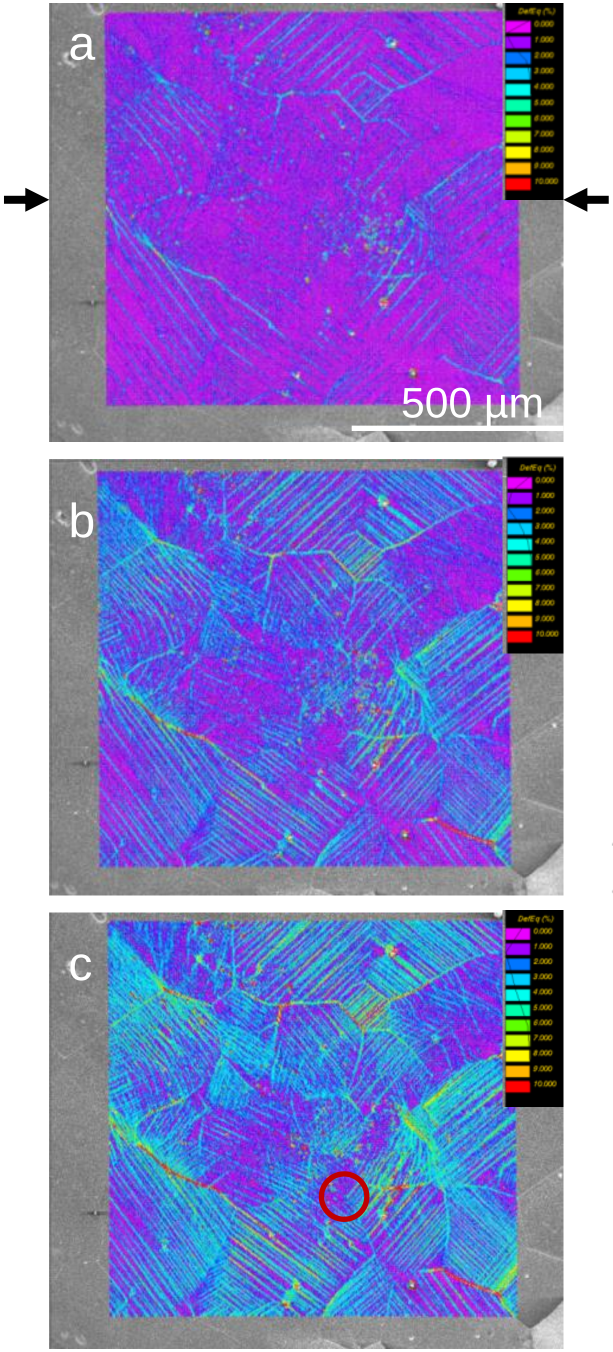
Initial Critical Shears are such that:

At 20°C not enough "easy" systems available

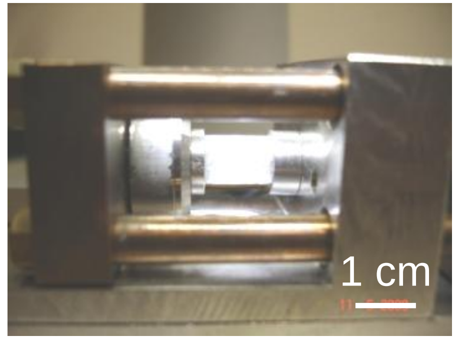
What consequence for the polycrystal?

Uniaxial compression of NaCl polycrystalline sample at 20°C and 300°C in a SEM



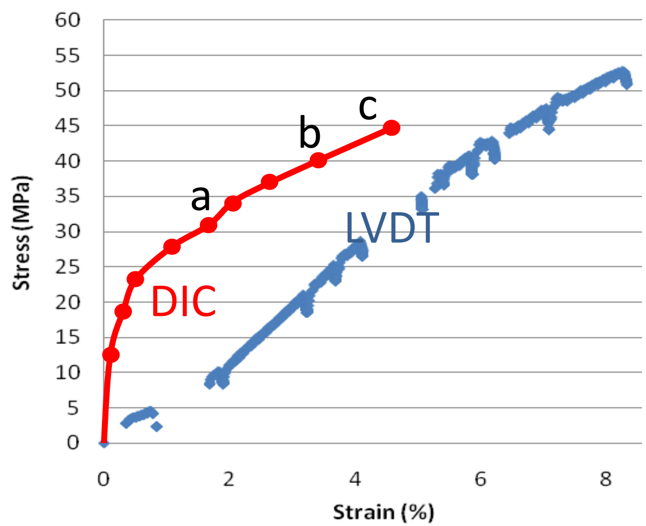


« In-situ » SEM - FFM - DIC



Large grains (200 - 500µm)

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



10 % strain locallizes at interfaces.

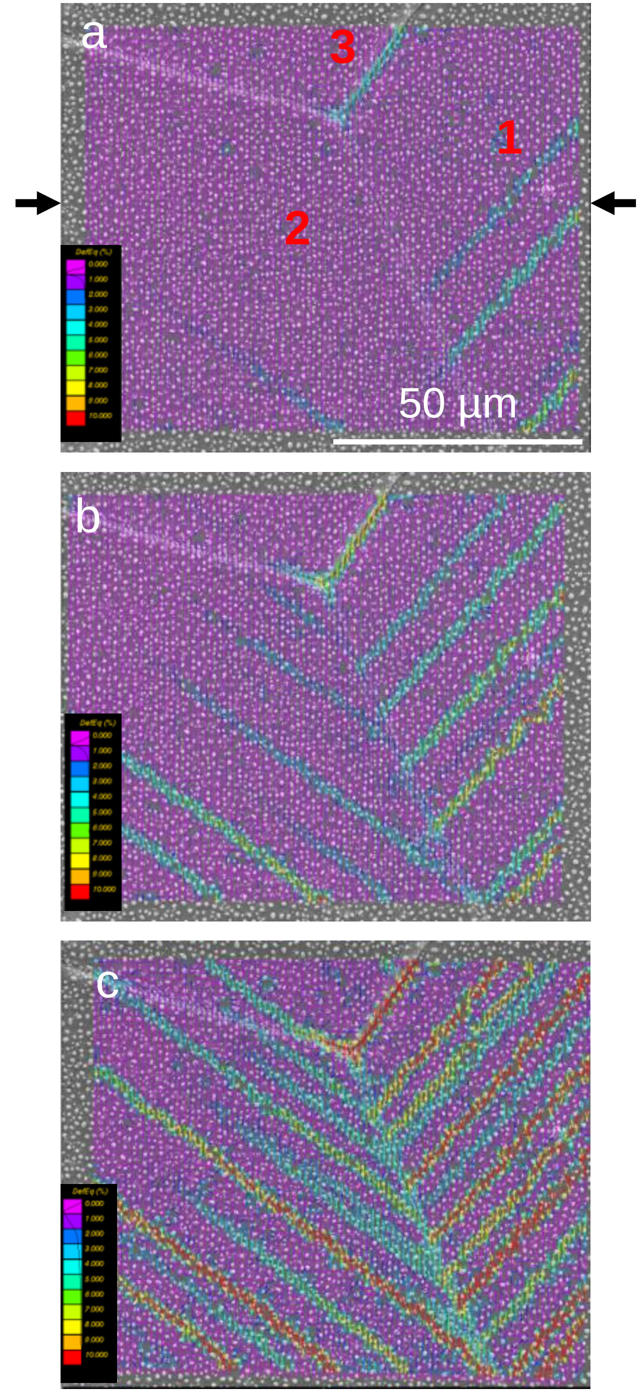
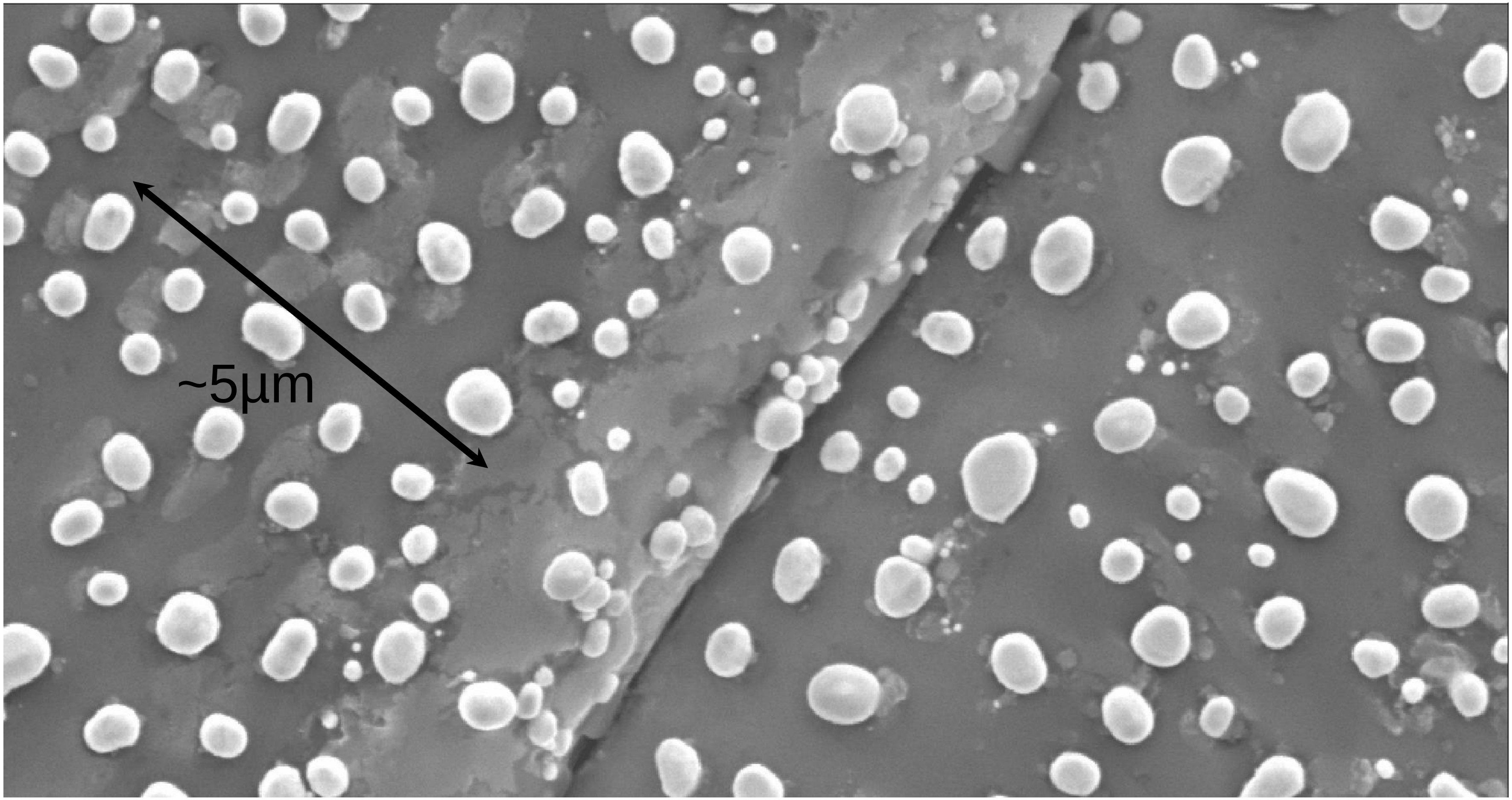
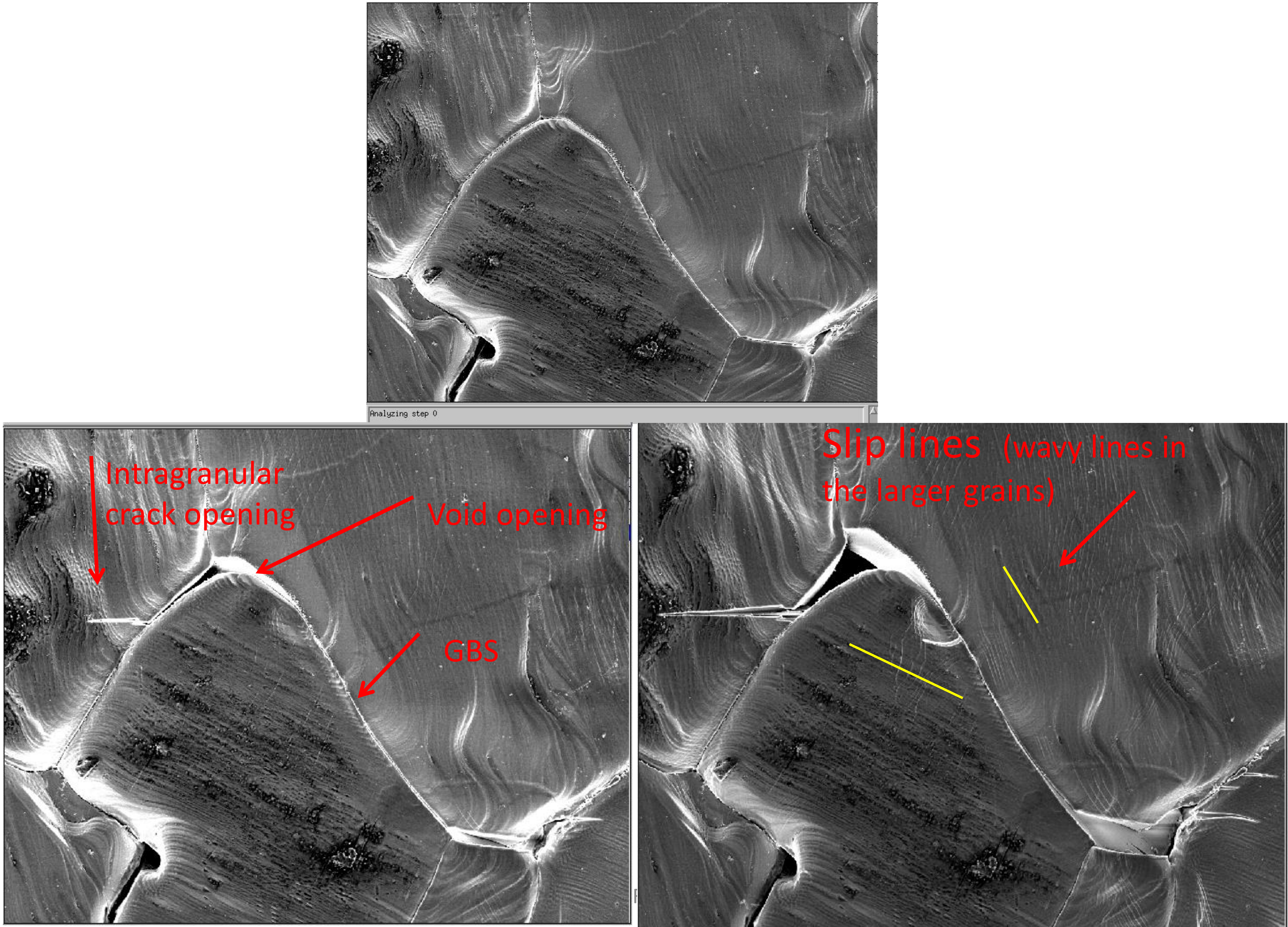


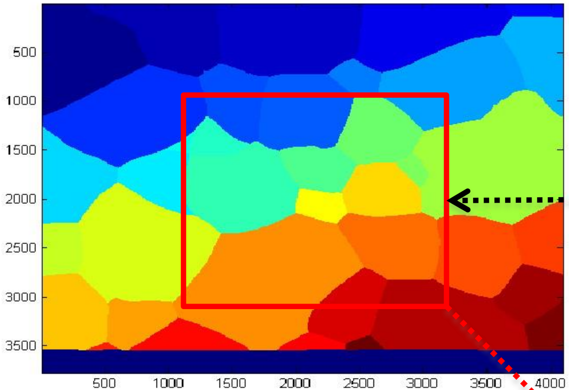
Illustration of CSP and GBS



NaCl Polycrystal SEM Mechanical Testing and Observation

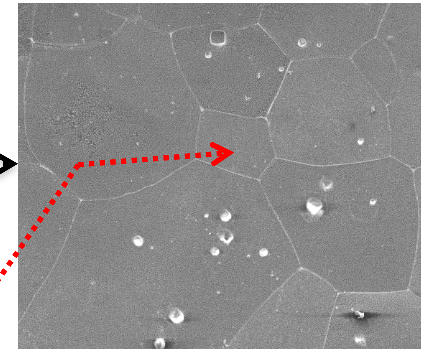


NaCl Polycrystal SEM Mechanical Testing and Observation



Labelled image of the ROI (Ω) microstructure (grains)

GBS Glide quantification



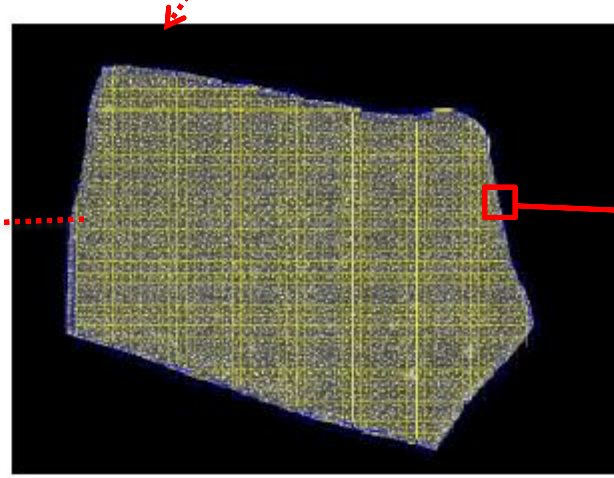
ROI (Ω) at the sample's surface

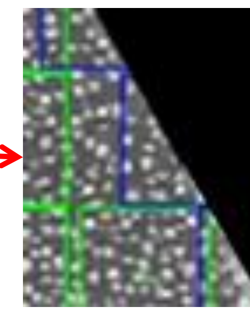
$$\underline{\underline{\Delta F}} = \underline{\underline{F}}_{\Omega} - \sum_{g=1}^{G} \omega_g \underline{\underline{F}}_{\Omega_g}$$

GBS

Average deformation of the ROI

Average deformation of one grain





Modified DIC using Masks

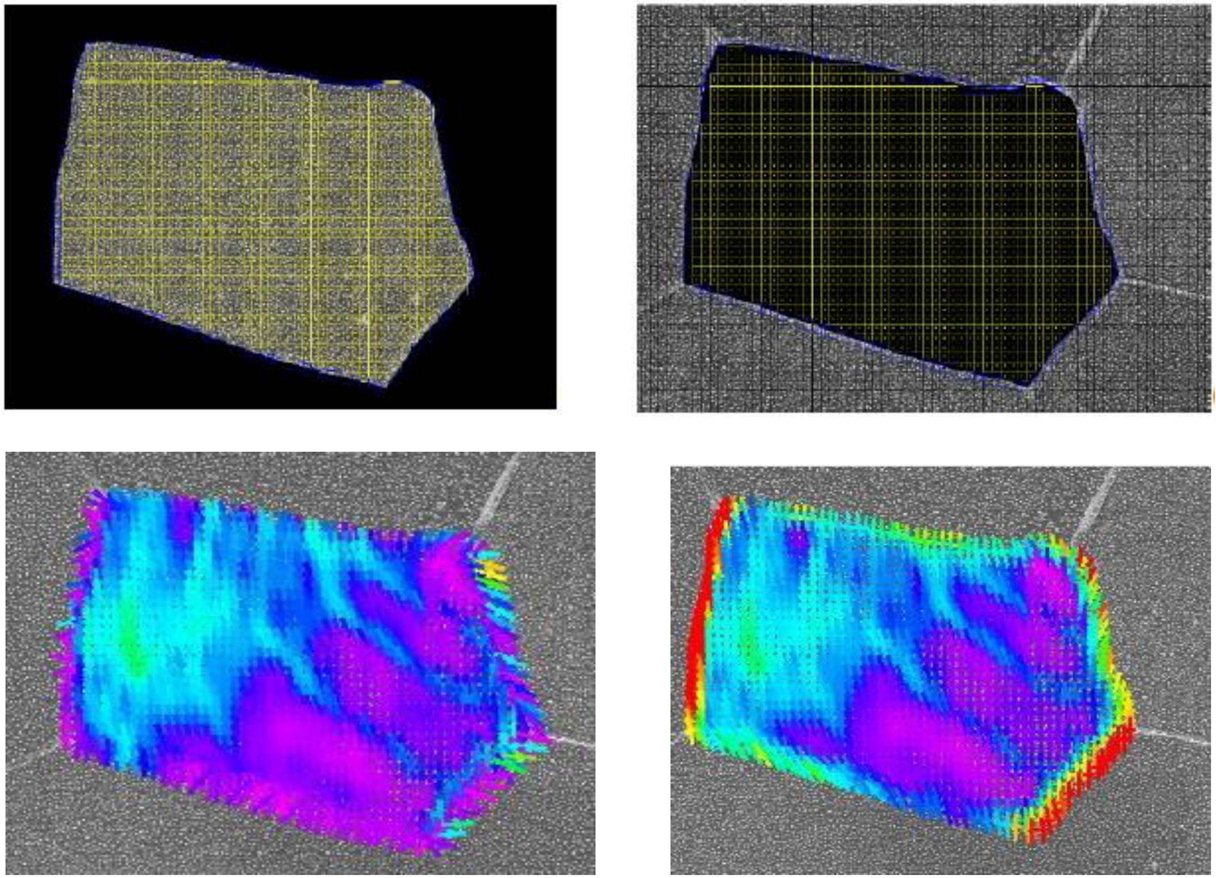
$$\underline{\underline{F}}_{\Omega_g} - 1 = \frac{1}{\Omega g} \int_{\partial\Omega_g} \underline{u}_g \otimes \underline{n} dl$$

A. Gaye and M. Bornert

NaCl Polycrystal SEM Mechanical Testing and Observation

Using masks, one may evaluatet

$$\underline{\underline{\Delta F}} = \frac{1}{|\Omega|}\int_{W} (\underline{u}^{\text{extra}} - \underline{u}^{\text{intra}}) \otimes \underline{n}\, dl$$

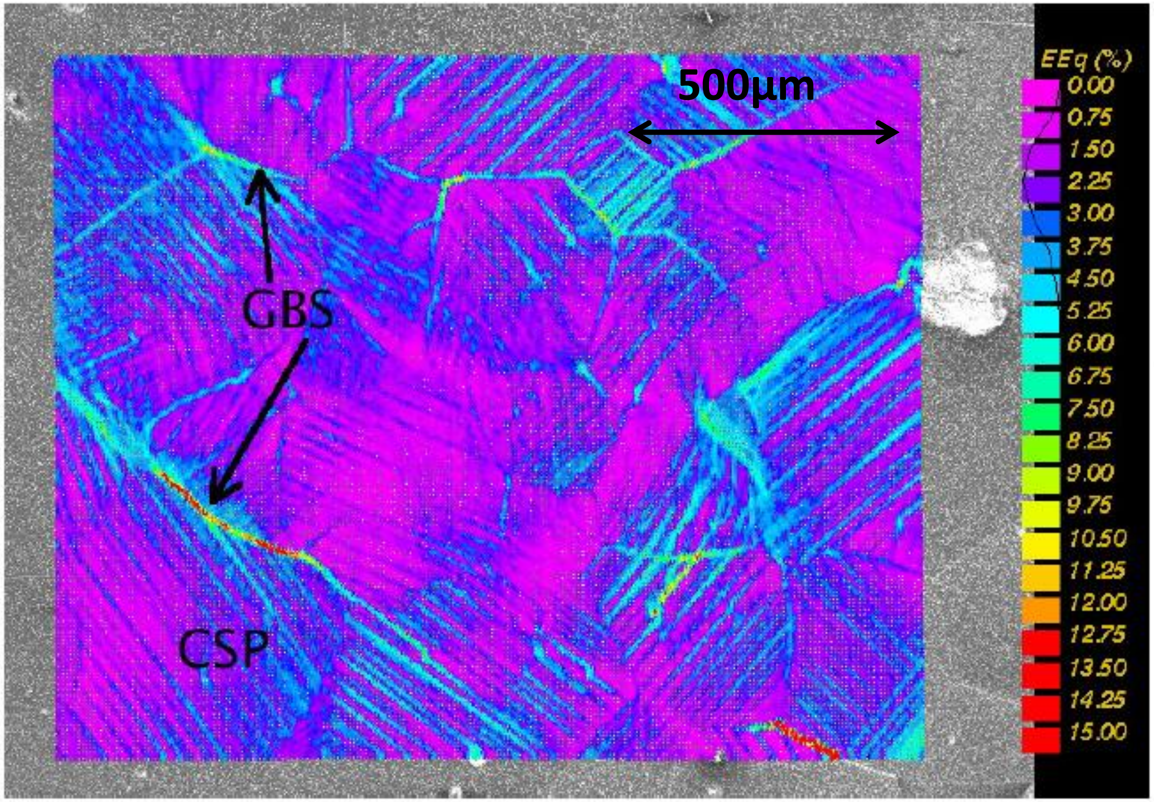


Intragranular Inter+Intragranulat

Component of displacement gradient

NaCl Polycrystal SEM Mechanical Testing and Observation

Direction of macroscopic compression

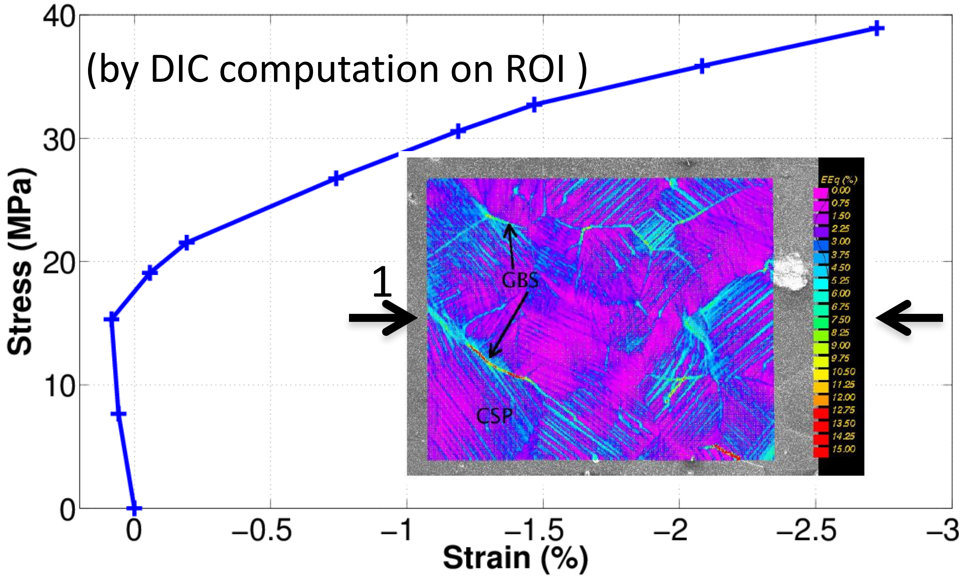


Average compressive strain is 2%.

Local gauge length is 8µm.

local strain map from SEM observations: Second invariant of in-plane strain tensor

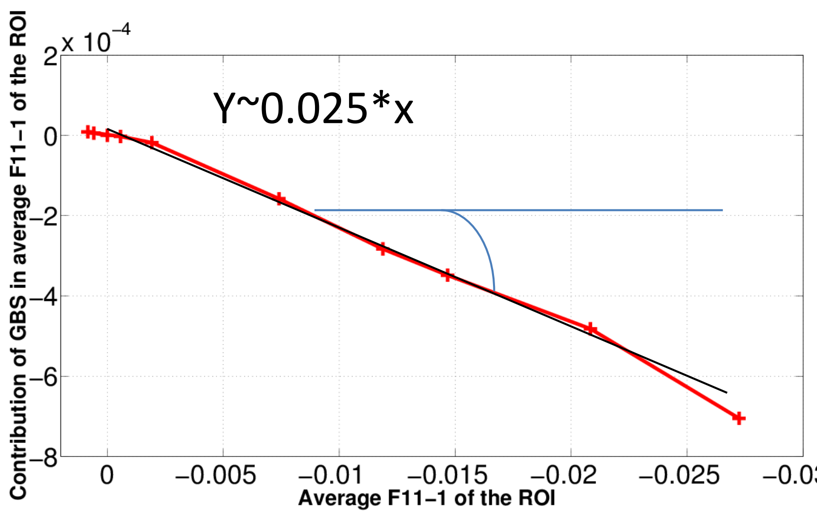
Dominant crystal slip plasticity (CSP)
and
Minor grain boundary sliding (GBS)



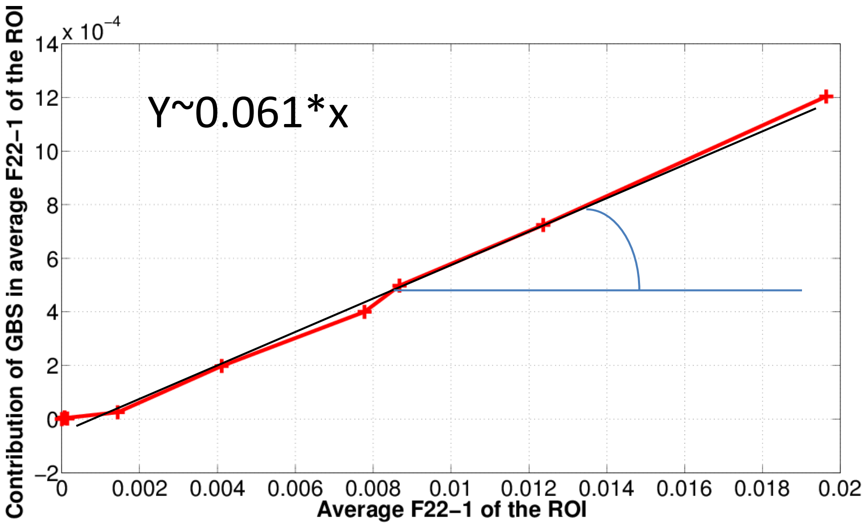
Room temperature

Almost linear hardening
Not enough "easy glide" systems
$(\tau^{\{100\}} \gg \tau^{\{110\}})$
Constant ratio GBS/CSP

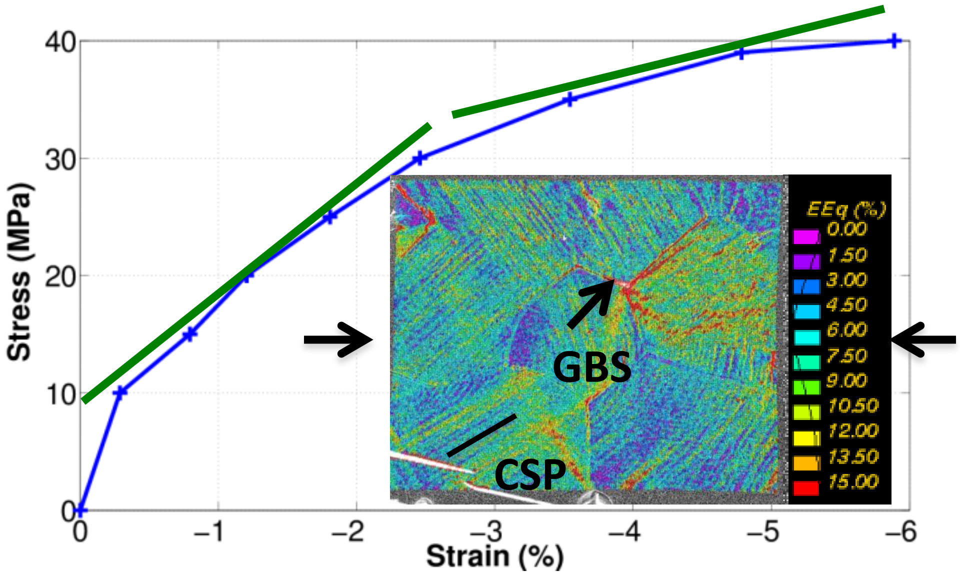
GBS11 is 2.5 % of macroscopic deformation 11



GBS22 is 6% of macroscopic deformation 22



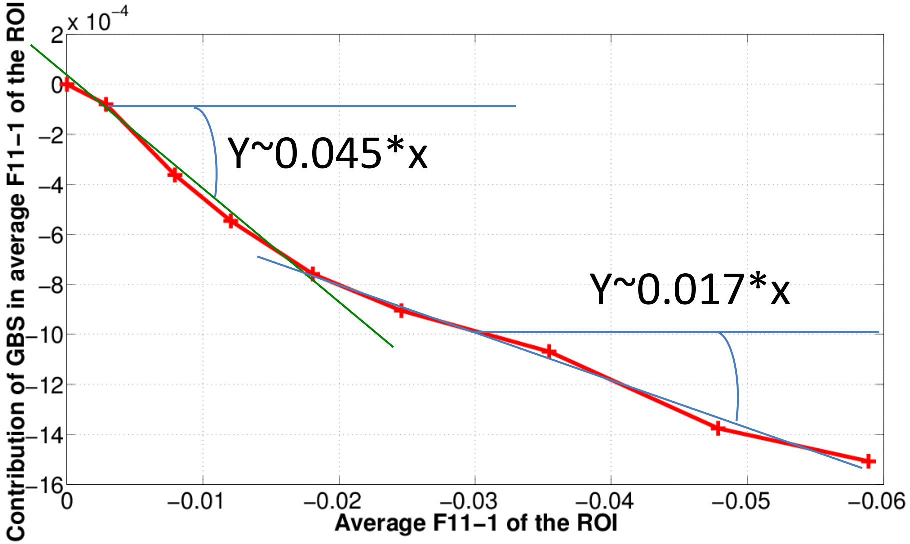
NaCl Polycrystal SEM Mechanical Testing and Observation

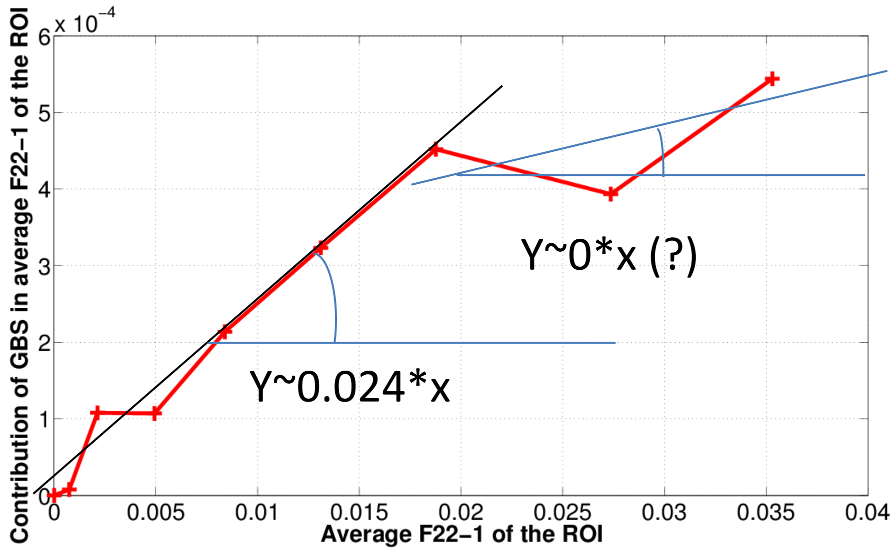


Local gauge length is 3µm, average compressive strain is 6%

350°C

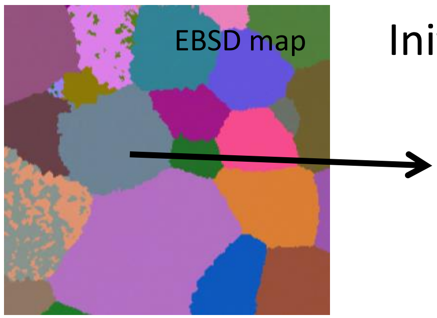
Two-stage hardening
Two regimes with different ratios GBS/CSP
Hardening effects may favor activation of more slip systems



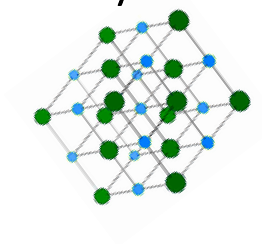


=> Importance of the identification of the active slip planes

What information do we have ?

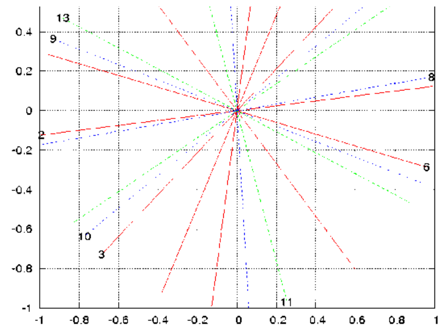


Initial crystal orientation

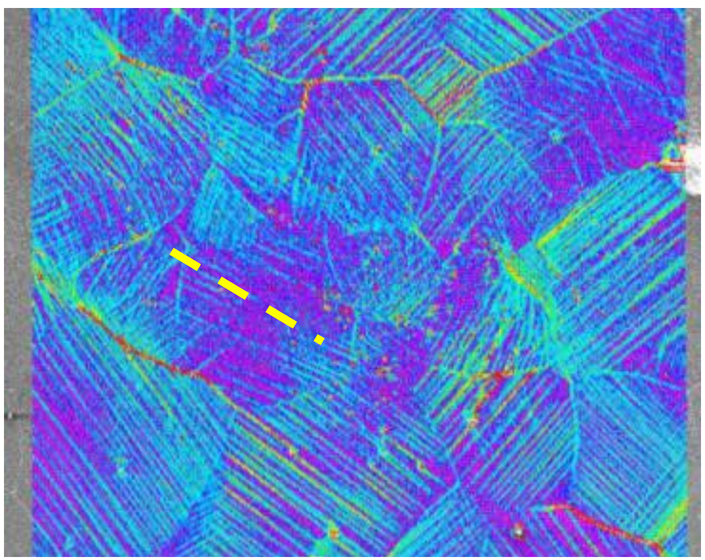


Euler angles
(Ψ, θ, Φ)

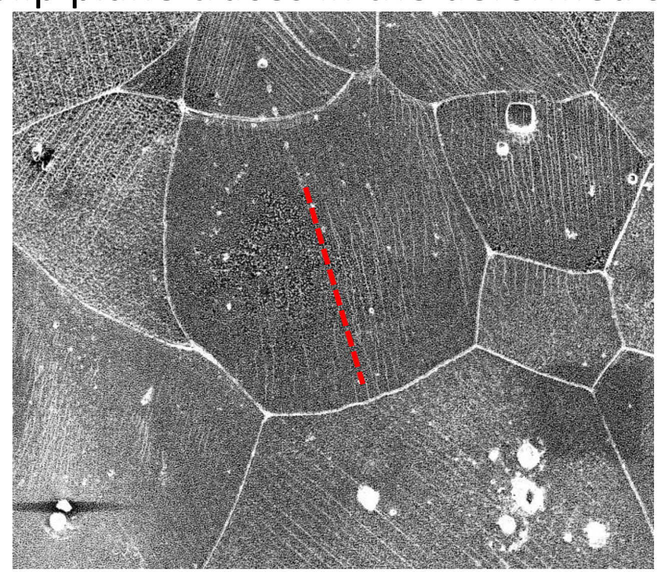
Traces of the 13 glide planes
on the observation surface



DIC Strain field: lines // to slip plane traces



Slip plane traces in the deformed state



Sometimes the identification can be made …

NaCl Polycrystal Glide identification

… When in doubt or need of confirmation : a kinematic approach is not enough and additional assumptions have to be made to estimate the **local stress state** and then identify the **systems with highest Schmid factors**

- "Soft approach" :

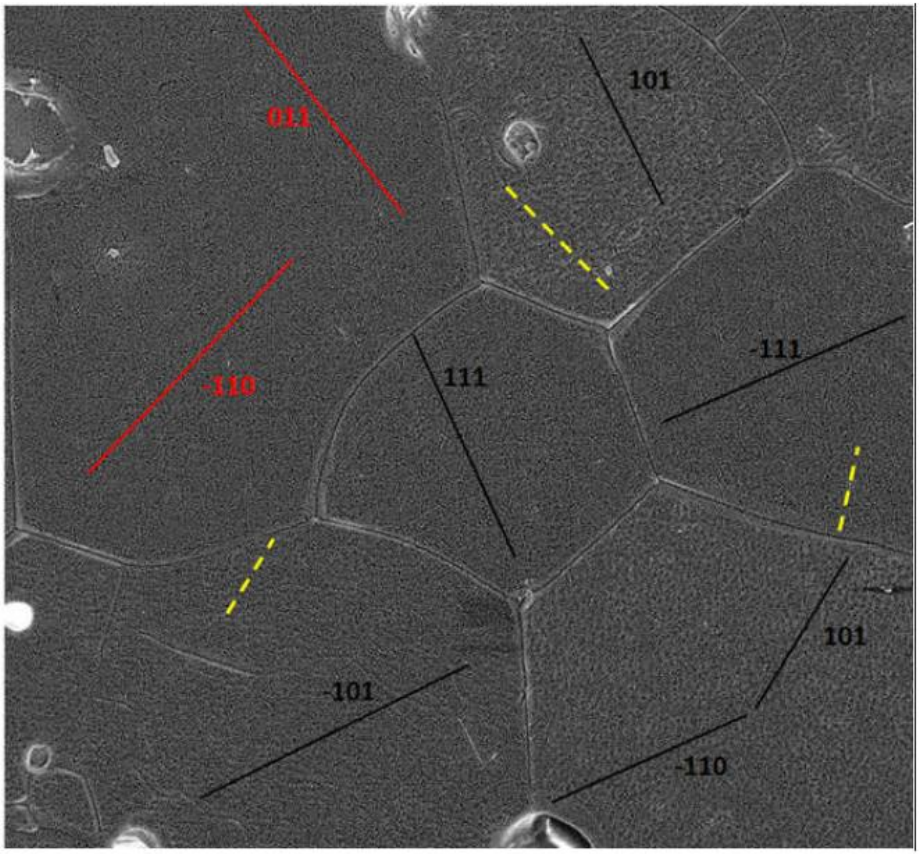
test Taylor, Sachs or relaxed Taylor hypotheses on a grain or a small group of grains to get their reorientation and Schmid factors
(use simplified version of codes developed by P. van Houtte , L. Delanney)

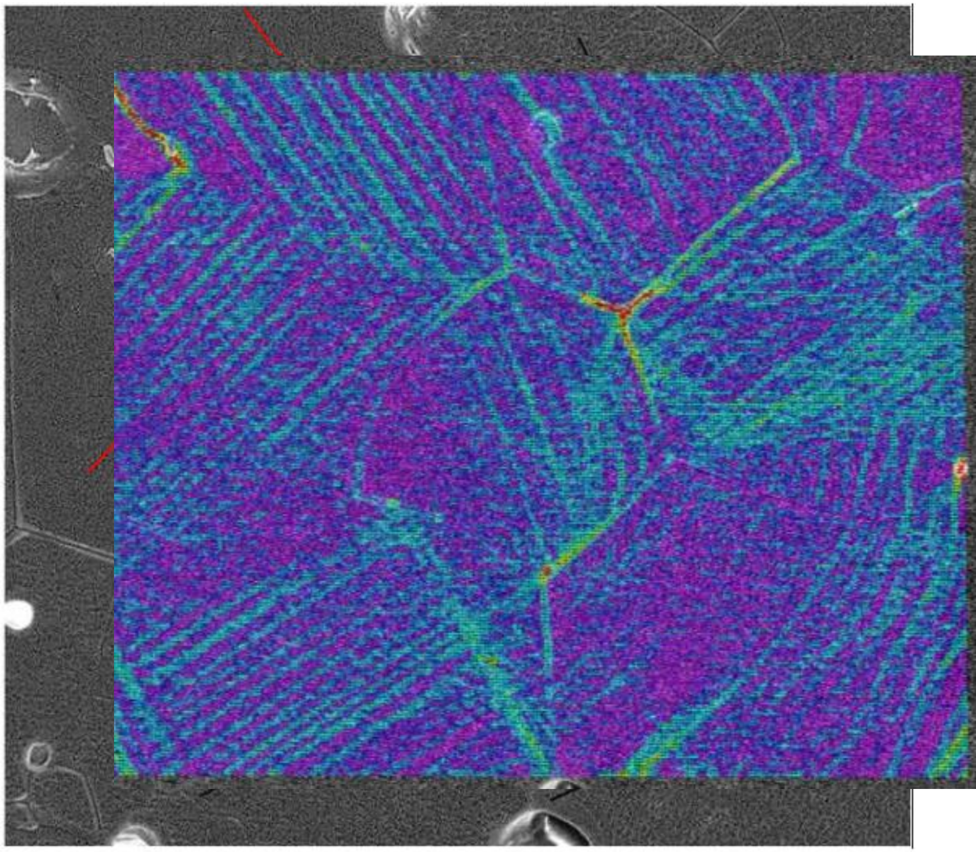
Input :
orientation
Plastic strain tensor shape
(imposed or relaxed terms)
Strain increments

Output:
Final orientation
Schmid factors
Activated slip systems
Slip plane traces

NaCl Polycrystal Glide identification

Planes and systems are identified by an approach based on taylor or sachs assumtions





Red : planes observed on the SEM image (and also on DIC strain maps)
Black: planes observed only on DIC strain maps
Yellow: local slip lines mostly near Grain Boundaries
Note:

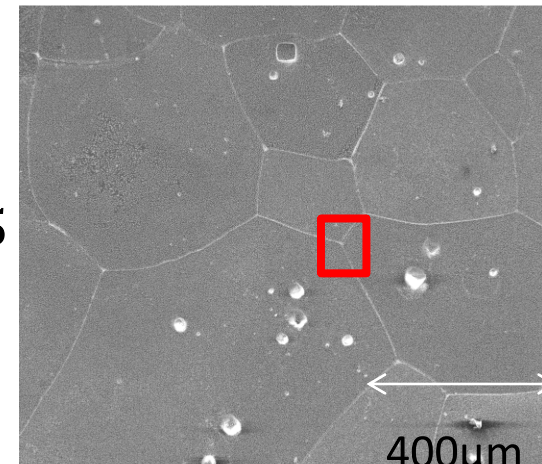
- Activation of {110} and {111} planes
- GBS at triple junction between 3 « non compatible » grains

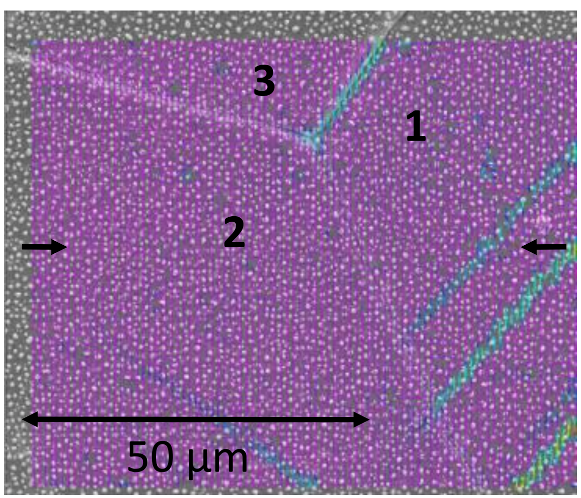
- “Hard approach” : use CPFEM code

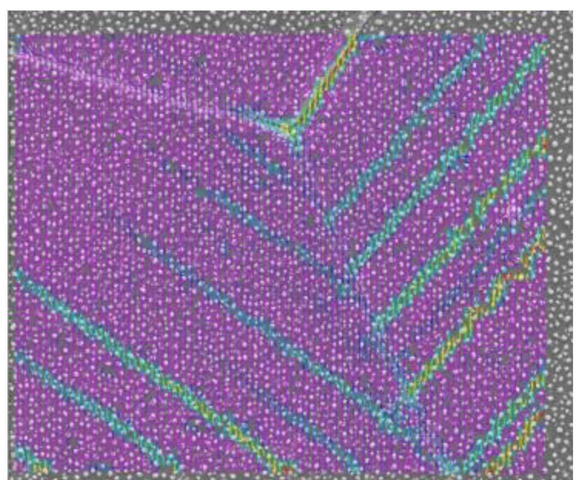
But many hypotheses as well : choice of RVE, choice of 3D extension (extrusion?), choice of BC, hardening rules
(ABAQUS + crystal plasticity UMAT)

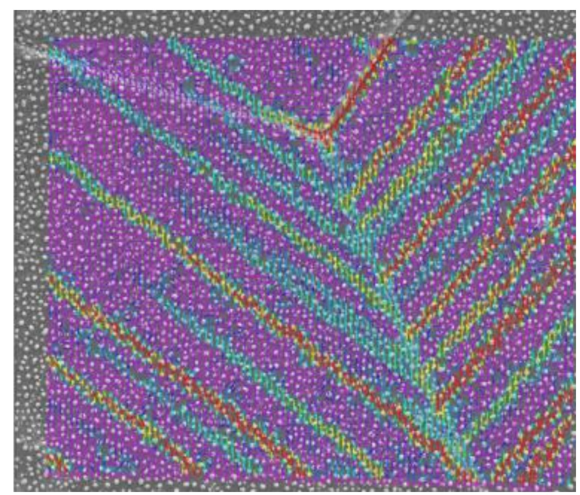
For instance : Let us apply CP FEM near the triple point

- Extrusion as 3D extension
- Uniaxial compression (horizontal axis)
- Initial critical shears $\frac{\tau_{\{111\}}}{\tau_{\{110\}}} = 3$ and $\frac{\tau_{\{100\}}}{\tau_{\{110\}}} = 4.5$
- Simple hardening rule
- 27 noded quadratic elements

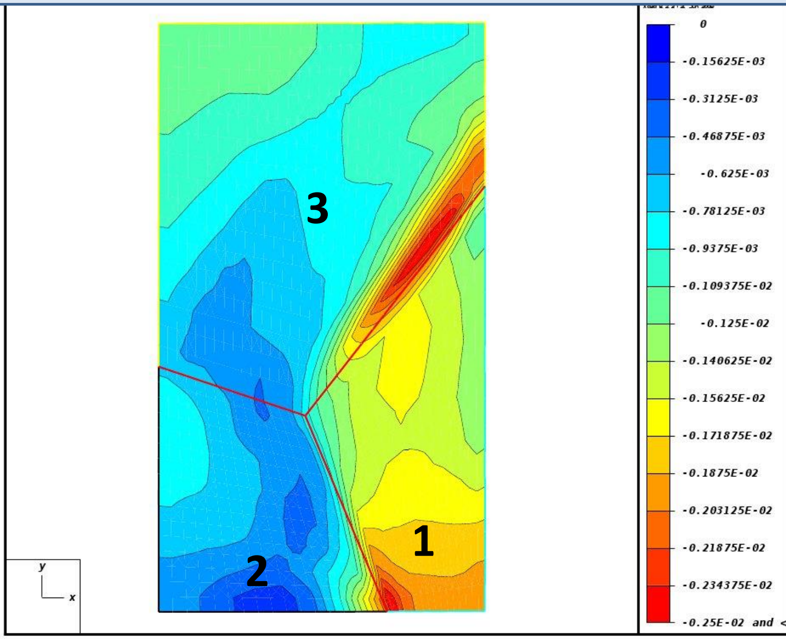






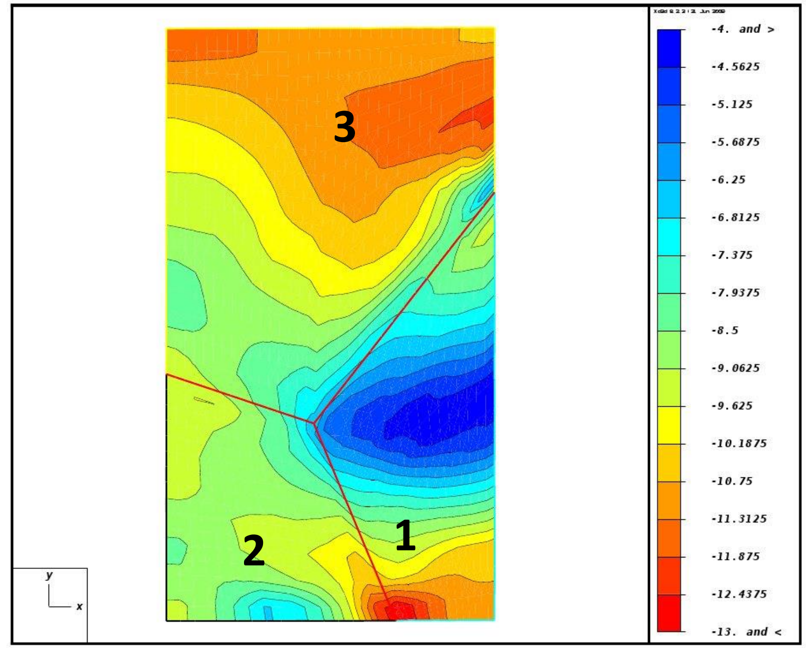


NaCl Polycrystal Glide identification



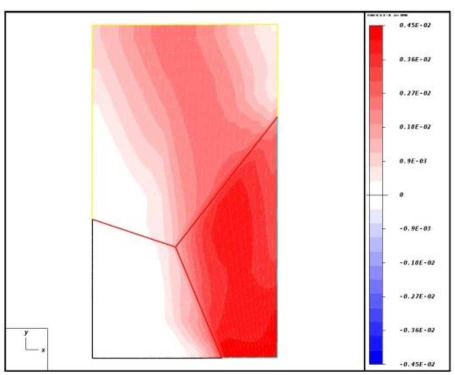
Axial strain

More axial strain in grain 1
Strain concentration near grain boundary 1-3



Axial stress

Stress lower in grain 1
Grain boundary effect near bottom (stress concentration)
Grain 3 sustains higher axial stress

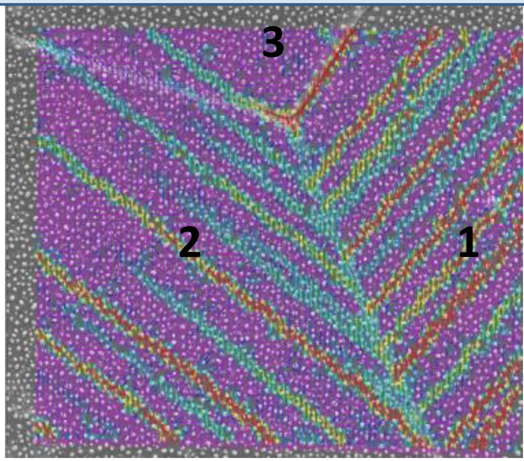


Shear strain

How does this relate to crystal plasticity ?

Let us look at grain orientations and Schmid factors

NaCl Polycrystal Glide identification

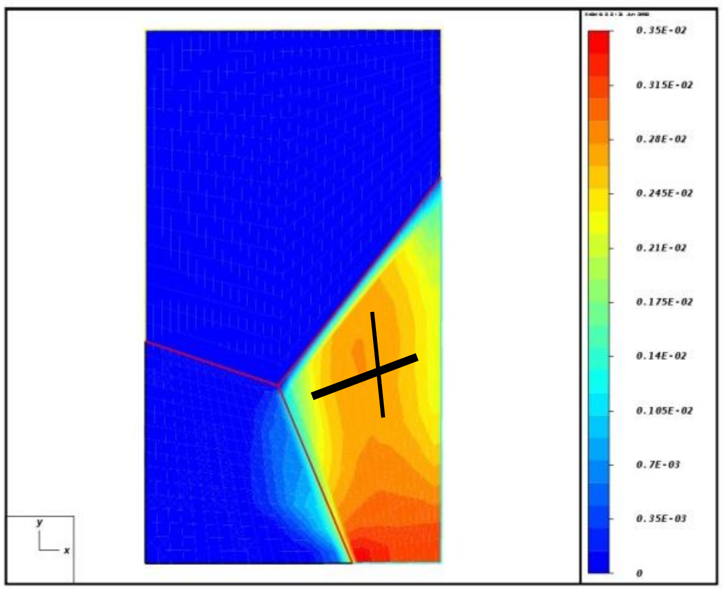


CMV computed equivalent strain

Assuming uniaxial compression for the evaluation of the Schmid factors (SF)

In grain 1
(-110)[110] and (110)[-110] SF=0.25
(-111)[110] SF=0.45
(100)[0-11] SF= 0.47

$$\frac{SF_{\{111\}}}{SF_{\{110\}}} = 1.8 \ < 3 \ and \ \frac{SF_{\{100\}}}{SF_{\{110\}}} = 1.9 \ < 4.5$$

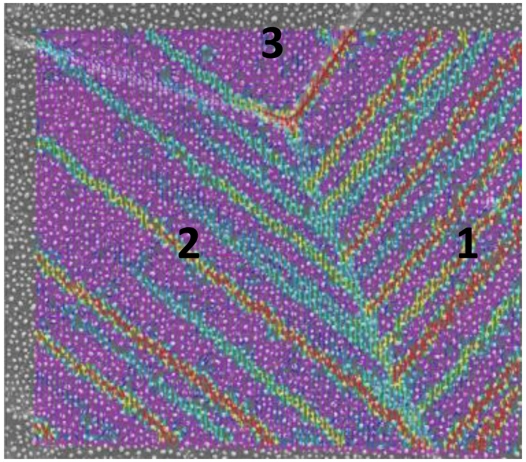


Slip γ on dodecahedral system

Glide is found as expected on (110)[-110] and (-110)[110]

(2 conjugate dodecahedral systems)

NaCl Polycrystal Glide identification



CMV computed equivalent strain

In grain 2

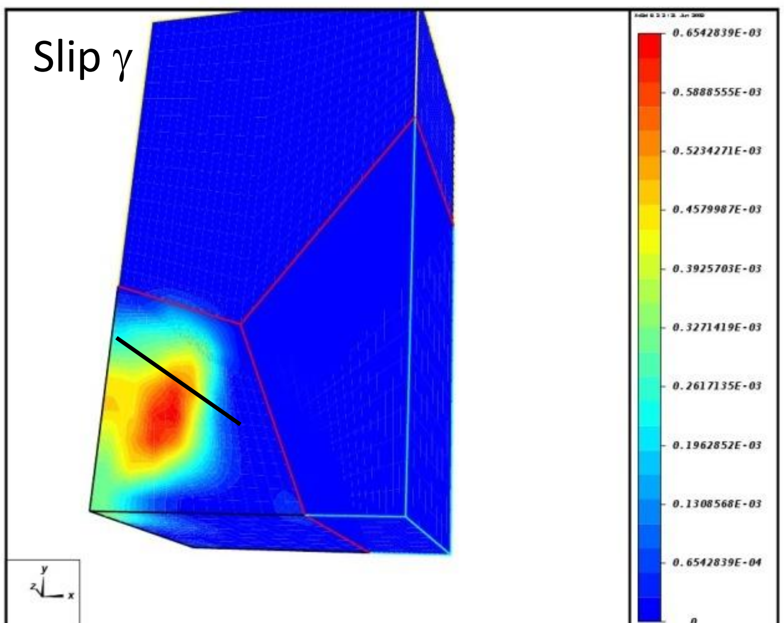
{110}<110> SF <0.02

(-111)[0-11] and (1-11)[-101] SF = 0.28

(001)[110] SF = 0.48 and (010)[-101] SF= 0.47

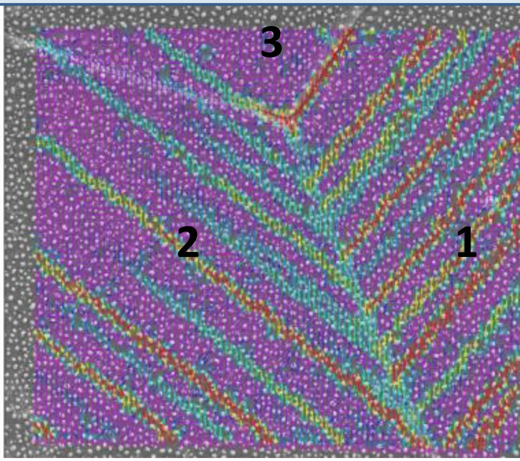
$$\frac{SF_{\{111\}}}{SF_{\{110\}}} = 14 > 3 \; and \; \frac{SF_{\{100\}}}{SF_{\{110\}}} = 24 > 4.5$$

Even at room temperature the cubic glide may be expected



Here we find glide on (010)[-101] (SF=0.47) which may correspond to the observed trace

NaCl Polycrystal Glide identification



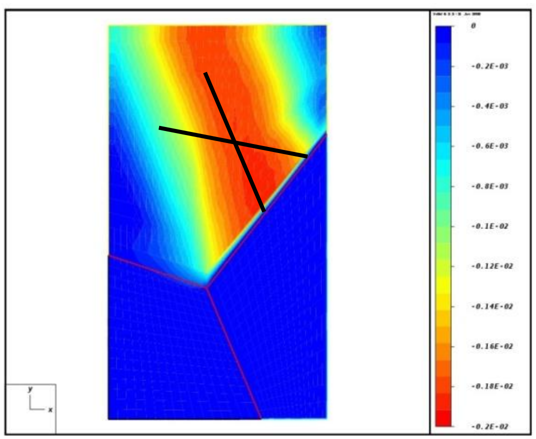
CMV computed equivalent strain

Or grain 3
(011)[0-11] and (0-11)[011] SF=0.15
(-111)[0-11] SF = 0.36
(010)[101] SF=0.5
(100)[0-11] SF=0.43

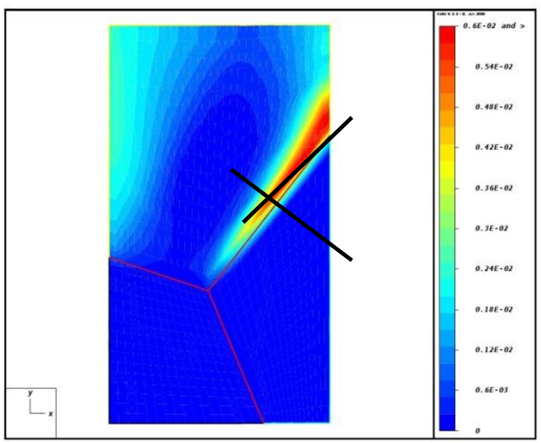
$$\frac{SF_{\{111\}}}{SF_{\{110\}}} = 2.4 < 3 \;\; and \;\; \frac{SF_{(010)}}{SF_{\{110\}}} = 3.3 < 4.5$$

The grain is poorly oriented for easy glide, but shows some local activations

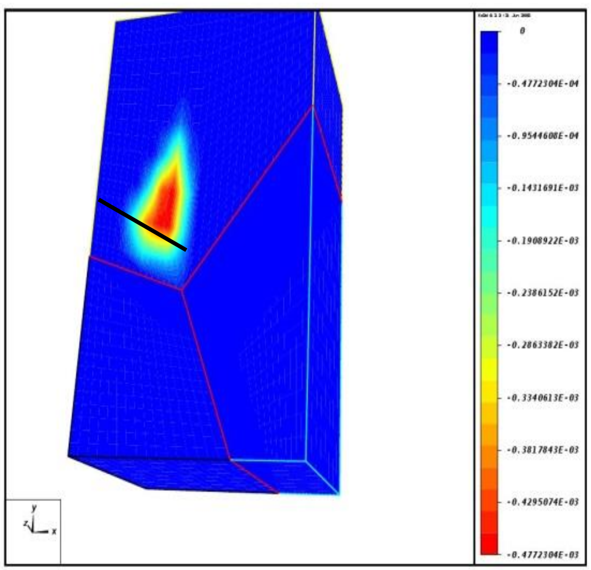
(110)[-110] and (-110)[110]



locally, (011)[0-11] and (0-11)[011]



cubic glide (100)[0-11]



Slip γ on selected systems named above the figure

3D investigations

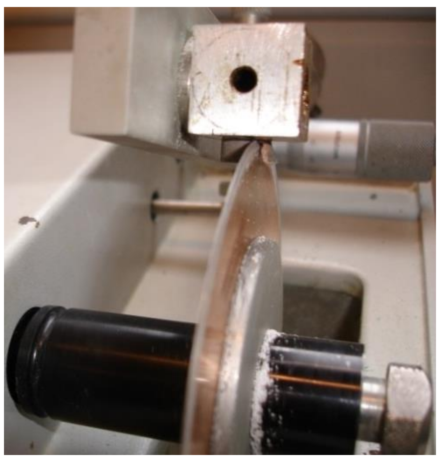
Can we make the link between surface and volume observations ?

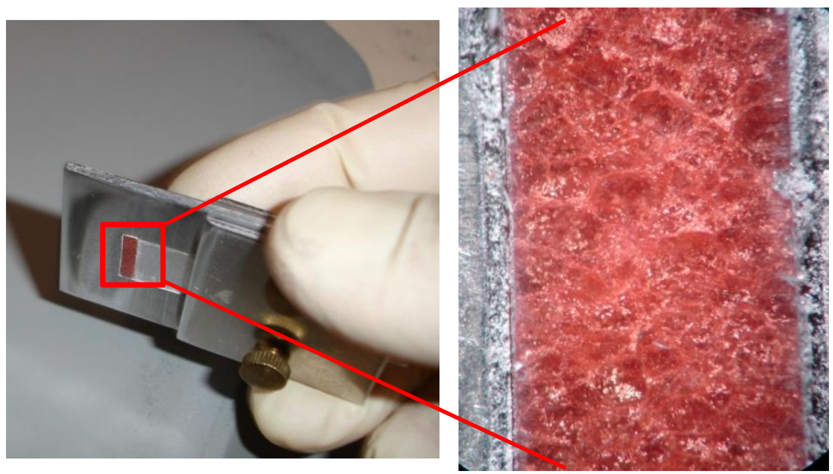
-> Test and observe same sample by XCRT and SEM

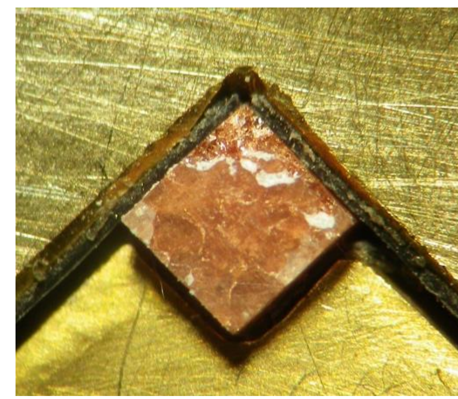
Complex procedure involving transfer of sample between synchrotron line and SEM !

Technical difficulties : sample has to have volume markers (Cu particles), precision for the 3D reconstruction has to be good, alteration by Xray irradiation should be addressed, still too many grains for identifying the orientations in the volume (DCT)

Sample preparation

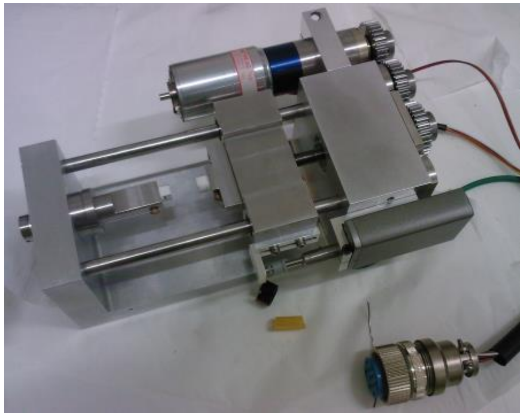






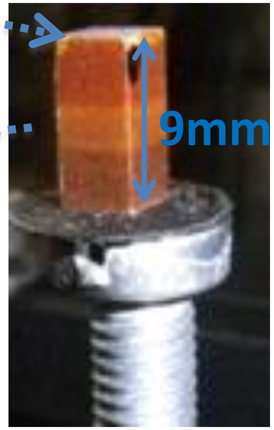
3D investigations

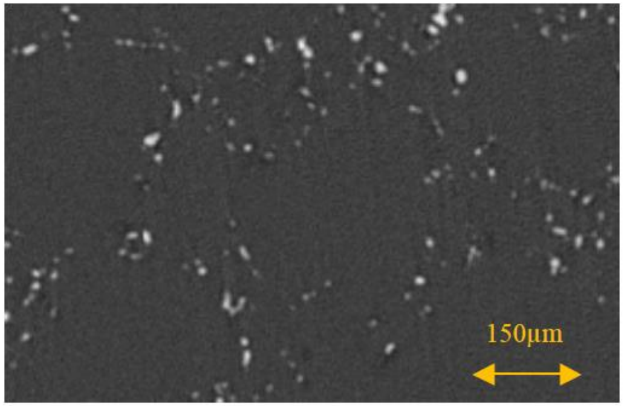
Mechanical loading device for uniaxial compression test



Room temperature

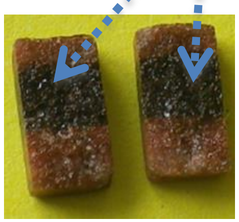
- F_{max} = 5000 N
- V = 1µm. s^{-1}
- Strain rate = ~$10^{-4}s^{-1}$



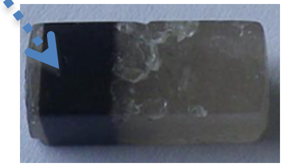


Cross section through CT volume of polycristalline halite with additional copper markers

Irradiation point defects

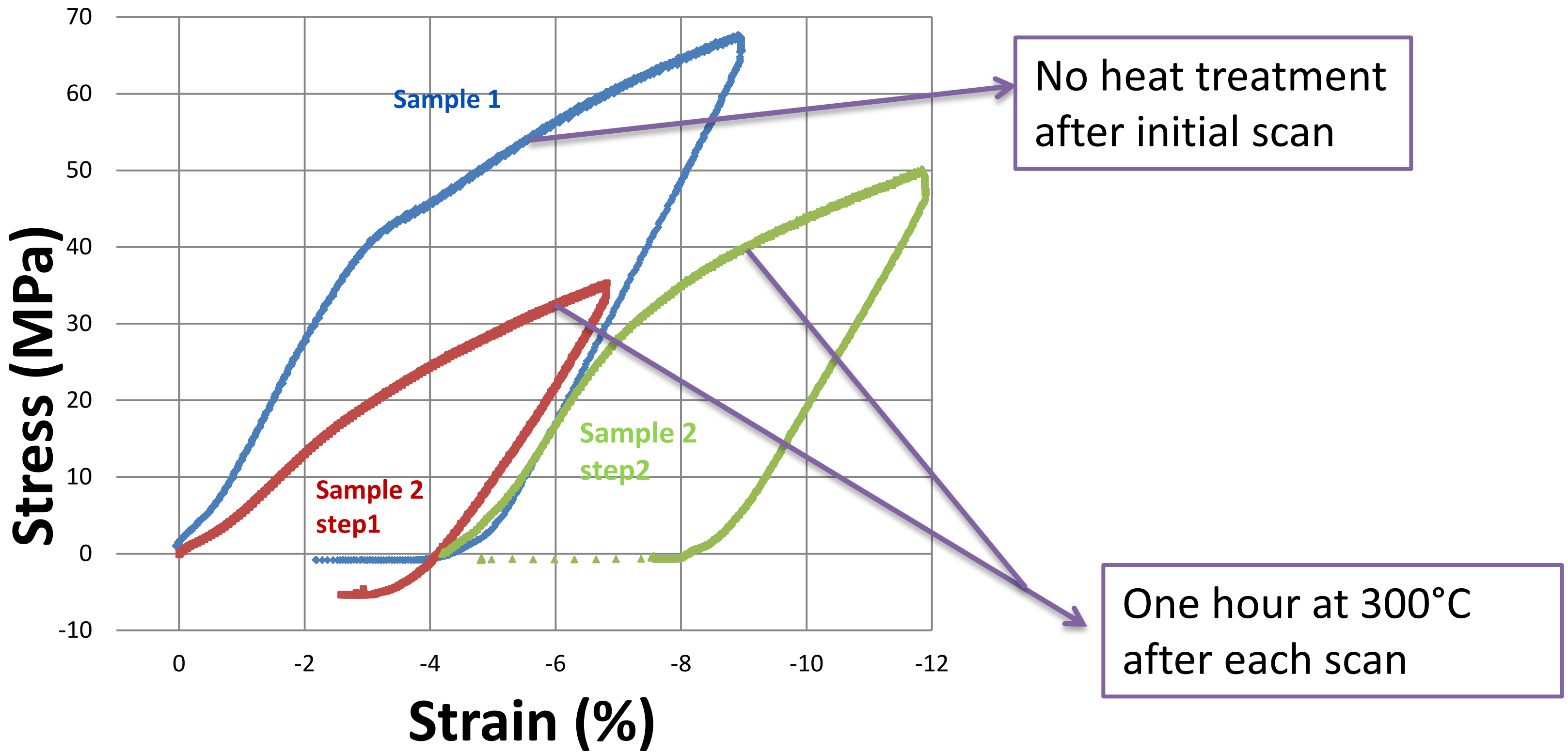


Synthetic halite + copper particle



Pur synthetic halite

Synchrotron radiation damages the material:
appearance of point defects (Farbe Zentrum)
Experimental sequence: Initial XRCT scan, Heat treatment, Mechanical loading-unloading cycle step1, Scan, Heat treatment, …



3D investigations

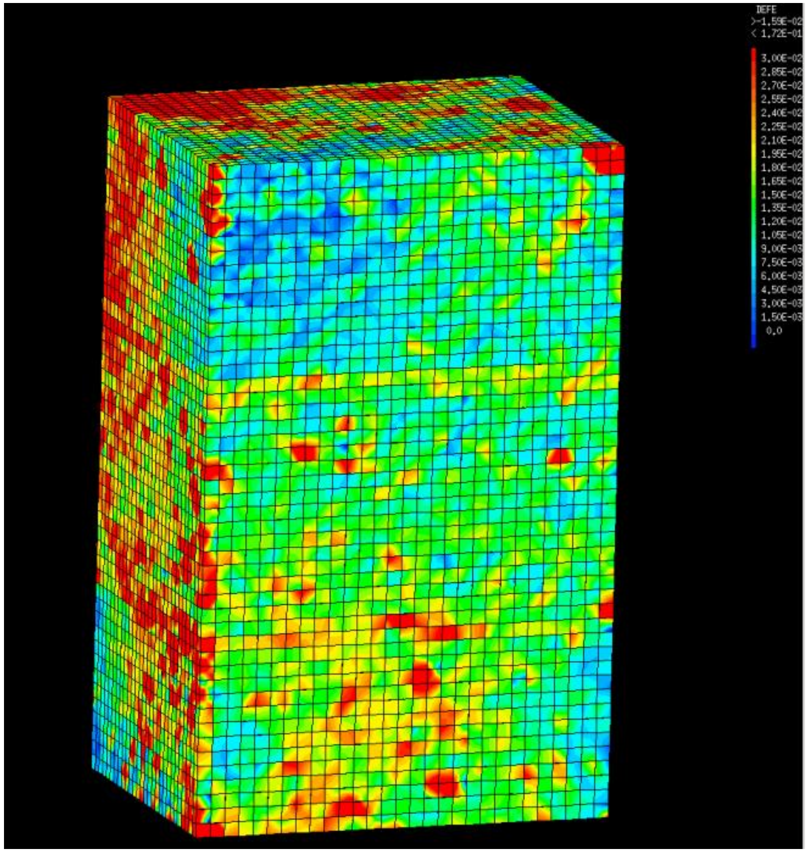
cycle 1 -1,0525%
cycle 2 -2,7307%
cycle 3 -5,0205
cycle 4 -9,0831

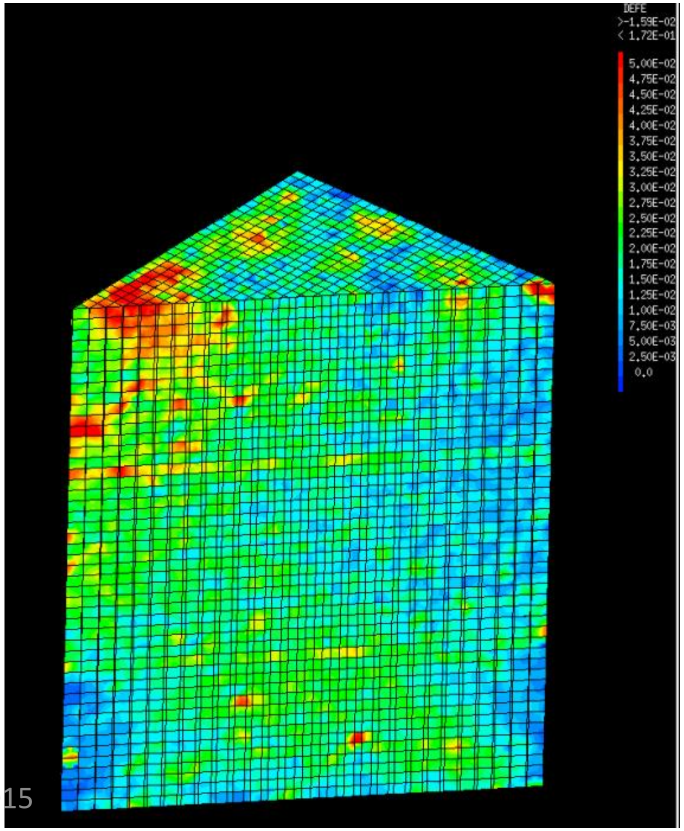
} Strain computed by 2D DIC over ROI

29x30x48 points
step 40 voxels, correlation 40x40x40 with subvoxel optimization
1voxel=3,5µm

Volume= 2.20287E+09 ******* Average deformation *******
EPXX= 7.84539E-03 EPXY=-1.27108E-04 EPXZ= 1.22615E-03
EPXY=-1.27108E-04 EPYY= 7.45521E-03 EPYZ= 7.08331E-05
EPXZ= 1.22615E-03 EPYZ= 7.08331E-05 EPZZ=-1.60655E-02
****

Cycle 1 -1,0525%

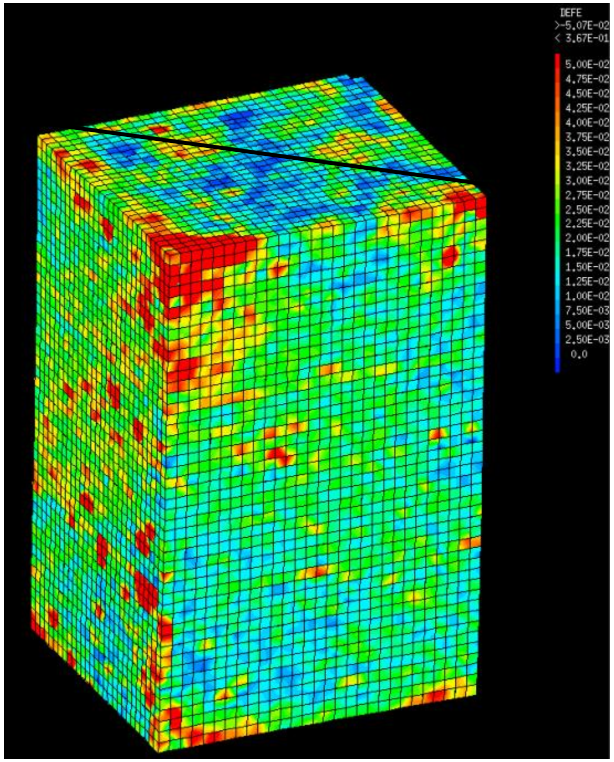


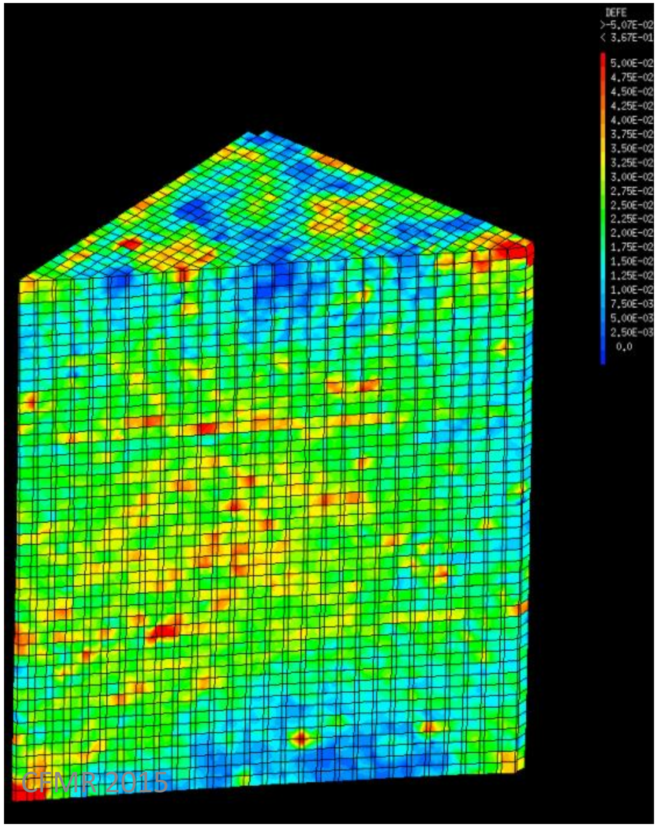


Next step : DIC between end of step 1 and end of step 2 :
Final strain Cycle1 -1,0525% and Cycle2 -2,7307% (2D DIC)

Volume= 2.65620E+09 ******* Average strain *******

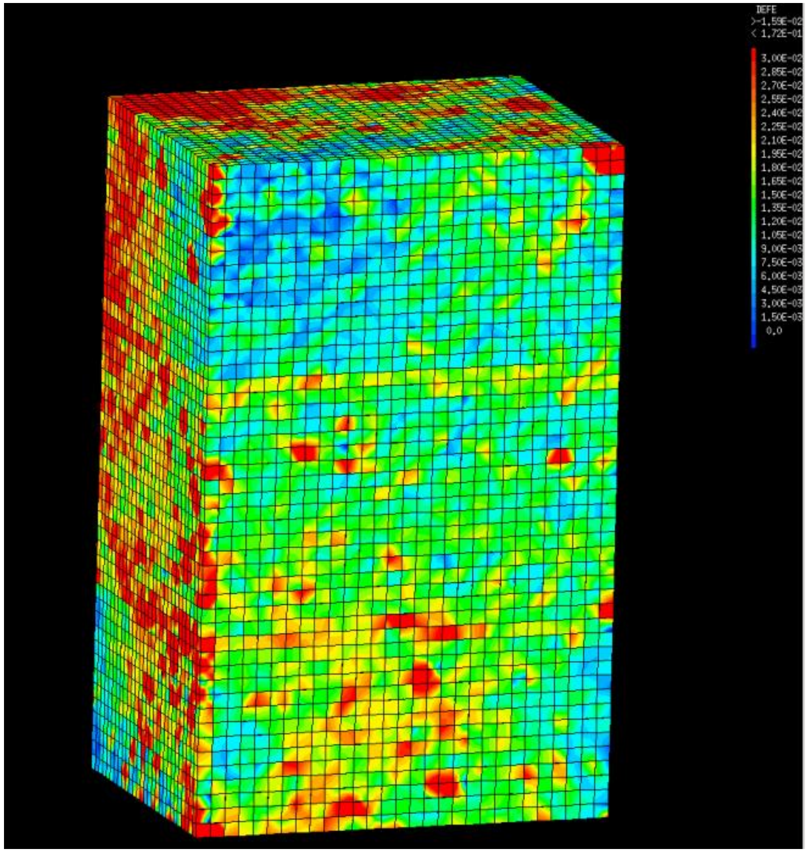
EPXX= 7.92053E-03	EPXY=-1.57482E-04	EPXZ= 1.24274E-03
EPXY=-1.57482E-04	EPYY= 7.56926E-03	EPYZ= 4.54490E-05
EPXZ= 1.24274E-03	EPYZ= 4.54490E-05	EPZZ=-1.62257E-02



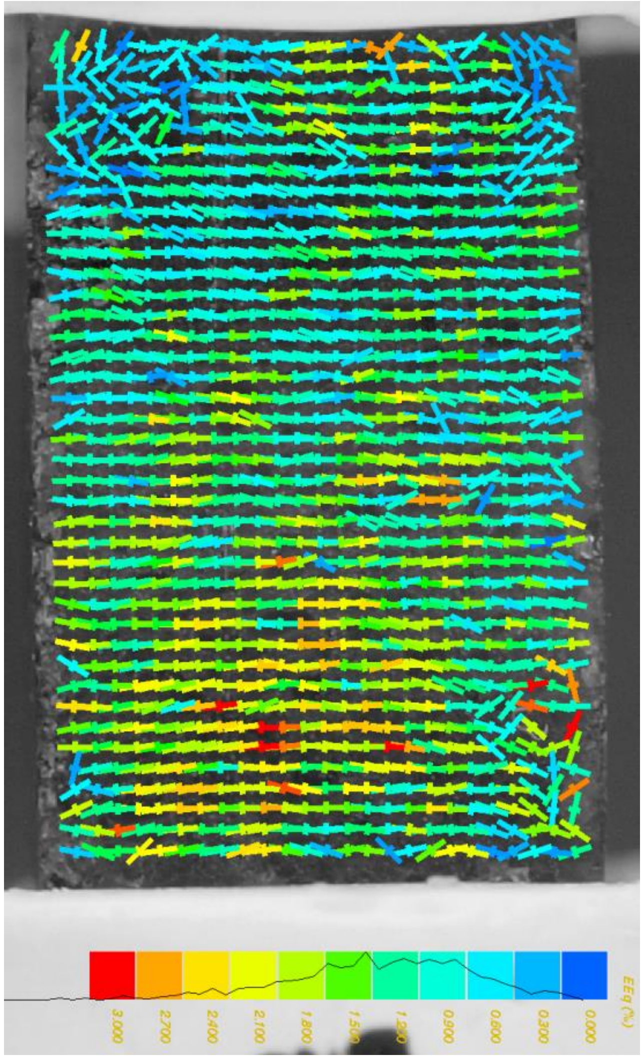


3D investigations

Strain field comparison 2D-3D end of step 1



Volume= 2.20287E+09 ******* Deformation moyenne *******
EPXX= 7.84539E-03 EPXY=-1.27108E-04 EPXZ= 1.22615E-03
EPXY=-1.27108E-04 EPYY= 7.45521E-03 EPYZ= 7.08331E-05
EPXZ= 1.22615E-03 EPYZ= 7.08331E-05 EPZZ=-1.60655E-02
****

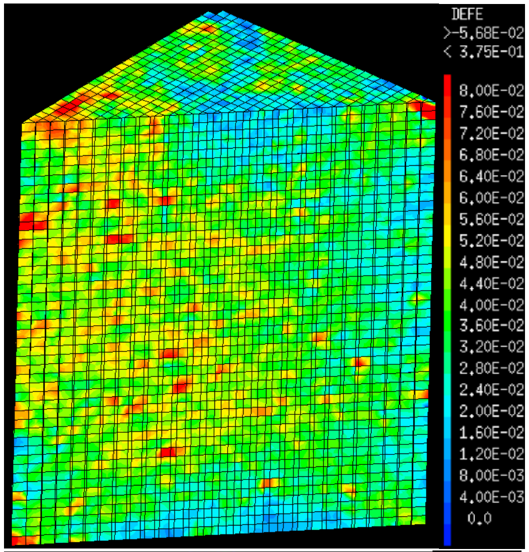


Cycle 1 -1,05%

Deviatoric component

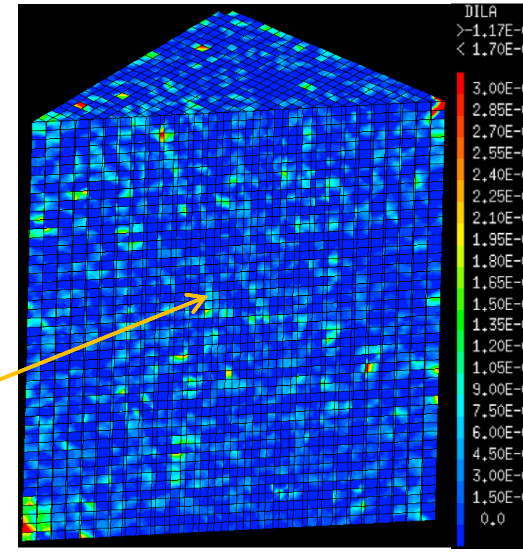
Volumic strain

7 mm

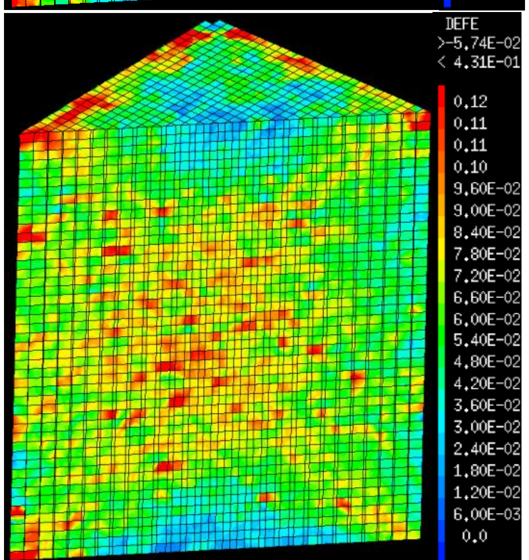


Grains ~ 50 to 200 µm

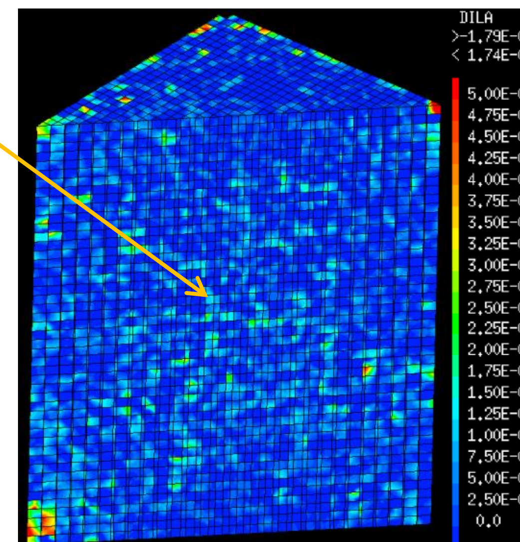
3,6 %



Microcracks
//
Compression axis

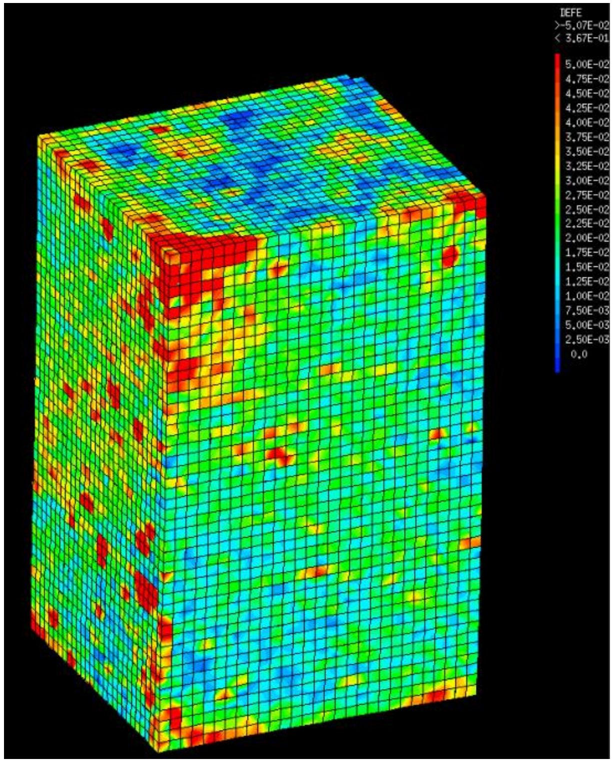


6,0 %



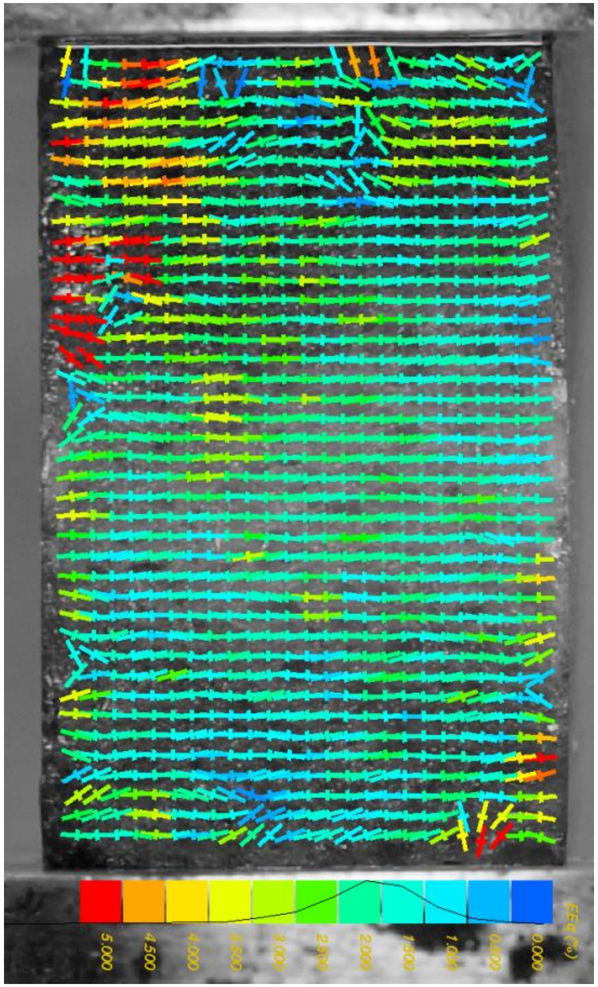
Measure gage 70 µm

Strain field comparison 2D-3D: step 2 wrt step1

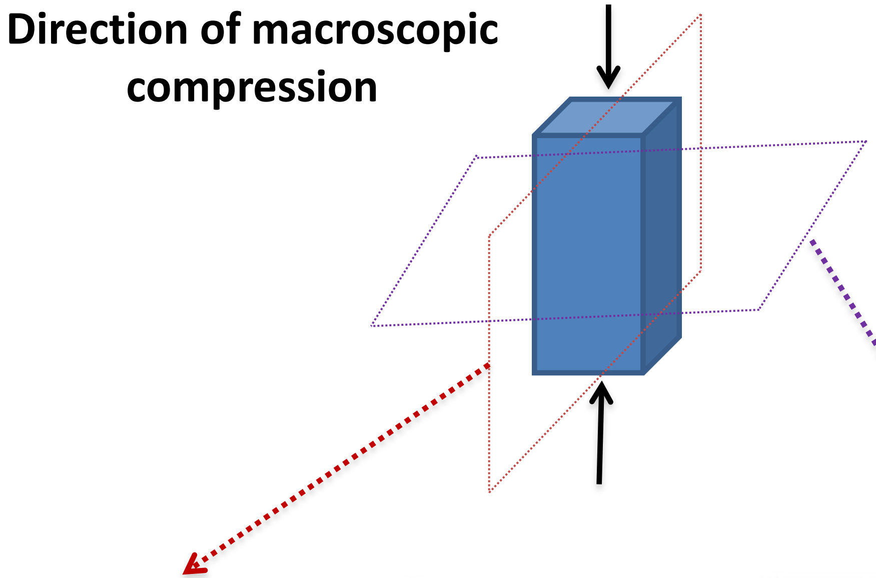


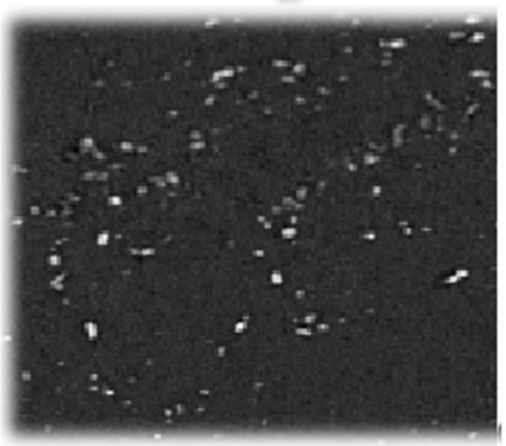
Volume= 2.65620E+09 ******* Deformation moyenne *******

EPXX= 7.92053E-03	EPXY=-1.57482E-04	EPXZ= 1.24274E-03
EPXY=-1.57482E-04	EPYY= 7.56926E-03	EPYZ= 4.54490E-05
EPXZ= 1.24274E-03	EPYZ= 4.54490E-05	EPZZ=-1.62257E-02



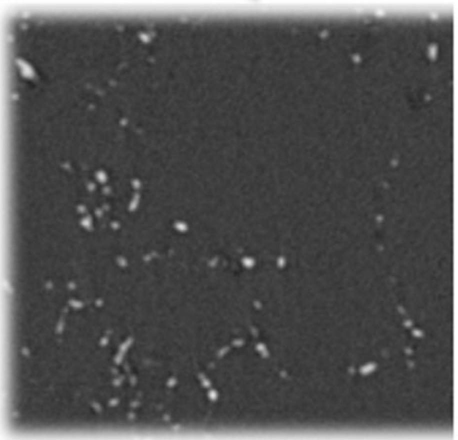
Cycle1 -1,0525% VS cycle2 -2,7307%





Vertical cross section through the sample

(Total strain 6%)



Horizontal cross section through the sample

GBO observed from the beginning of the test

Conclusions - Perspectives

Main Results

- Evidence of two deformation mechanisms at both 20°C and 300°C
- Quantification of GBS and CSP
- Identification by observation and simple mechanical assumption of active slip systems
- Correlation between 2d and 3D observations
- Development of many techniques that may be used for other materials

Perspectives

- Complete 3D analysis (and comparison with 2D)
    - Including orientations in volume (DCT)

- Modelling real multicrystals, not only with classical CP-FEM but
- Include a grain boundary mechanism :

  Damage, cohesive zone … or
  at low strain rates solution transfer.