Multiscale viscoplastic behaviour of halite: micromechanical approaches by full field measurements

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**Geotechnical applications:**

- Temporary storage of hydrocarbons, compressed air (halite deep caverns).
- Long term storage of nuclear wastes (halite mines).

**Pilot Power Plant since 1978,**

Huntorf, Germany

(E.N. Kraftwerke).

2 caverns, 300 000 m³

$P_{\text{min}} = 4 \text{ MPa},$

$P_{\text{max}} = 7 \text{ MPa}$

(290MW)

Compressed Air Energy Storage (CAES)

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**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

\[ \Sigma(t) \]

\[ \frac{F}{S} \]

Micro

- Microstructure
- Mechanisms
- Interactions
- Heterogeneities
- Scales
- REV

Fields!

\[ \sigma(x,t) \]

Phenomenological flow laws

(analytical/mean field)

Physically sound flow laws

Geotechnical applications

But, no extrapolations!

Full field measurements

LVDT 5 cm

Strain

\[ E(t) \]

\[ \frac{N}{L} \]

\[ \epsilon(x,t) \]

Numerical modelling

(full field)
Natural Halite samples:

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

How representative (REV)?

7 cm

Synthetic Halite samples:

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

Best candidates for μ-mechanical testing.
Sample synthesis (from 99.9% NaCl powder)

- **As-is** hot pressed
  - $T = 200°C, 150$ Mpa, 7 days

- Annealed
  - $T = 750°C, 0.1$ MPa, 7 days

- Crushed hot pressed
  - $T = 200°C, 150$ Mpa, 7 days
Macroscopic constitutive relations

Classical creep tests (RT, triaxial)

Cyclic Load (8 h period)

Power-law creep: $n = 3$

Very slow creep tests (Varangéville mine)

Dead load rig

Loading plate

Sample

4 LVDT

5 cm

$\dot{\varepsilon} = 10^{-11} - 10^{-12} \text{ s}^{-1}$

$\sigma = 0.1 - 0.25 \text{ MPa}$
Macroscopic tests $\rightarrow$ phenomenological flow laws

**Lab tests**

\[ \dot{\varepsilon}_{CL} = 8.1 \times 10^{-5} \exp\left(\frac{-51600 \text{ J mol}^{-1}}{RT}\right)(\sigma_1 - \sigma_3)^{3.4} \]

Power-law creep (Munson, 1997)

**Mine tests**

Test at Varangéville Mine

14.4°C – 0.1 MPa

5 - 20 MPa

0 - 120 °C

Storage conditions (undefined mechanism)

$\rightarrow$ misleading extrapolations!
Multi-scale micro mechanical testing:
Micro-extensometry, or Full (mechanical) Field Measurements (FFM)

Observation scales:

km  m  mm  µm  nm

Macro-Opt  Micro-Opt  SEM  (µ)XRCT  (nanoXRCT)

Opt. Microscopy-FFM
Mag. X1 (24×36 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

Analysis domain
Image spatial resolution

FEG - SEM- FEI quanta 600

Sample

Vmin = 1µm/s
Fmax = 100 KN

2 cm

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

1- Image acquisition during loading

Images $\rightarrow$ displacements $\rightarrow$ deformations

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 $C$.

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

Displacement field

3 – Calculation deformations

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

Strain field

CorrManuV© (M.Bornert)
Displacement surface markers

Paint Spray
(50 - 100 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

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

(final macro strain ~5%)
Coarse grains (200 - 500 µm)  Strain rate = ~ $10^{-4}$ s$^{-1}$
« In-situ » SEM - FFM - DIC

Coarse grains (200 - 500 µm)

Strain rate = ~ $10^{-4}$ s$^{-1}$
Large grains (200 - 500\,\mu m)

Strain rate = \sim 10^{-4}\,\text{s}^{-1}

10\% strain localizes at interfaces.
Example of intracristalline plasticity and grain boundary sliding.

- red → grain boundary sliding.
- black → intracristalline plasticity
Example of intracristalline plasticity and grain boundary sliding.

- **Red arrow**: Grain boundary sliding.
- **Black arrow**: Intracristalline plasticity.

- **10 µm**: Scale bar for intracristalline plasticity.
- **30 µm**: Scale bar for grain boundary sliding.
Identification of slip systems

\[ \sigma_{ij} = \begin{pmatrix} \sigma_{xx} & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix} \]

\[ S_{ij} = \begin{pmatrix} S_{xx} & S_{xy} & S_{xz} \\ S_{yx} & S_{yy} & S_{yz} \\ S_{zx} & S_{zy} & S_{zz} \end{pmatrix} \]

\(<110>\) slip direction

Euler angles \((\Psi, \theta, \Phi)\)

\{001\} slip plane

Traces of all possible planes on the grain surface.
« In-situ » SEM - FFM - DIC

Fine grains (< 100 µm)

Strain rate = \sim 10^{-4} s^{-1}

50% strain localizes at interfaces.
Bimodal grain-size distribution
(< 100 and 200-400 µm)

Strain rate = \(~ 10^{-4}\text{s}^{-1}\)
Interfacial localization, CSP - GBS interactions and damage
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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-operative 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