

Towards quantified microstructures through microphysical understanding

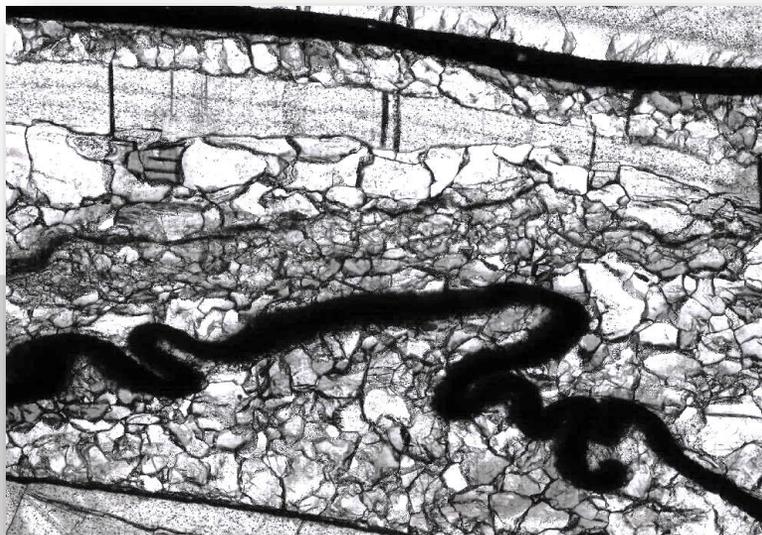
CCC program – Microstructural analysis and creep laws

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and Teams

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Cavern
Closure
Consortium

With contributions by Richard Bakker,
Marinus den Hartogh, Els Wijermars,
Martijn ter Braack (Nobian)



Y.A. Kuperus, C.A. Visser, D. Wiersma



Johannes Herrmann, Christopher Rölke,
and Dirk Naumann (IfG)



In Memoriam

Pierre Bérest
1950 - 2022



János L. Urai
1953 - 2023



Many thanks to
Chris J. Spiers and
Co-workers

KEM-17 micro-scale: first conclusion

Prediction of cavern convergence after abandonment requires extrapolation of engineering constitutive laws to heterogeneous salt at strain rates much lower than in the laboratory. This extrapolation is not **based on the available microphysical understanding of the deformation.** Accuracy of predictions of convergence can be strongly improved by including this knowledge.

Towards quantified microstructures through microphysical understanding

Long term cavern behaviour:

Total strain rate:

$$\dot{\epsilon} = \dot{\epsilon}^{el} + \dot{\epsilon}^{tr} + \dot{\epsilon}^{dc} + \dot{\epsilon}^{ps}$$

elastic-
transient-
dislocation-
pressure-
-creep

solution-

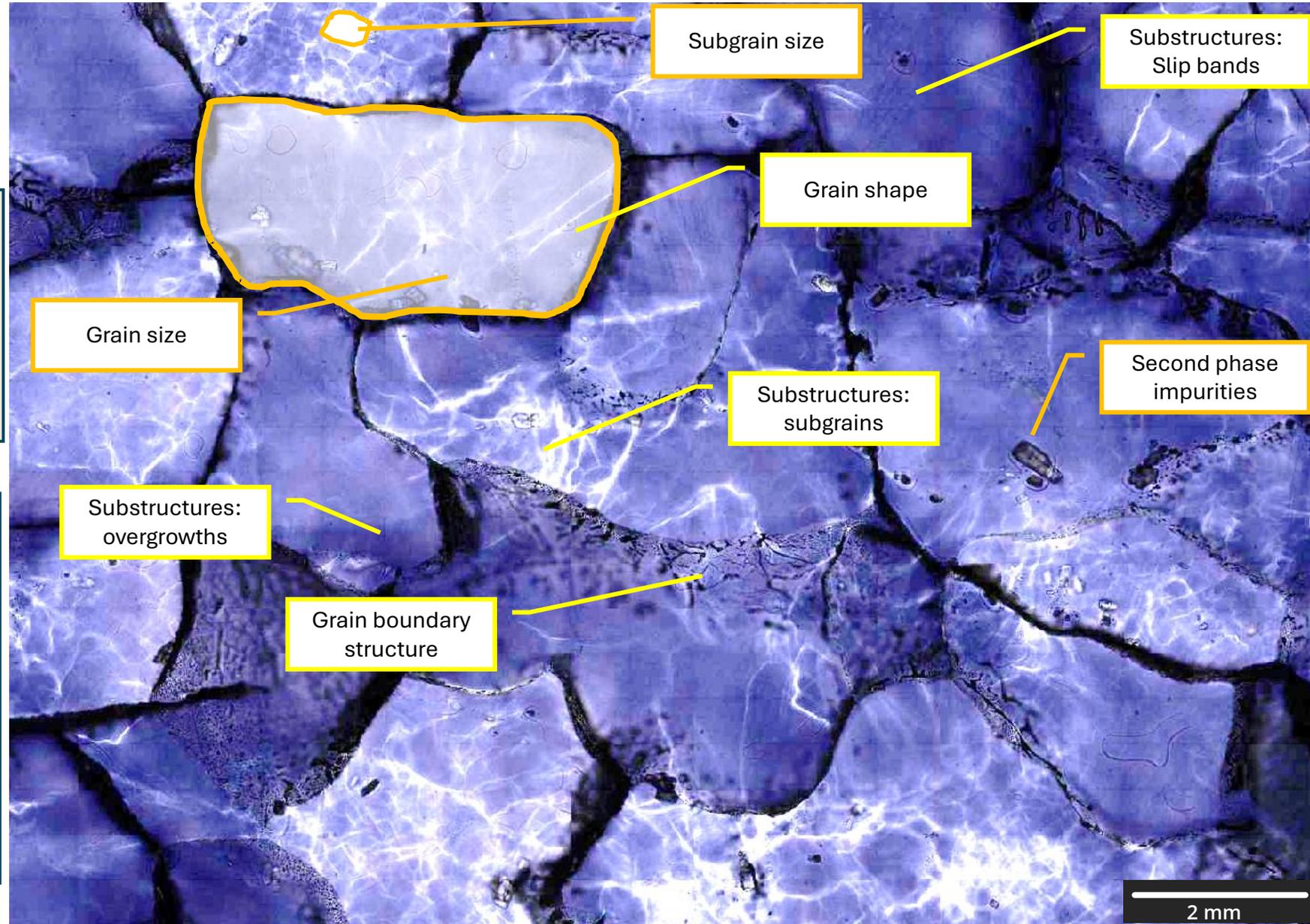
Dislocation creep:

$$\dot{\epsilon}^{dc} = A_{dc} e^{-\frac{Q_{dc}}{RT}} \sigma^n$$

Pressure solution creep:

$$\dot{\epsilon}^{ps} = A_{ps} e^{-\frac{Q_{ps}}{RT}} \frac{\sigma}{TD^m}$$

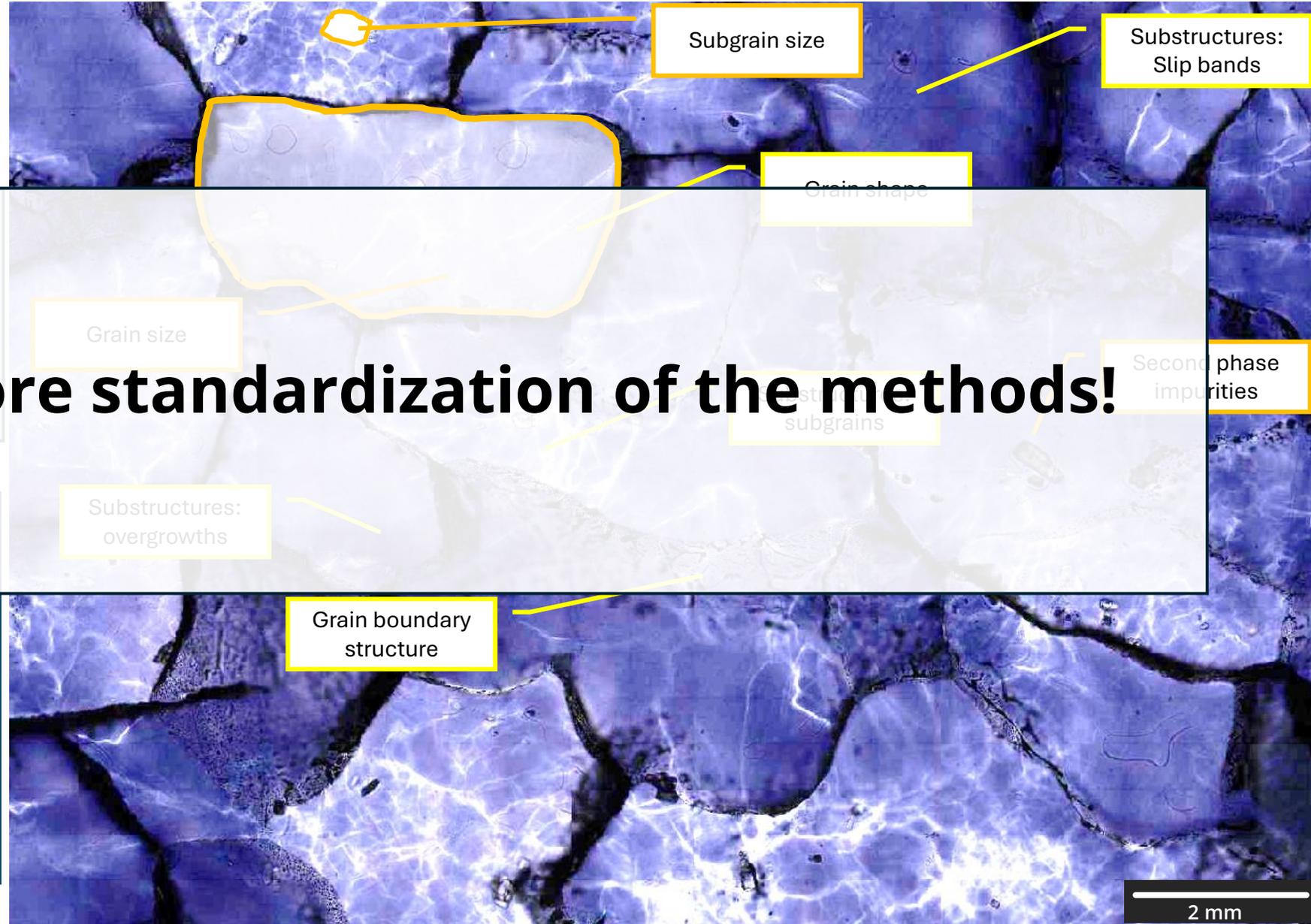
Pre-factor A_{dc} and A_{ps} , Activation energy Q_{dc} and Q_{ps} , Gas constant R , Temperature T , Grain size D , effective differential stress σ , exponent n and m



2 mm

Towards quantified microstructures through microphysical understanding

Long term cavern behaviour:



We need more standardization of the methods!

Total strain rate:

$$\dot{\epsilon} = \dot{\epsilon}^{el} + \dot{\epsilon}^{tr} + \dot{\epsilon}^{dc} + \dot{\epsilon}^{ps}$$

elastic-transformation-dislocation-precipitation

Dislocation creep:

$$\dot{\epsilon}^{dc} = A_{dc} e^{-\frac{Q_{dc}}{RT}} \sigma^n$$

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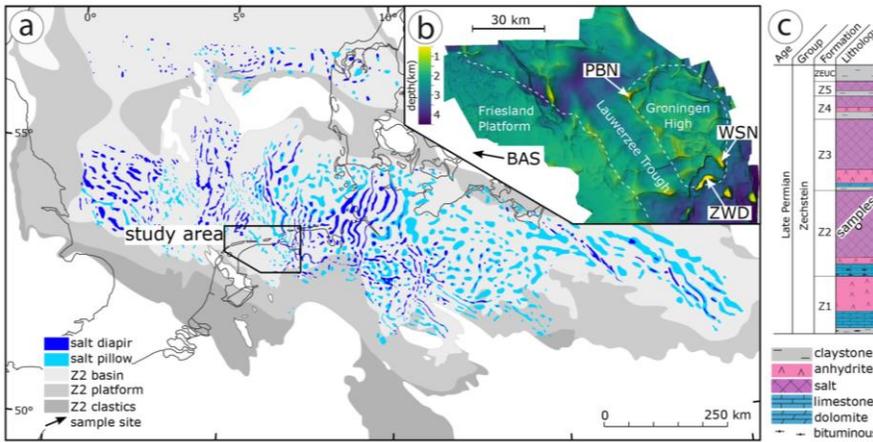
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KEM-17 micro-scale: first conclusion

Prediction of cavern convergence after abandonment requires extrapolation of engineering constitutive laws to heterogeneous salt at strain rates much lower than in the laboratory. This extrapolation is not **based on the available microphysical understanding of the deformation.** Accuracy of predictions of convergence can be strongly improved by including this knowledge.

A first look at microstructures of rock salt core from two different salt cavern fields in the Netherlands shows that these are heterogeneous and quite different. A full materials science-based analysis will much improve predictions of abandoned caverns in these cases

Natural Laboratory



KEM-17 – Over-pressured salt solution mining caverns and possible leakage mechanisms

Knowledge Programme on Effects of Mining, The Dutch Ministry of Economic Affairs, Urai et al., 2019, Baumann et al., 2019 and Brouard et al., 2019 (CCC)

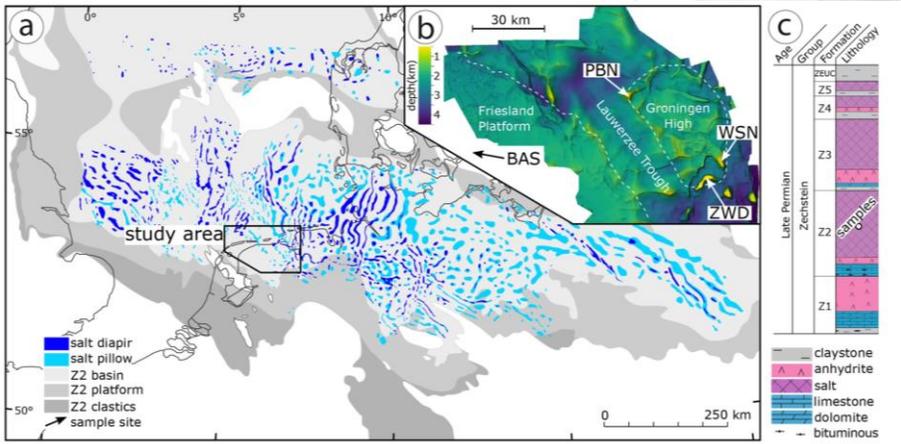
Large grain-size-dependent rheology contrasts of halite at low differential stress (...)

Barabasch, Schmatz, Klaver, Urai et al. 2023, Solid Earth, 14, 271–291.

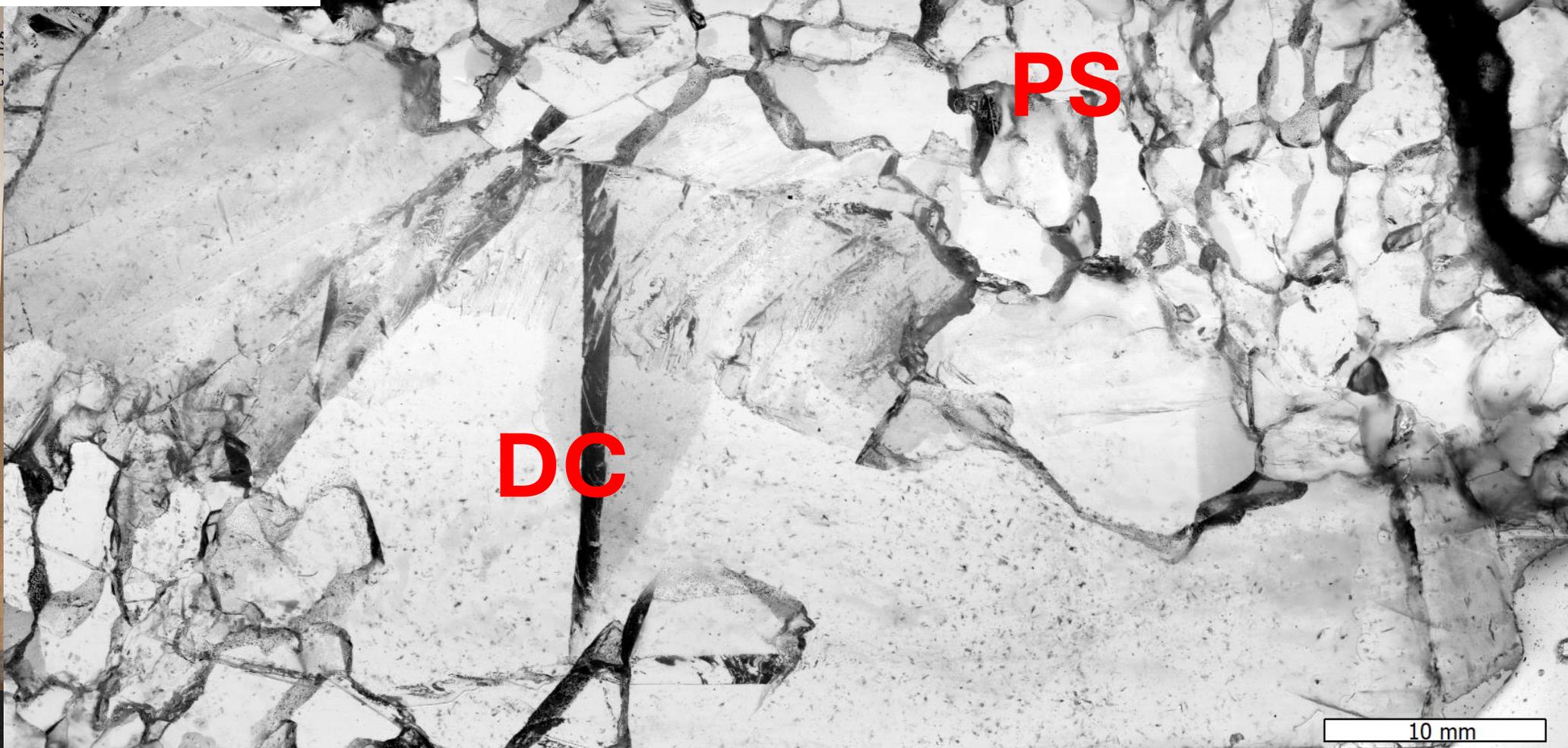
Abandonment of solution mining cavern fields

2021-ongoing, CCC in cooperation with Nobian Industrial Chemicals B.V.





Natural Laboratory



Natural Laboratory

Conclusions

Halite mega-grains deform by dislocation creep

Recrystallized Halite deforms by dislocation creep and pressure solution

Rheology of the salt is determined by the properties of the mixture

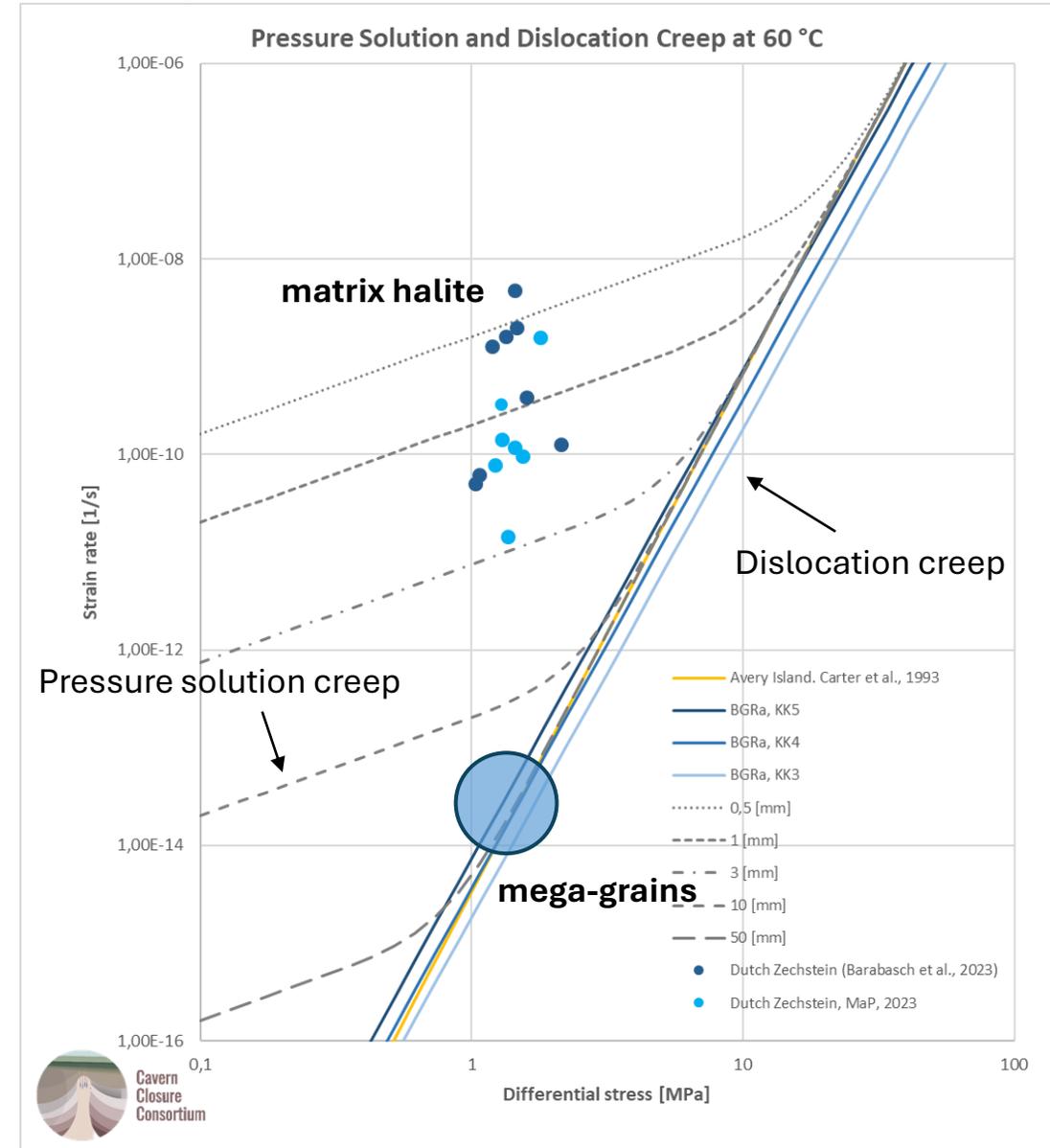
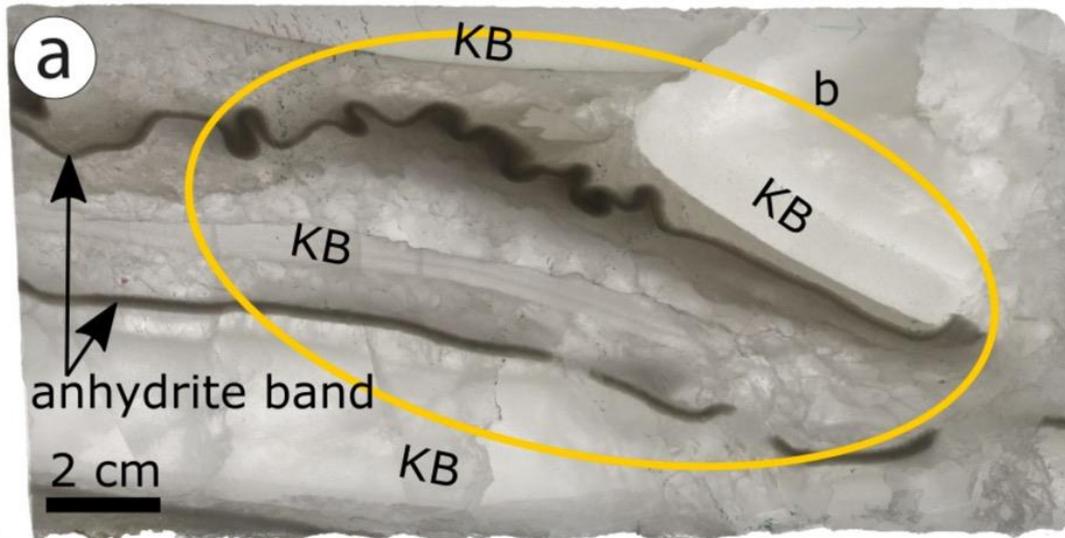
$$\dot{\epsilon}_{DC} = A e^{-\frac{Q_{DC}}{RT}} (\sigma_1 - \sigma_3)^n \quad (1)$$

for dislocation creep and Eq. (2)

$$\dot{\epsilon}_{PS} = B e^{-\frac{Q_{PS}}{RT}} \left(\frac{\sigma_1 - \sigma_3}{TD_G^m} \right) \quad (2)$$

for solution-precipitation creep, with the sum of both being the total strain rate (Eq. 3):

$$\dot{\epsilon} = \dot{\epsilon}_{PS} + \dot{\epsilon}_{DC} \quad (3)$$



KEM-17 micro-scale: first conclusion

Prediction of cavern convergence after abandonment requires extrapolation of engineering constitutive laws to heterogeneous salt at **strain rates much lower than in the laboratory.** This extrapolation is not **based on the available microphysical understanding of the deformation.** Accuracy of predictions of convergence can be strongly improved by including this knowledge.

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CREEP TESTS PERFORMED IN MINE DRIFTS IN THE 0.05-4.5 MPa RANGE

Pierre Bérest, Hakim Gharbi
LMS, Ecole Polytechnique, France

Benoit Brouard
Brouard Consulting, France

Salt Mechanics Webinar on “Creep at low deviatoric stresses” – 12 October 2021

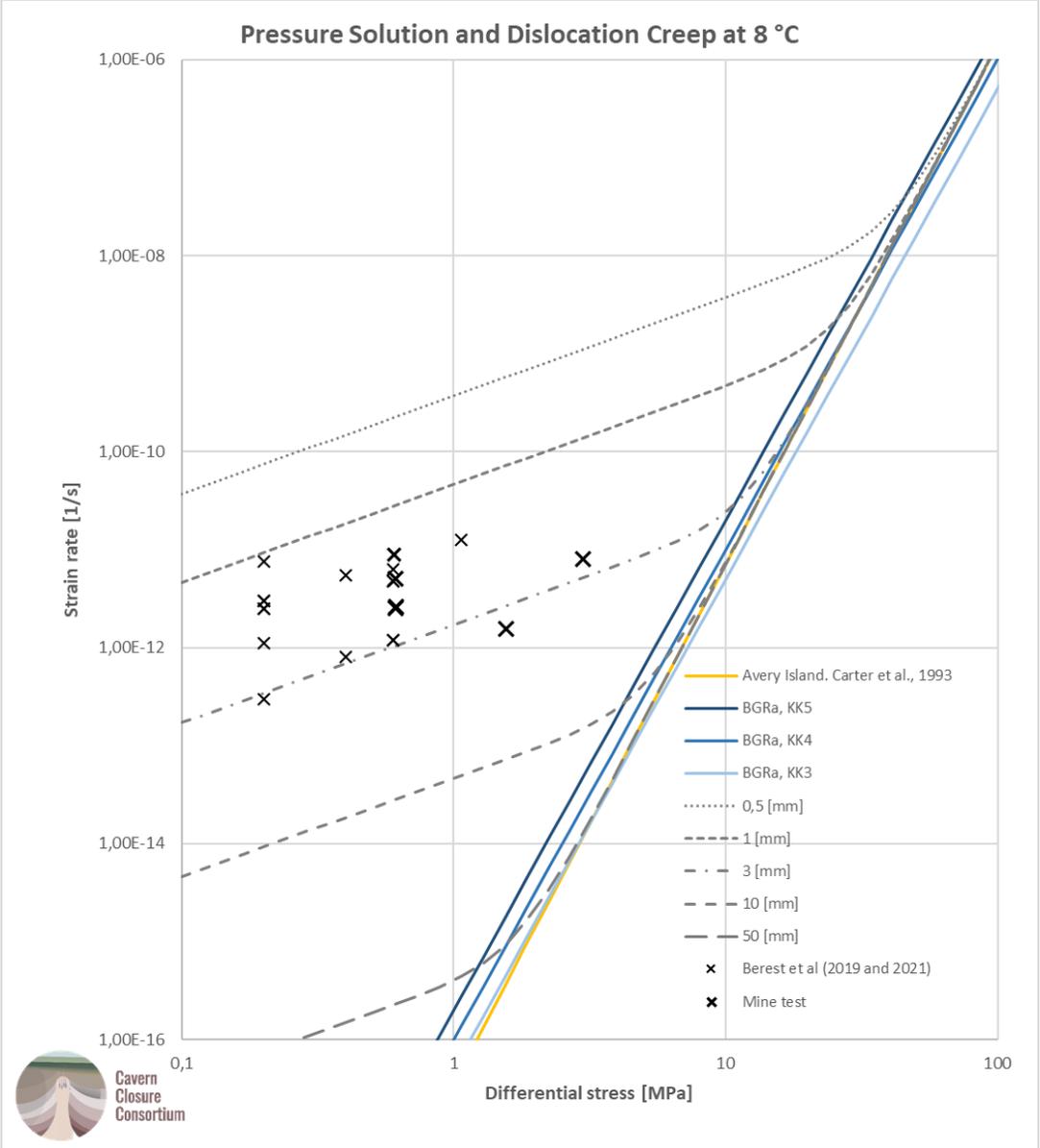
Bérest et al.: Creep tests at low deviatoric stress (<5MPa) under stable conditions in a salt mine

Salt	Load (MPa)	Duration (months)	Final Strain rate ($\times 10^{-12}$ /s)	Average grain size (mm)
Landes #1	0.60	12	9.0	4
Avery Island #2	0.60	8	4.8	8
Hauterives	0.60	8	1.2	10

Strain rate is a decreasing function of grain size
 data are too rough to fully confirm $1/D^3$ as predicted by Spiers, Urai & et. al

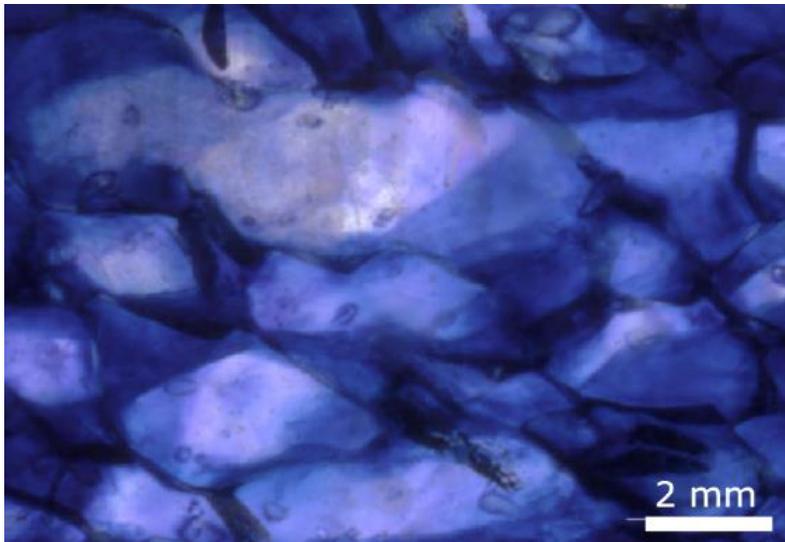
Bérest et al.: Salt Mechanics Webinar on “Creep at low deviatoric stresses” – 12 October 2021
Bérest et al. 2023

Trend confirmed by a number of recent tests 

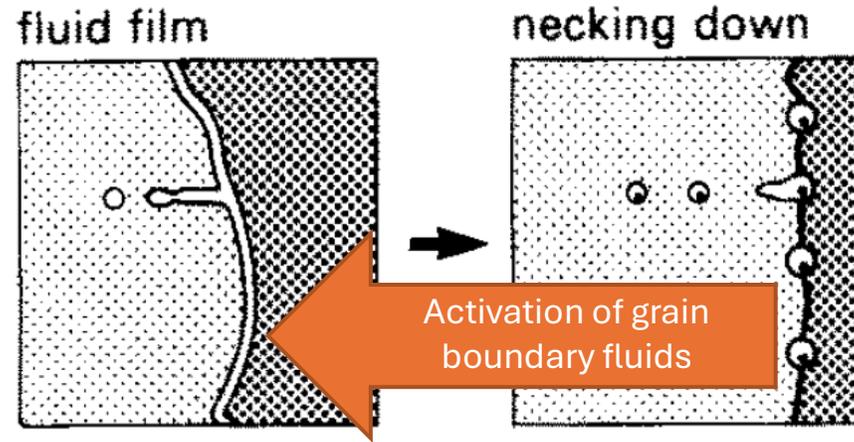


Evidence from the microstructures: Is PS-creep active during the mine test?

WHAT DO WE EXPECT?

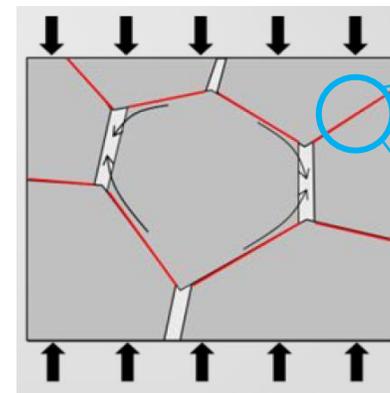


Activation of grain boundary fluids



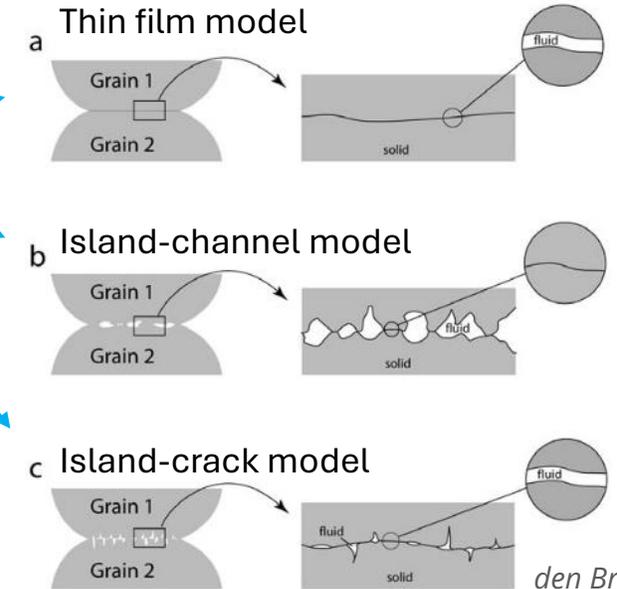
Drury & Urai 1990

Grain overgrowths and grain boundary modification



Stress-induced dissolution and precipitation
(Pressure Solution Creep)

Sketch with courtesy of C.J. Spiers

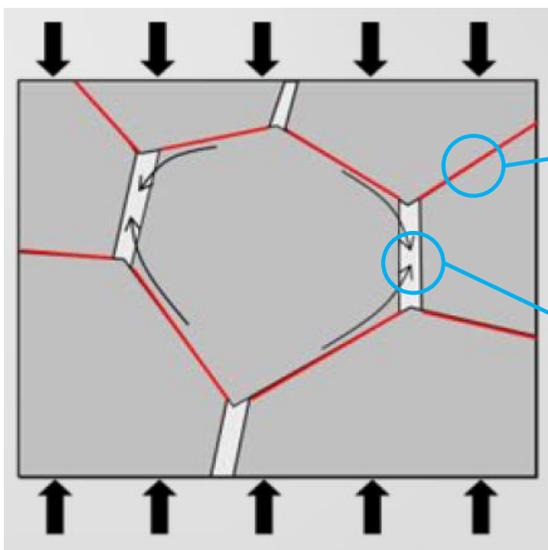


den Brok et al., 2002

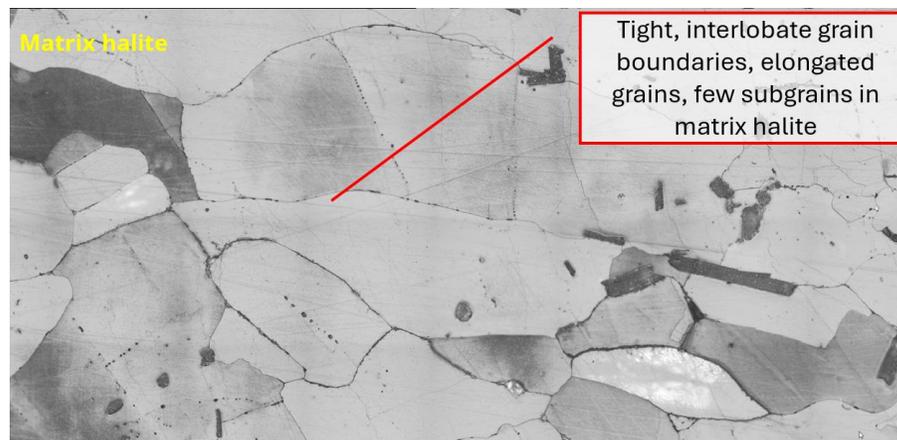
Evidence from the microstructures: Is PS-creep active during the mine test?

Samples were creep-tested at 0.6MPa and 3MPa deviatoric stress for the period of 8 month

WHAT WE OBSERVE:

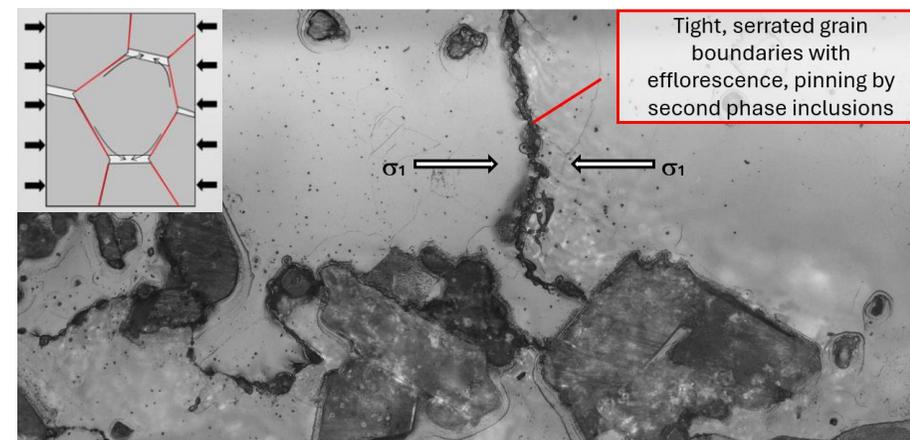


Stress-induced dissolution and precipitation
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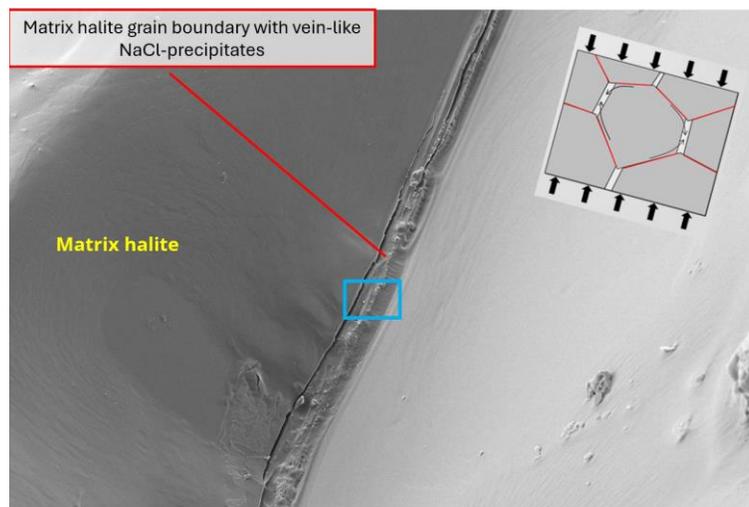
Initial sample

500µm



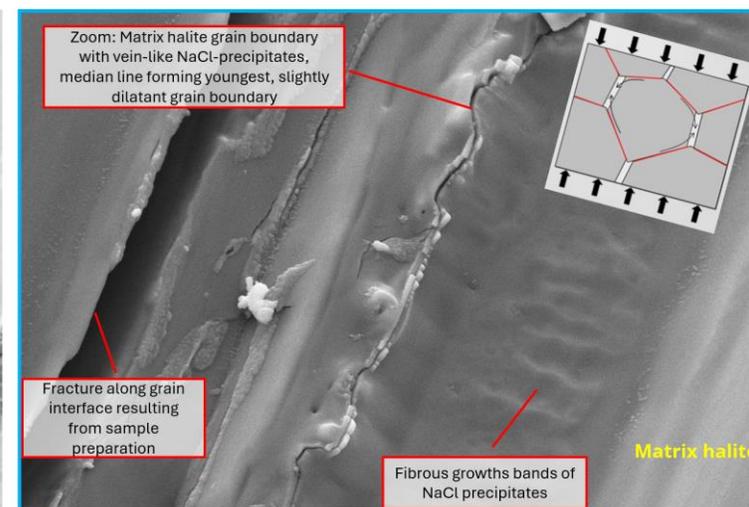
Creep-tested sample

100µm



Creep-tested sample

20µm



Matrix halite

2µm

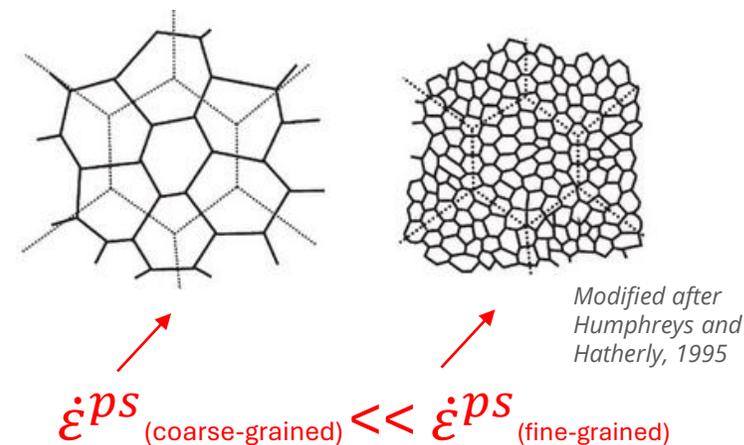
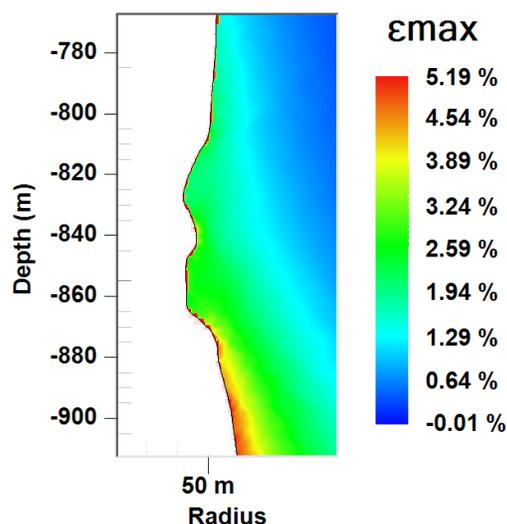
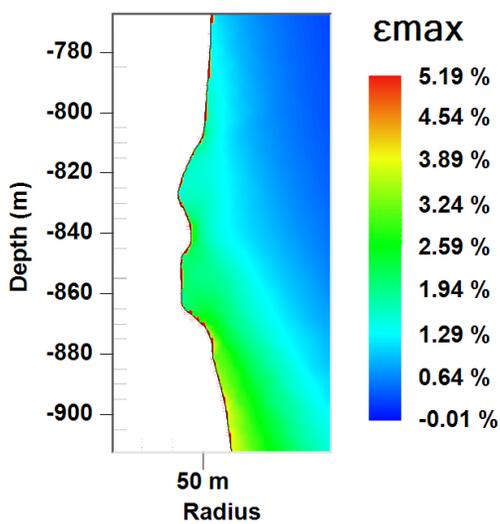
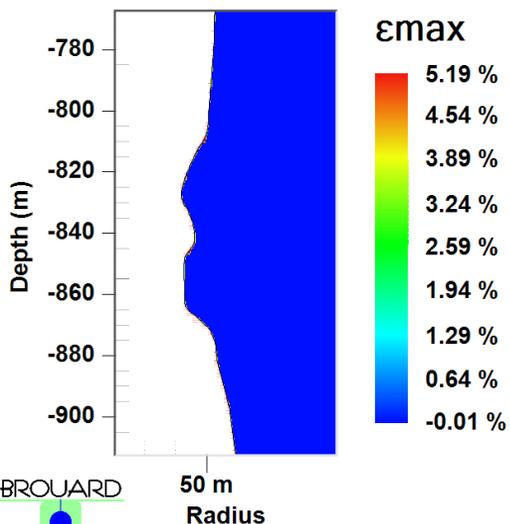
Urai et al.: long-term creep evolution in the vicinity of solution-mined cavern after abandonment

Maximum principal strain in the near field of a solution-mined cavern after plugging and abandonment

50 years later

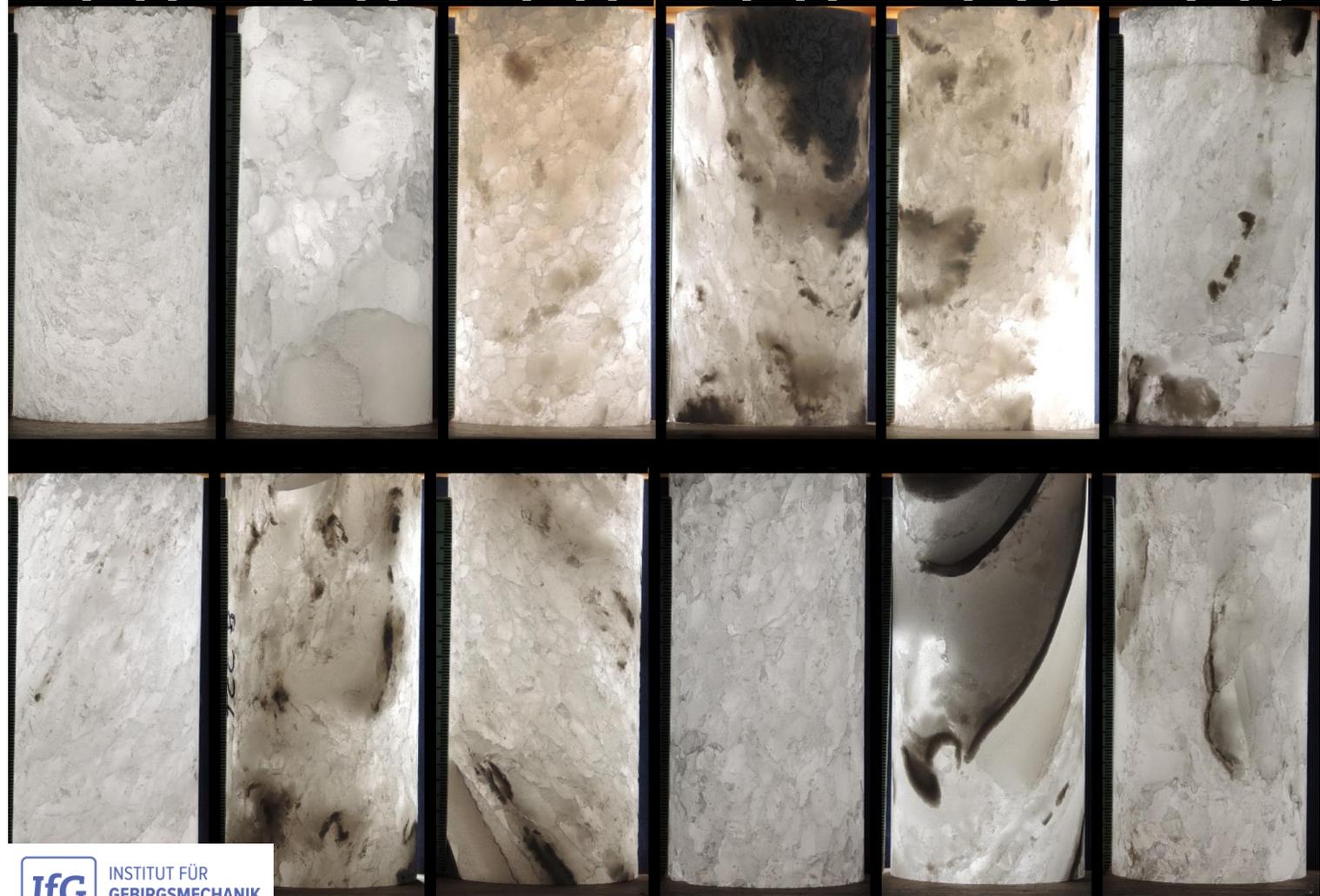
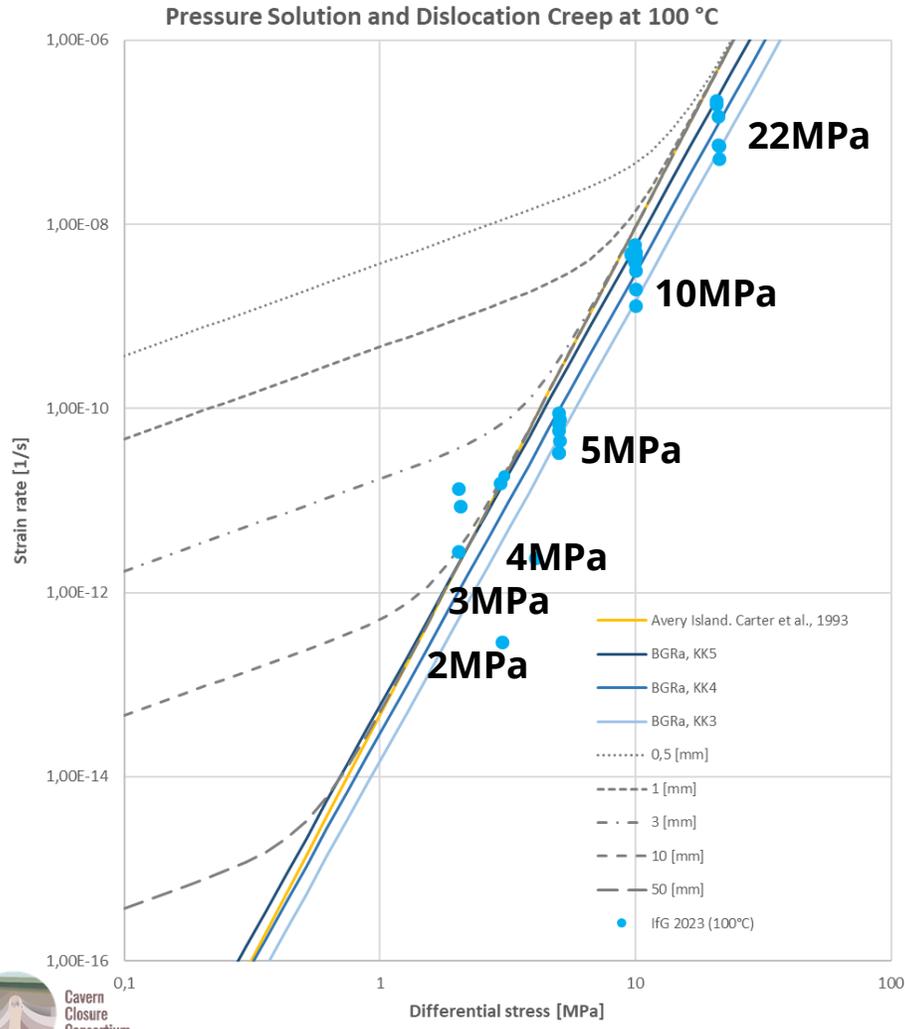
210 years later

The idea: Stress-strain field at cavern wall after plugging and abandonment may lead to diminished grain size, which accelerates creep rates of grain size sensitive creep close to the cavern wall.



Assessing the creep behavior of natural salt with diminished grain size

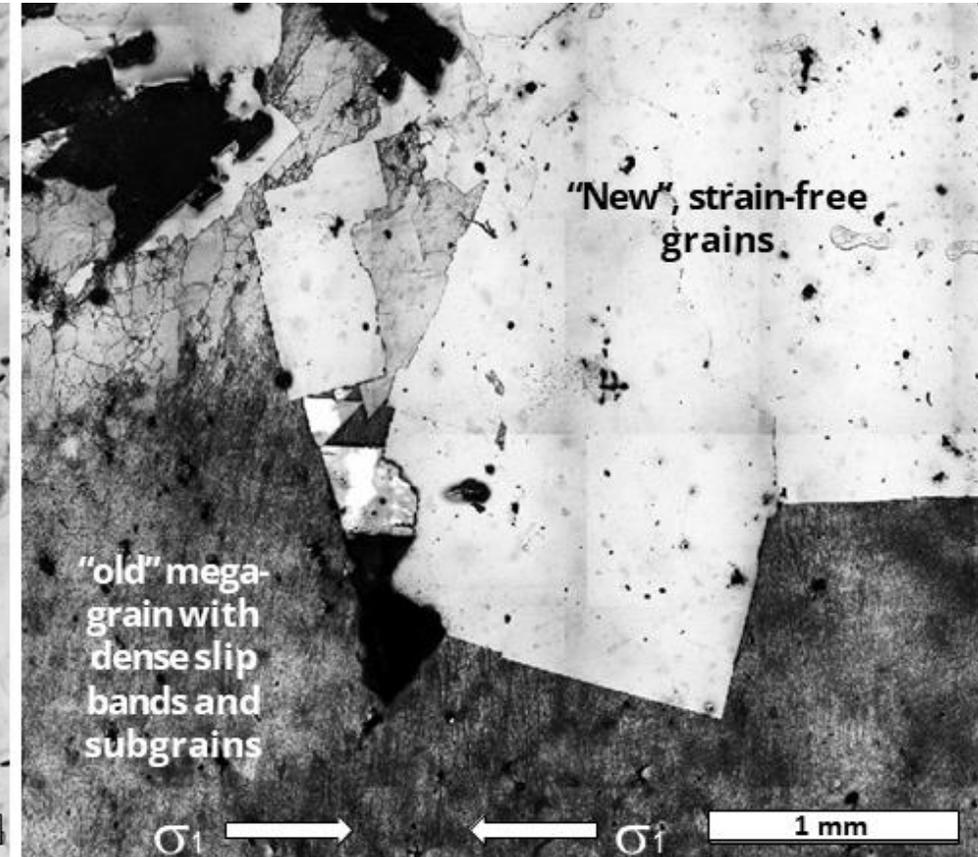
Natural rock salt from the Dutch Zechstein with matrix halite, halite mega-grains, and anhydrite



Deformed at IfG, Leipzig

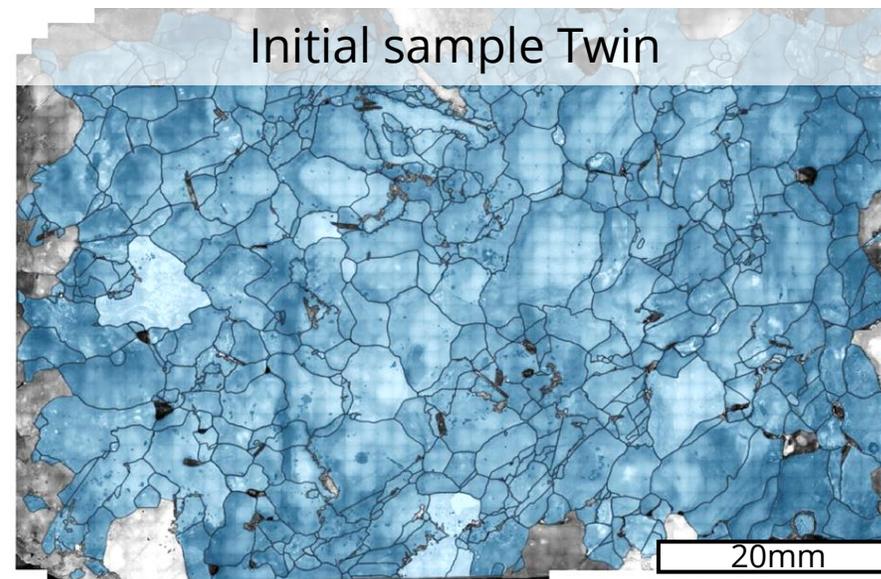
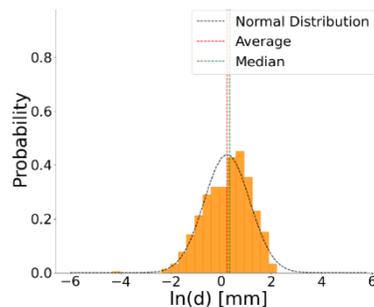
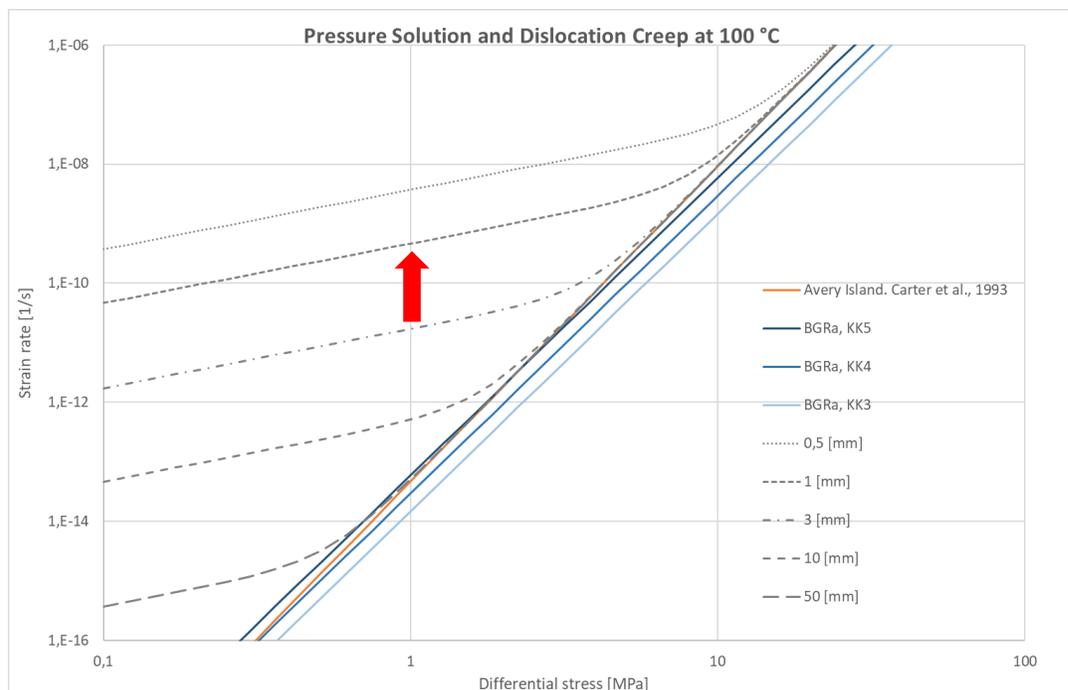
Grain size evolution after the first load step

- Pre-test at 30MPa differential stress for the duration of approx. 24hours
- Around 25 area% of matrix halite recrystallized during pre-test by fluid assisted grain boundary migration recrystallization

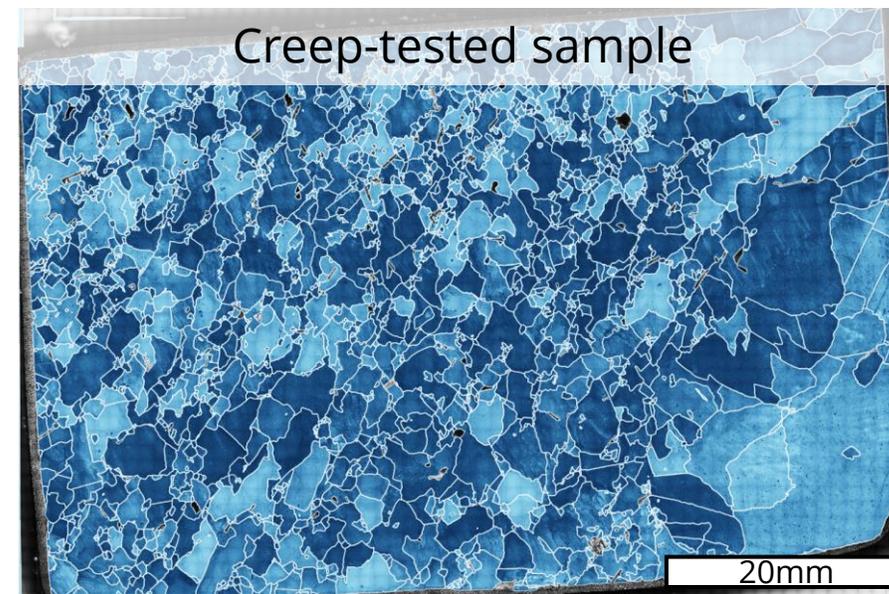
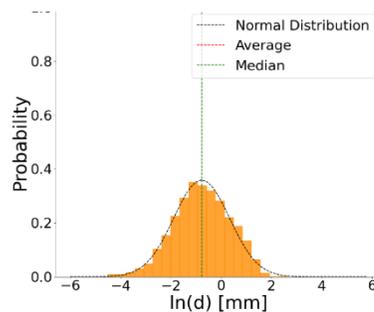


Grain size evolution after the final load step

- The salt is nearly fully recrystallized
- Different load steps are still recognizable (subgrain size)
- Median grain size was reduced by a factor of ~3
- 3x smaller grain size results in 100x faster strain rate for PS-creep

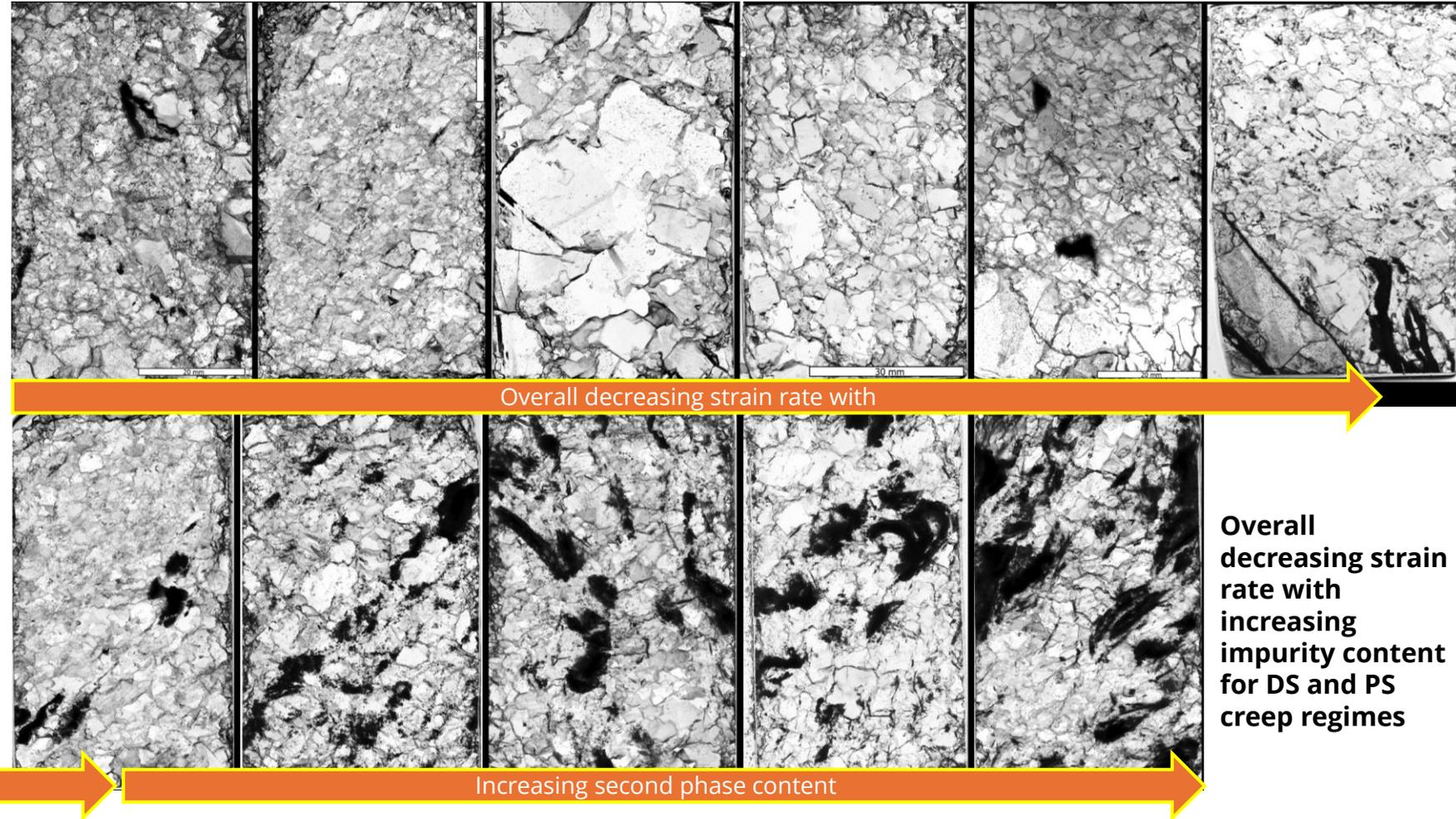


3x
smaller grain size

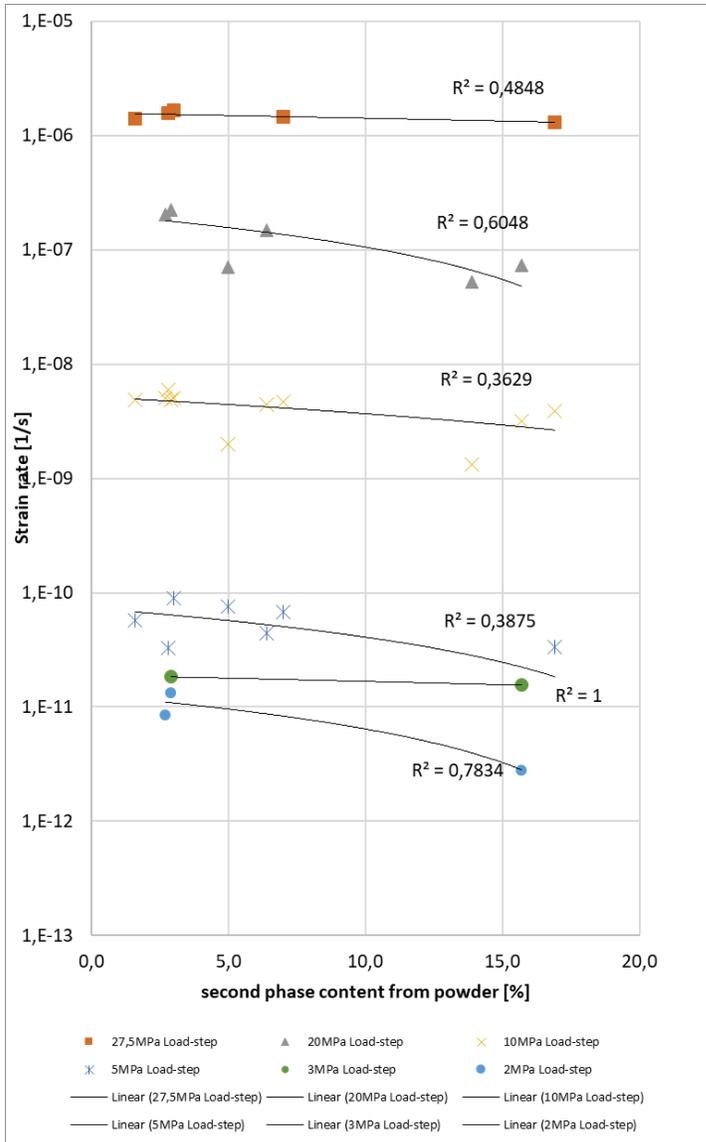


Effect of anhydrite content on the strain rate

Thin sections in transmitted light of 11 creep-tested samples sorted by overall strain rate



Overall decreasing strain rate with increasing impurity content for DS and PS creep regimes



Summary

The natural laboratory:

Dislocation creep and pressure solution creep were active during natural deformation. Mixed rheology is inferred.

Sophisticated laboratory testing:

High-resolution creep-tests in a mine allow measuring PS creep at low deviatoric stress, confirmed by microstructural analysis.

High strains at the cavern wall after plugging may complicate abandonment because of the diminished grain size.

Creep is affected by the heterogeneity of the natural rock salt at all scales.

Constitutive modelling and simulations:

→ CCC presentations by Benoit Brouard and Tobias Baumann

