Rock salt rheology and permeation: critical review of existing works and recommendations for improving current practice

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

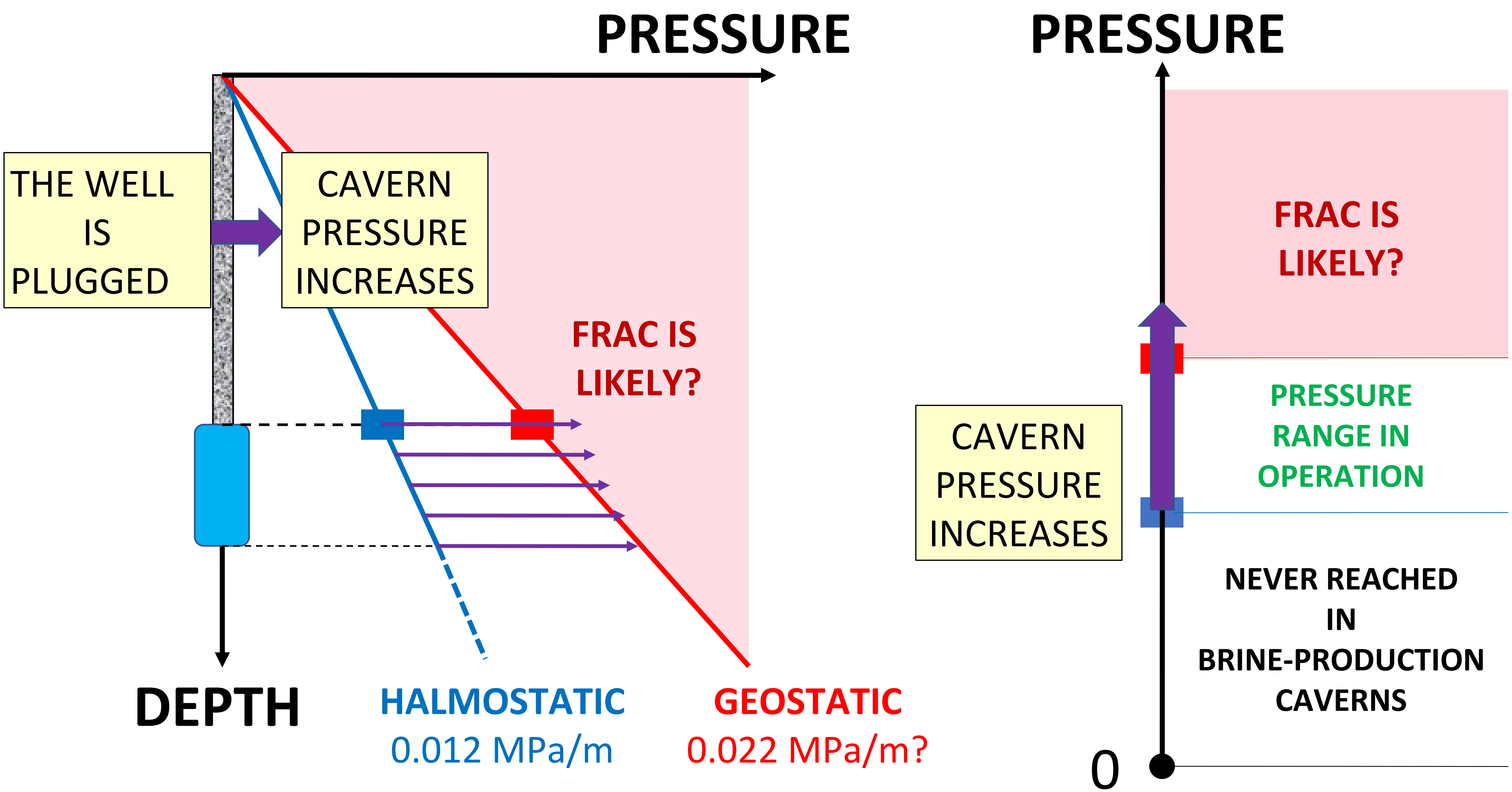
Phase 1: micro-scale processes

Supervised by:
Gerco Hoedeman, State Supervision of Mines, Ministry of Economic Affairs and Climate,

Authors: Prof. Dr. Janos L. Urai, Dr. Joyce Schmatz, Dr. Jop Klaver

Microstructure and Pores

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



IS FINAL PRESSURE LARGER THAN GEOSTATIC PRESSURE?

permeation in an abandoned salt cavern

the conventional view

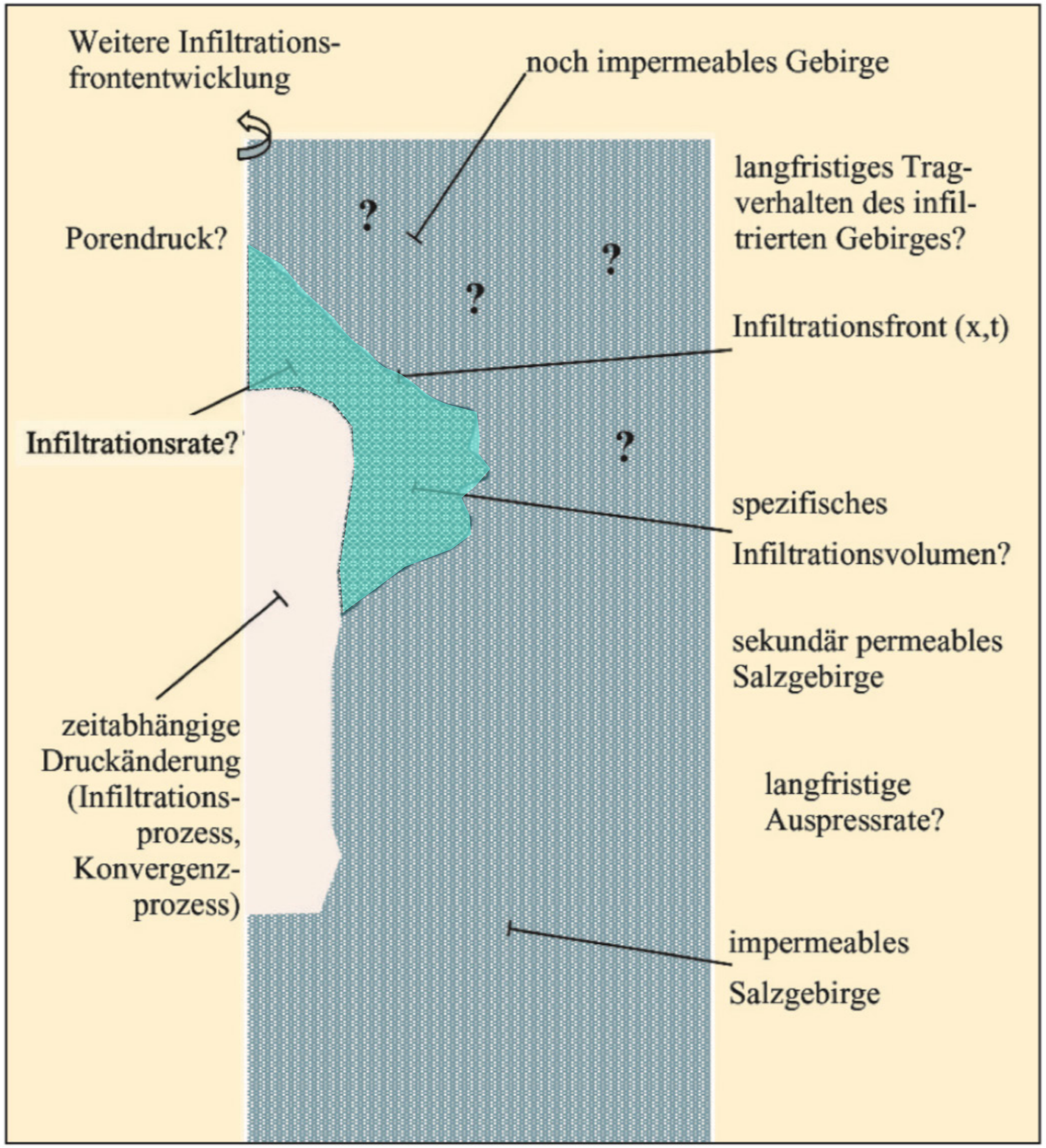


Abb. 2 Einige Fragestellungen zum Langzeitverhalten einer verschlossenen fluidgefüllten Salzkaverne

the view of this report

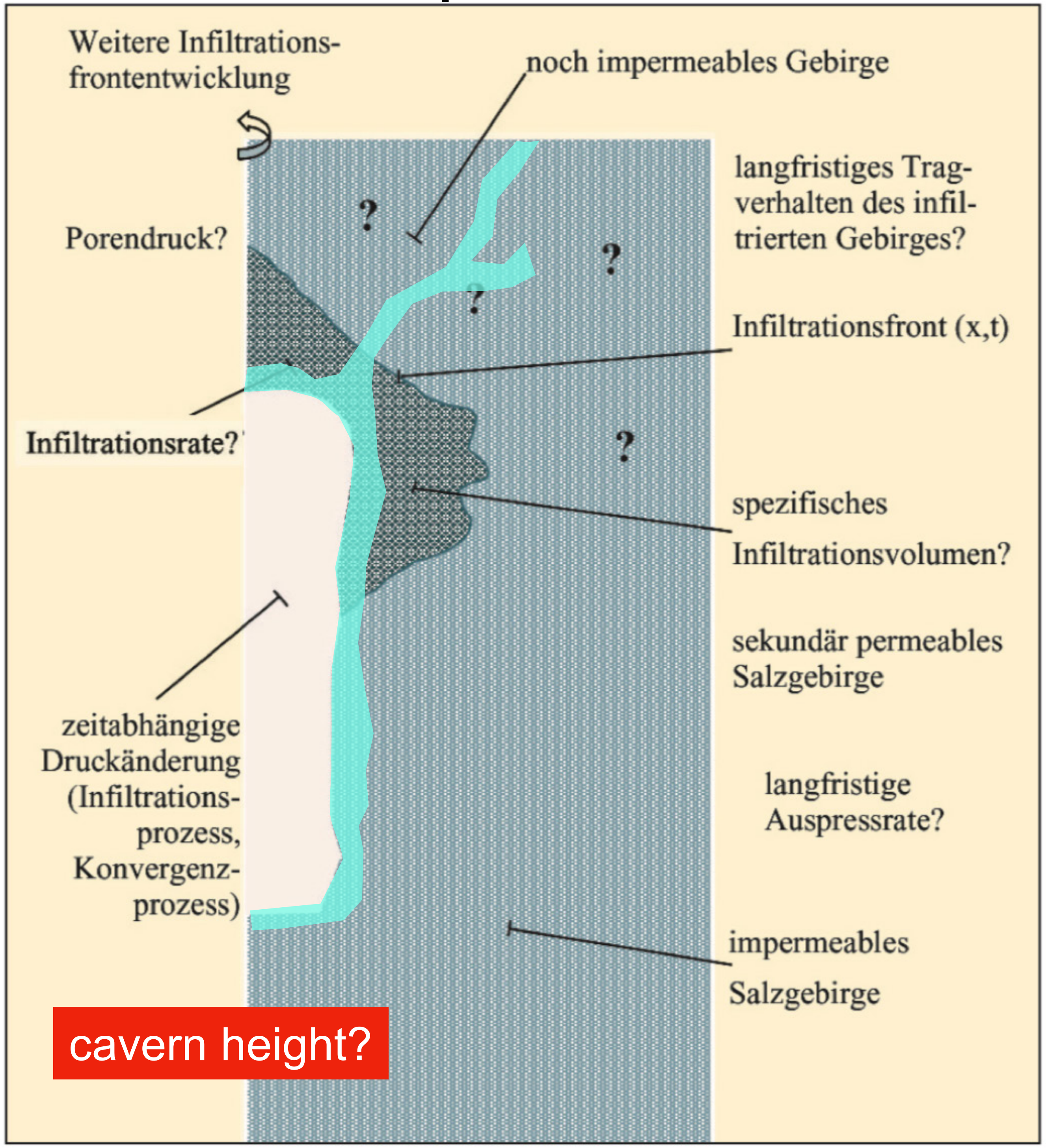


Abb. 2 Einige Fragestellungen zum Langzeitverhalten einer verschlossenen fluidgefüllten Salzkaverne

Lux, K.-H., 2006. Zum langfristigen Tragverhalten von verschlossenen solegefüllten Salzkavernen – ein neuer Ansatz zu physikalischer Modellierung und numerischer Simulation: Rechnerische Analysen und grundlegende Erkenntnisse [Long-term behaviour of sealed liquid-filled salt cavities – a new approach for physical modelling

Recommendation: incorporate materials science in cavern engineering

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. Predictions can be strongly improved by including this knowledge.

Prediction of **brine migration** after abandonment requires extrapolation of engineering constitutive laws to heterogeneous salt and rates much lower than in the laboratory. This extrapolation is not based on microphysical understanding of permeation. Predictions can be strongly improved by including this knowledge.

Existing experiments and microstructural arguments show that kinetics of permeation in high pressure caverns vary with microstructure and impurities. We propose that **permeation after cavern abandonment will be strongly heterogeneous and localised**, and not diffuse over large volumes as predicted by current models.

The controversy on salt rheology

Very Slow Creep Tests on Salt Samples

Pierre Bérest[1*], Benoit Brouard[2], Dieter Bruckner[3], Kerry DeVries[4], Hakim Gharbi[1], Grégoire Hévin[5], Gerd Hofer[6], Christopher Spiers[7] Stefan Stimmisher[6], Janos L. Urai[8]

8th US/German Workshop on Salt Repository Resea

6-8, 2017

Langer (1984) stated that reliably extrapolating the creep equations at low deformation rates can only be carried out on the basis of deformation mechanisms. The micro-mechanisms that govern salt creep have been discussed by Munson and Dawson (1984), Langer (1984), Blum and Fleischman (1988), and Hampel (2015). A deformation-mechanism map

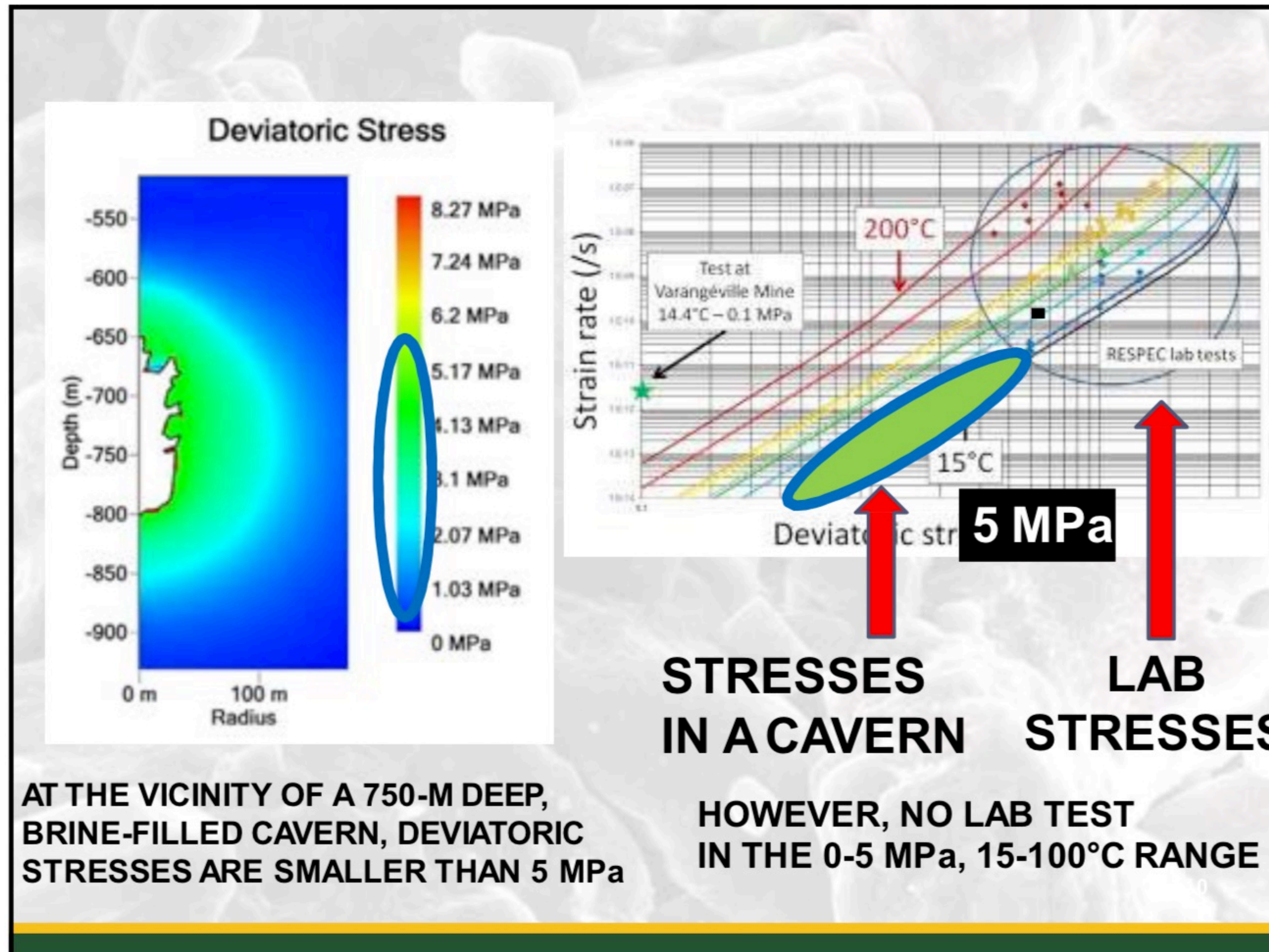
Rock Mechanics and Rock Engineering
https://doi.org/10.1007/s00603-019-01778-9

ORIGINAL PAPER

Very Slow Creep Tests on Salt Samples

Pierre Bérest[1] · Hakim Gharbi[1] · Benoit Brouard[2] · Dieter Brückner[3] · Kerry DeVries[4] · Grégoire Hévin[5] · Gerd Hofer[6] · Christopher Spiers[7] · Janos Urai[8]

Received: 17 October 2018 / Accepted: 1 March 2019



Very Slow Creep Tests on Salt Samples

CREEP TESTS ARE PERFORMED IN A RANGE OF DEVIATORIC STRESSES (LARGER THAN 5 MPa) WHICH IS NOT THE RANGE ACTUALLY OBSERVED AT THE VICINITY OF A CAVERN.

Is extrapolation possible?

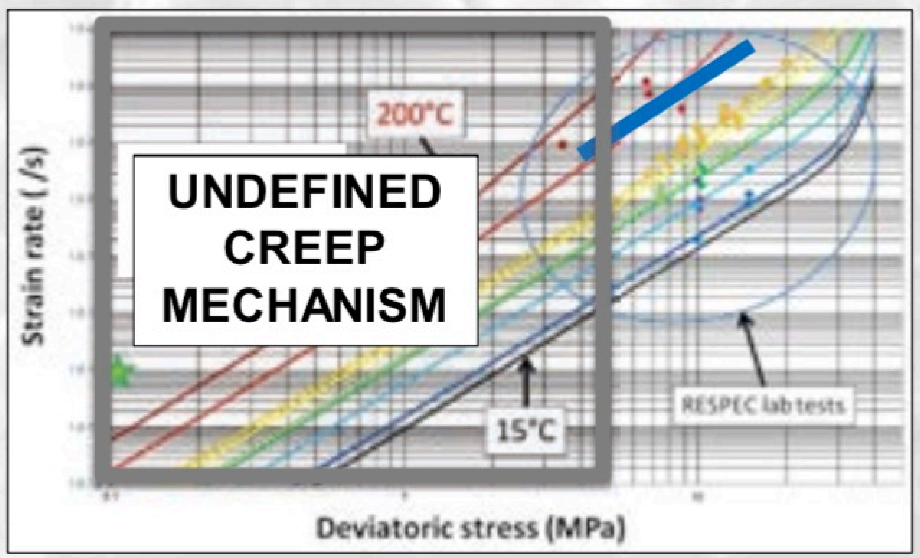
"…reliable extrapolation of the creep equations at low deformation rates can be carried out only c the basis of deformation mechanisms." (Lange 1984)

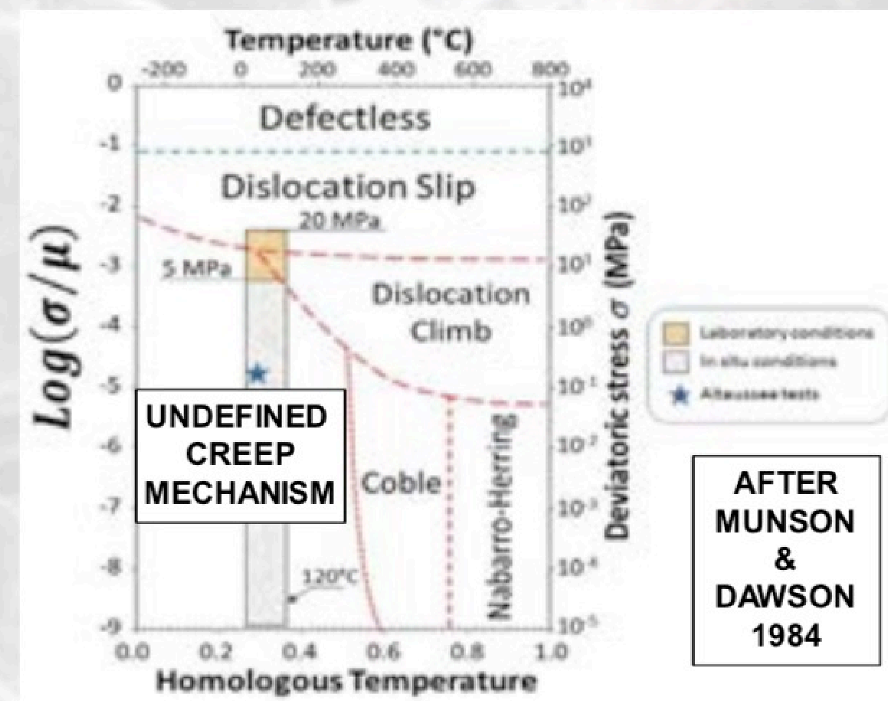
Very Slow Creep Tests on Salt Samples

Pierre Bérest[1*], Benoit Brouard[2], Dieter Bruckner[3], Kerry DeVries[4], Hakim Gharbi[1], Grégoire Hévin[5], Gerd Hofer[6], Christopher Spiers[7] Stefan Stimmisher[6], Janos L. Urai[8]

8th US/German Workshop on Salt Repository Research, Design, and Operation - Middelburg, The Netherlands September 6-8, 2017

THE SHORT ANSWER IS: NO





The micro-mechanisms responsible for salt creep in the σ < 5 MPa stress domain are poorly known

(However, Spiers & Urai suggested that pressure-solution was active in this domain)

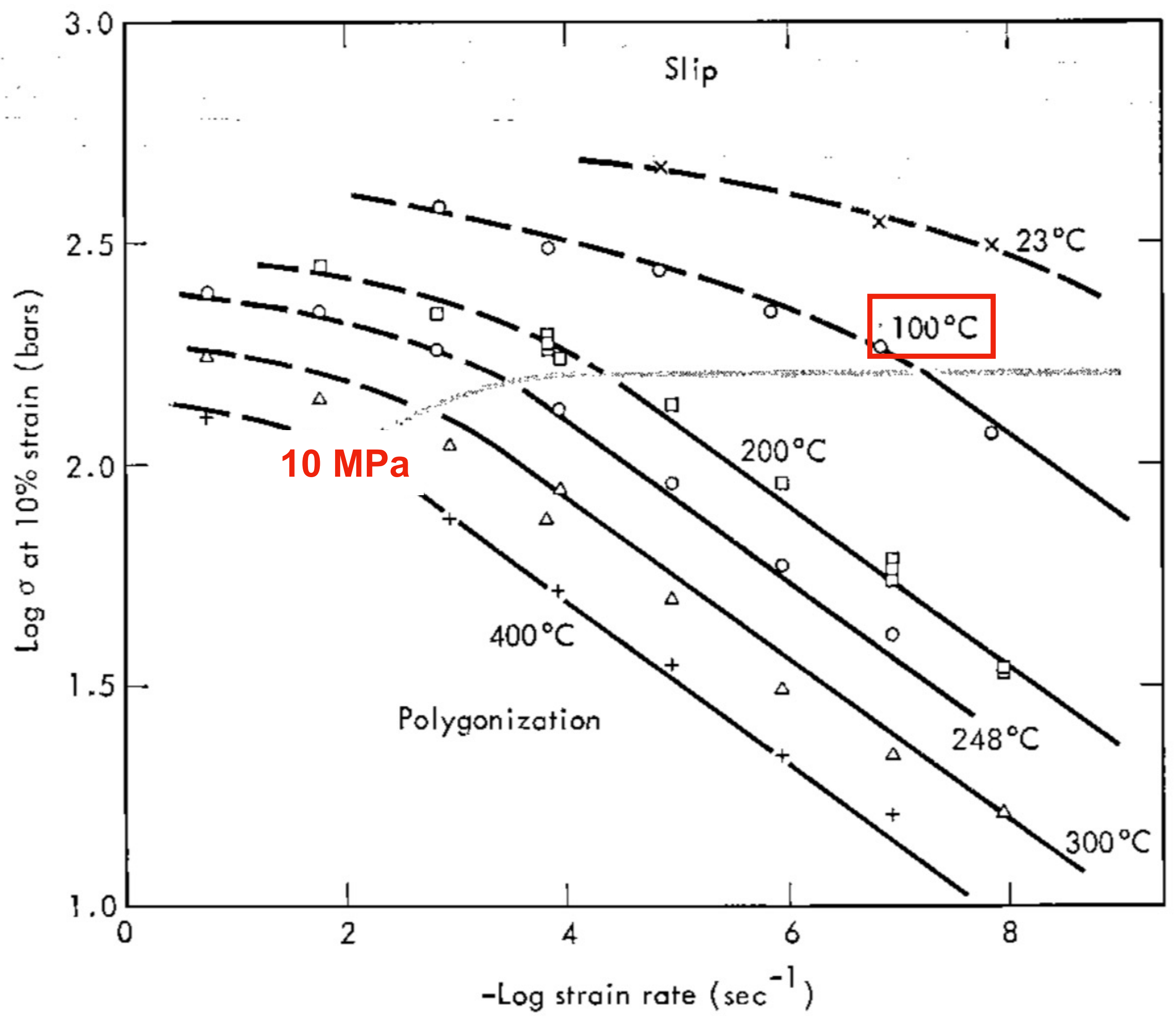
1972: Steady-State Flow in Polycrystalline Halite at Pressure of 2 Kilobars

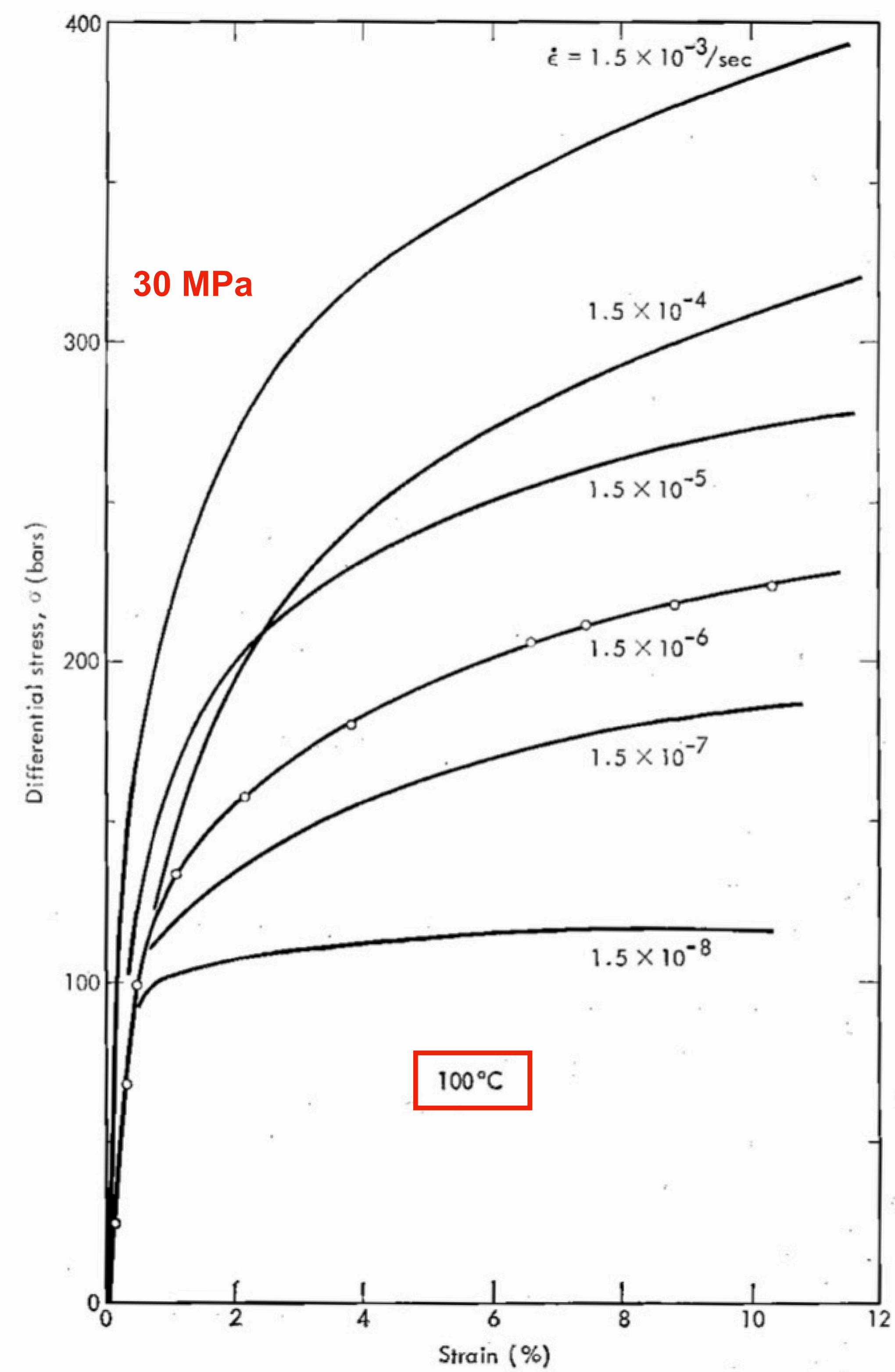
Steady-State Flow in Polycrystalline Halite at Pressure of 2 Kilobars

HUGH C. HEARD

Lawrence Livermore Laboratory, University of California
Livermore, California 94550

Early work on rock salt rheology identified the dislocation creep mechanisms, but the full range of microstructural tools to study grain boundaries, subgrain networks, recrystallization and pressure solution were not available.





Heard, H.C., **1972**. Steady-State Flow in Polycrystalline Halite at Pressure of 2 Kilobars. Flow and Fracture of Rocks, AGU Geophysical Monograph Series. 191–209.

cleavage chips with dislocation etch pits and subgrains

materials science before the Grain Boundary era

Early microstructural studies used techniques developed in solid state physics to decorate dislocations and subgrain boundaries on cleavage planes from broken samples.

These were prepared by breaking **cleavage chips** form the samples, thereby destroying the grain boundaries.

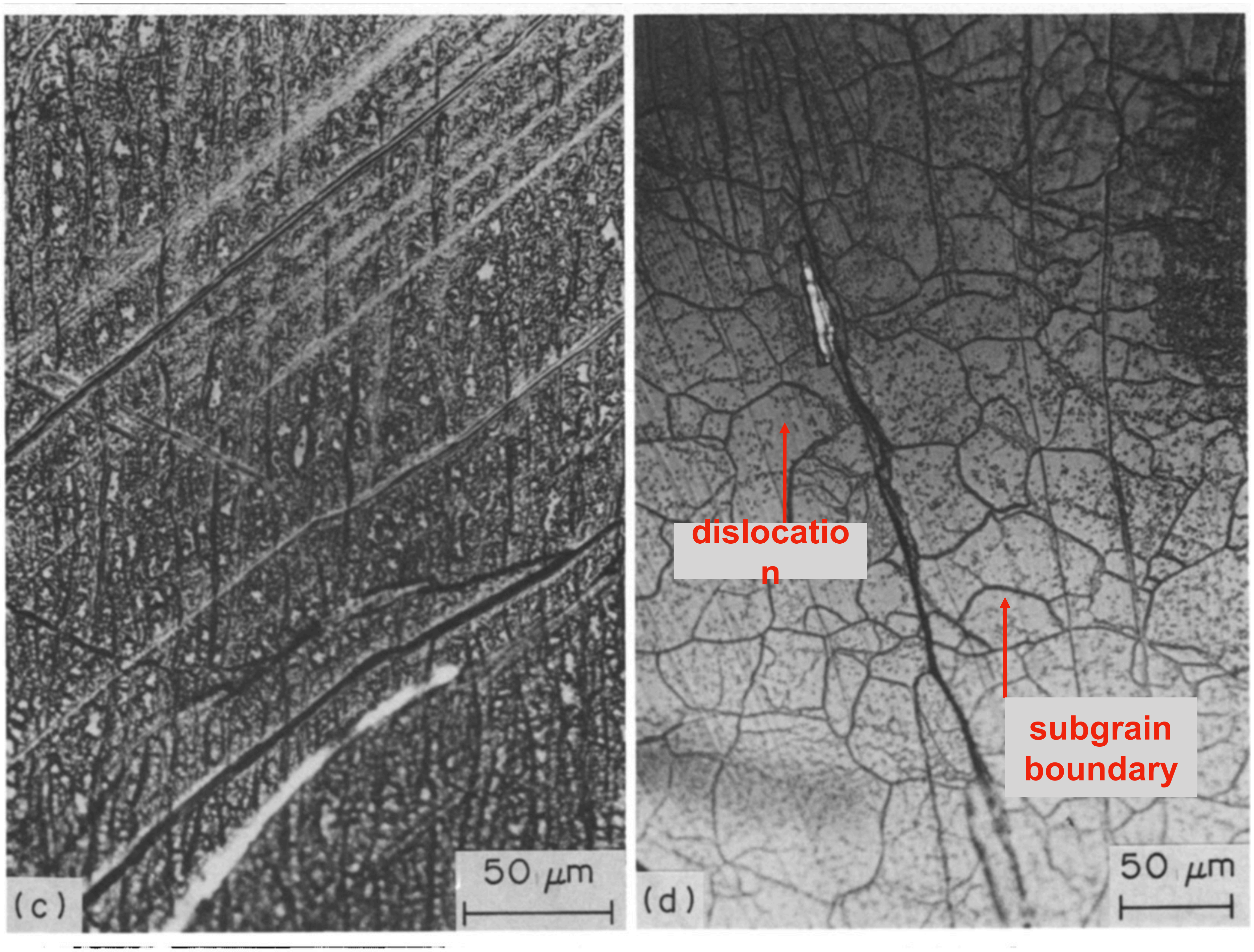
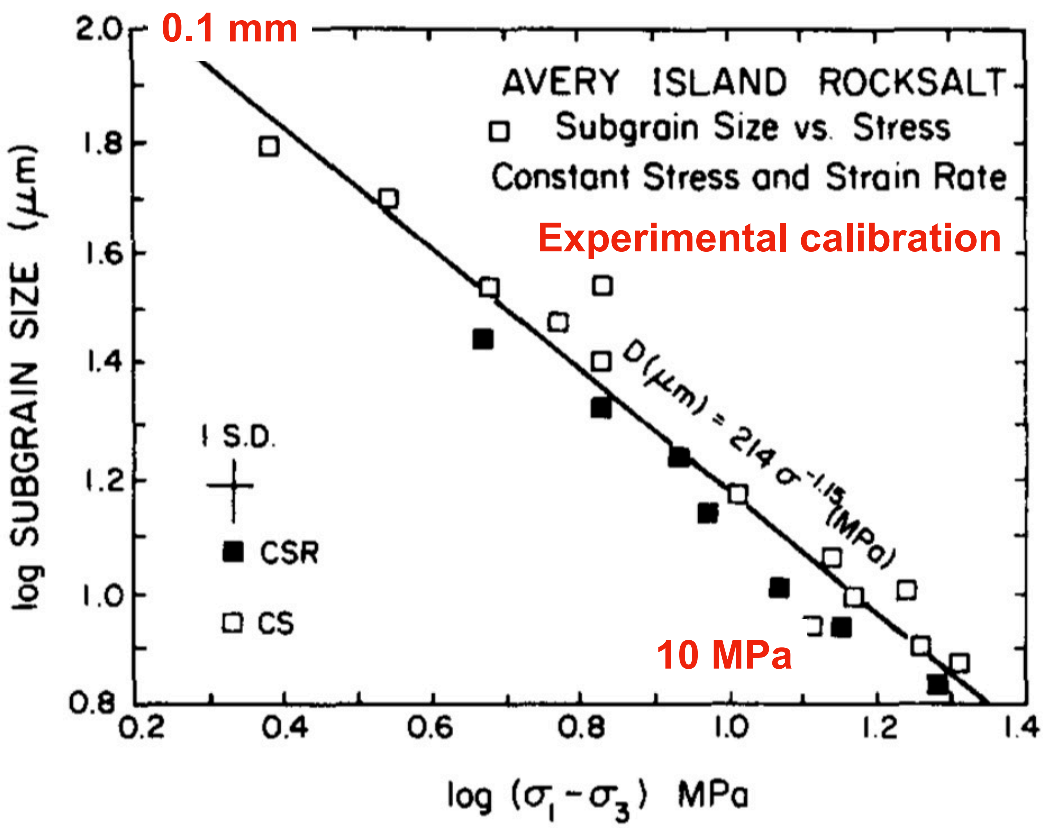
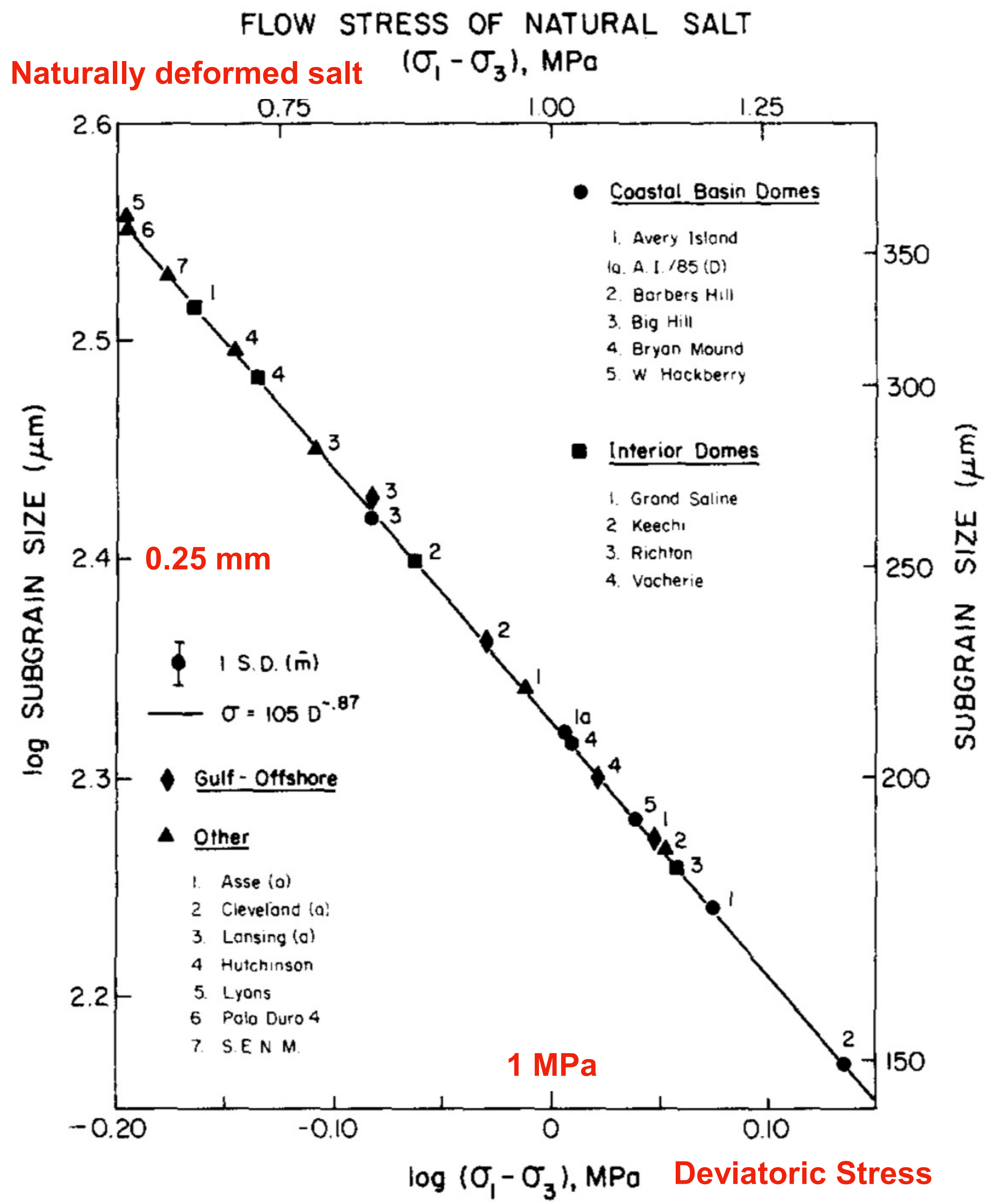


Fig. 2. Photomicrographs taken in incident light of etched surfaces (a & b) and cleavage chips from specimens 46 (a & c; 50°C, 10^{-9} s^{-1}) and 47 (b & d; 100°C, 10^{-9} s^{-1}). (a) Specimen 46; grain boundary migration is indicated by the serrated nature of the boundaries. (b) Specimen 47; boundary migration and development of networks of subgrains. (c) Specimen 46; etched cleavage chip showing high etch pit (dislocation) density and wavy slip bands (N–S-trending, dark irregular bands) indicative of extensive cross-slip (NE-trending bands are cleavage steps). (d) Specimen 47; etched cleavage chip showing subgrains, whose dark irregular boundaries enclose regions of low free dislocation density.

Carter, N.L., Horseman, S.T., Russell, J.E., Handin, J., 1993. Rheology of rocksalt. Journal of Structural Geology 15, 1257–1271

Subgrain size piezometry





This technique also allowed the calibration of subgrain size against differential stress, which can then be used to measure differential stress in naturally deformed rock salt. There is now a large dataset available on this worldwide, showing that the stress state in natural salt is not completely isotropic.

Differential stress in rock salt is typically:

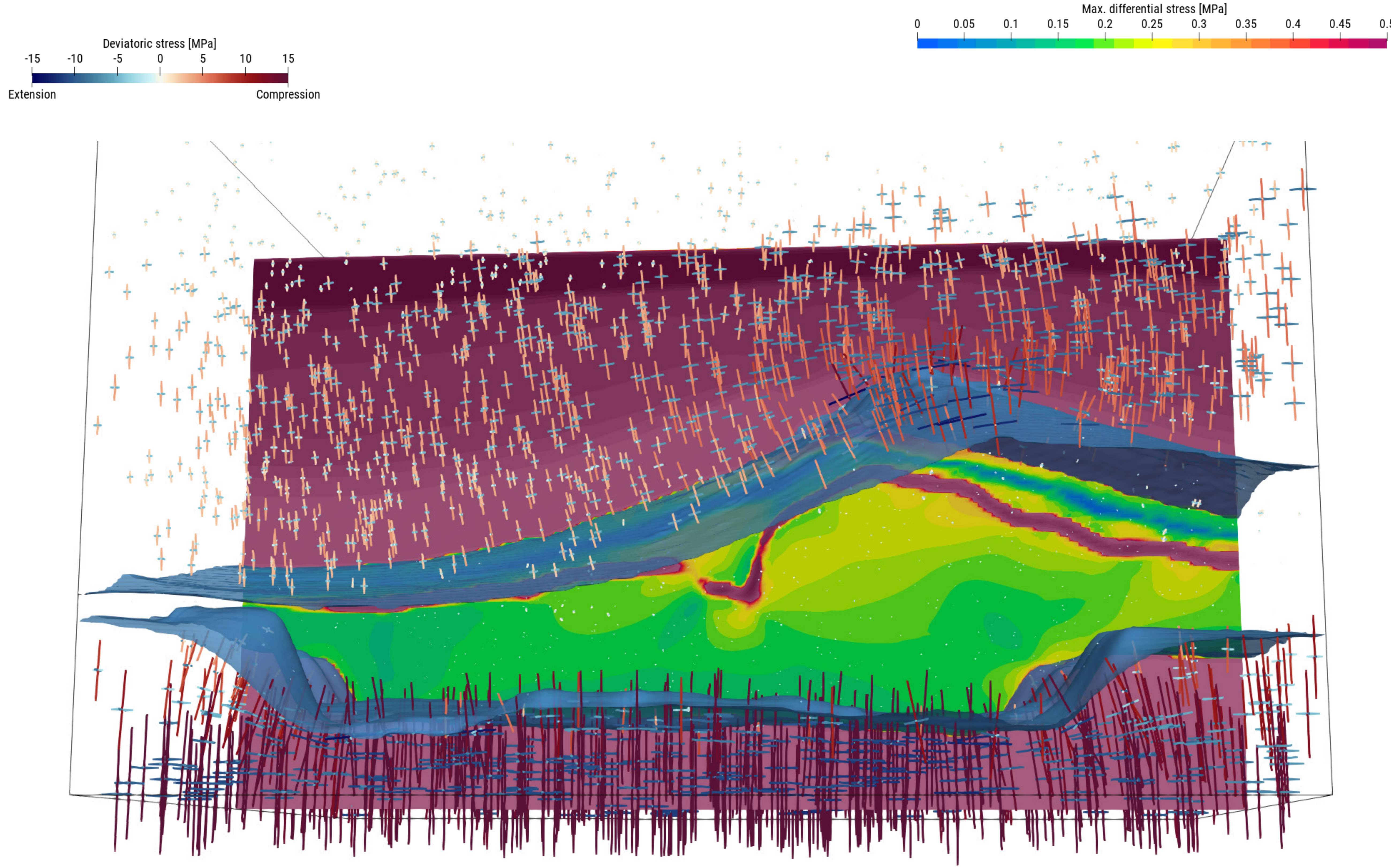
0.5 - 1 MPa bedded salt

0.5 - 2 MPa in domal salt

up to 5 MPa in cold diapir stem or thrusts

Carter, N.L., Horseman, S.T., Russell, J.E., Handin, J., 1993. Rheology of rocksalt. Journal of Structural Geology 15, 1257–1271

virgin stress field of a salt pillow



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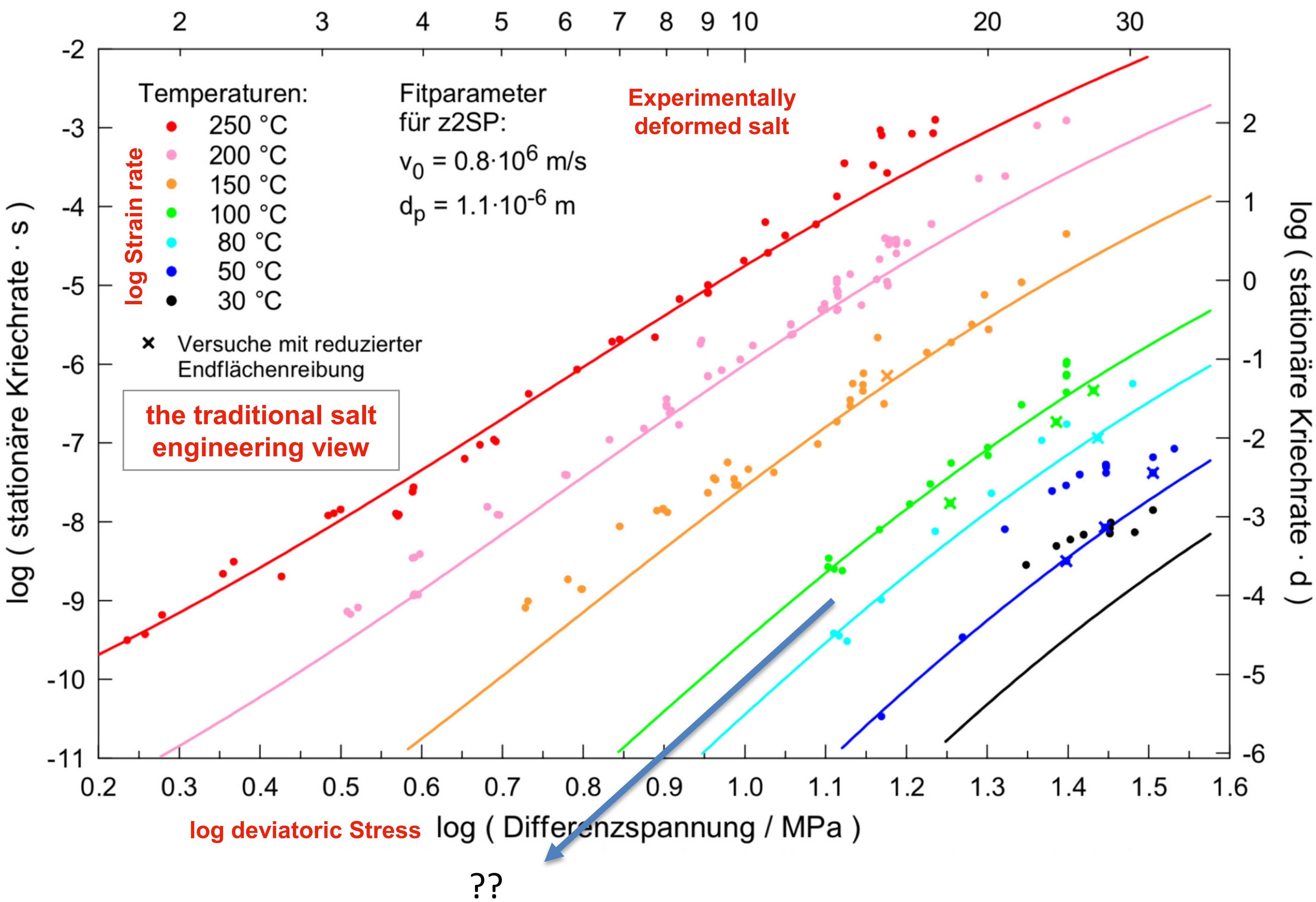
Tobias Baumann, Boris Kaus

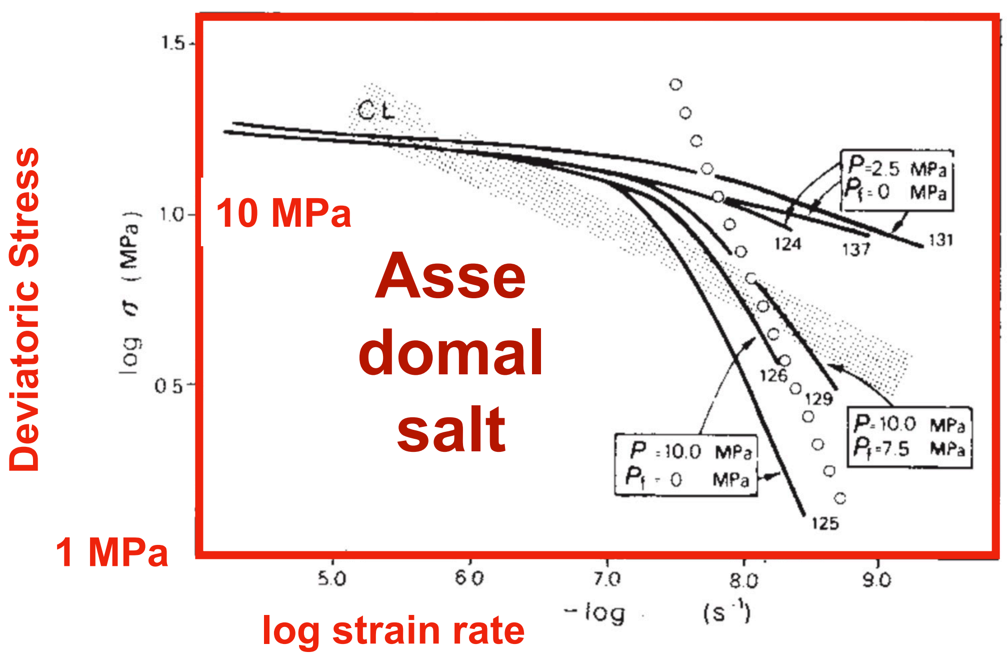
steady state power law creep of rock salt (BGR)

BGR_2003_Projekt-Gorleben_Thermomechanisches-Verhalten-von-Salzgestein Report, BGR, 9G21381100.pdf



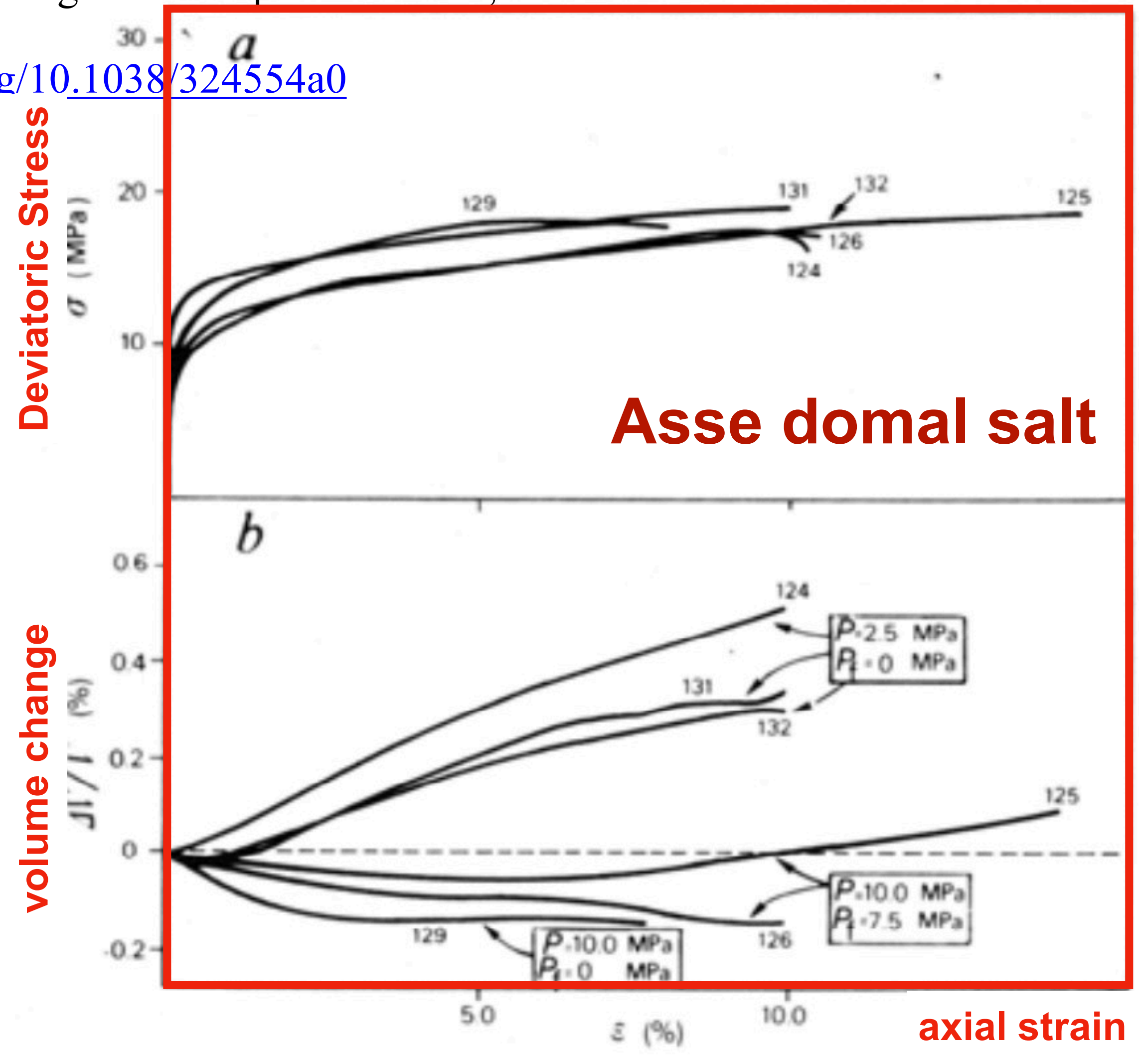
The salt engineering community has built on these early studies, performing a large amount of deformation experiments, with measurements of the stress-strain response, but almost exclusively without microstructural study.

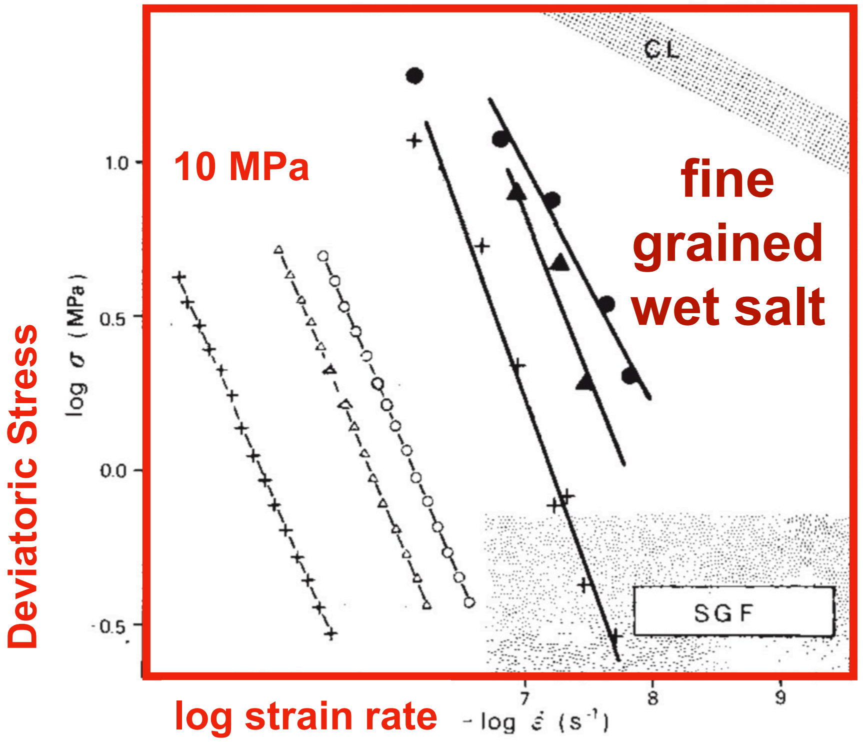
Major weakening during long term creep (1986)



Urai, J.L., Spiers, C.J., Zwart, H.J., Lister, G.S., 1986. Weakening of rock salt by water during long-term creep. Nature 324, 554–557.

https://doi.org/10.1038/324554a0





Major weakening during long term creep, controversial in engineering community

A series of deformation experiments on Asse domal salt with very low water content present as small unconnected brine inclusions at grain boundaries has shown, that in the non-dilatant field, and after rapid deformation to about 10% strain, at lowering the deviatoric stress to values below 10 MPa, these samples defomed many orders of magnitude faster than predicted for dislocation creep. Samples initially deformed in the dilatant field did not show this effect. This dramatic weakening was explained by the formation of thin brine films along the grain boundaries which dramatically increased their mobility, leading to recrystallization and grain refinement by grain boundary migration and the activation of pressure solution creep. The same effect was seen in wet, synthetic, fine grained rock salt samples which deform much faster than predicted by dislocation creep, and this deformation is well explained by pressure solution.

Urai, J.L., Spiers, C.J., 2007. The Effect of Grain Boundary Water on Deformation Mechanisms and Rheology of Rocksalt During Long-Term Deformation. The Mechanical Behavior of Salt – Understanding of THMC Processes in Salt. Hannover, Germany, 149–158.

1986:

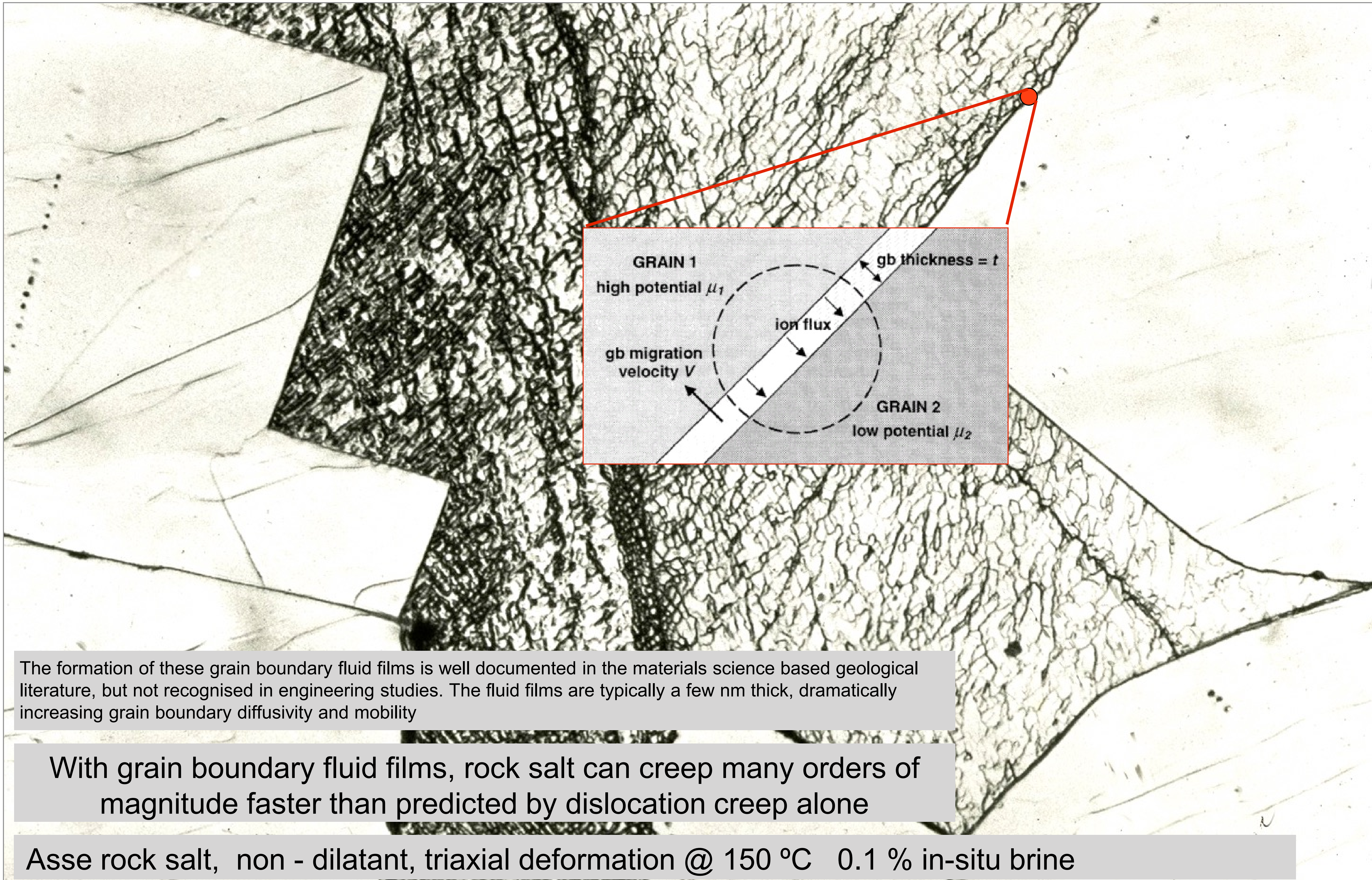
Dynamic recrystallization and pressure solution in Zechstein domal salt from Asse mine

grain boundaries can contain nm-scale thin fluid films which dramatically increase their mobility

this makes the salt flow many orders of magnitude faster than predicted by engineering models

Urai, J.L., Spiers, C.J., Zwart, H.J., Lister, G.S., 1986. Weakening of rock salt by water during long-term creep. Nature 324, 554–557.

Water- assisted Grain Boundary Migration in experiment



The formation of these grain boundary fluid films is well documented in the materials science based geological literature, but not recognised in engineering studies. The fluid films are typically a few nm thick, dramatically increasing grain boundary diffusivity and mobility

With grain boundary fluid films, rock salt can creep many orders of magnitude faster than predicted by dislocation creep alone

Asse rock salt, non - dilatant, triaxial deformation @ 150 ºC 0.1 % in-situ brine

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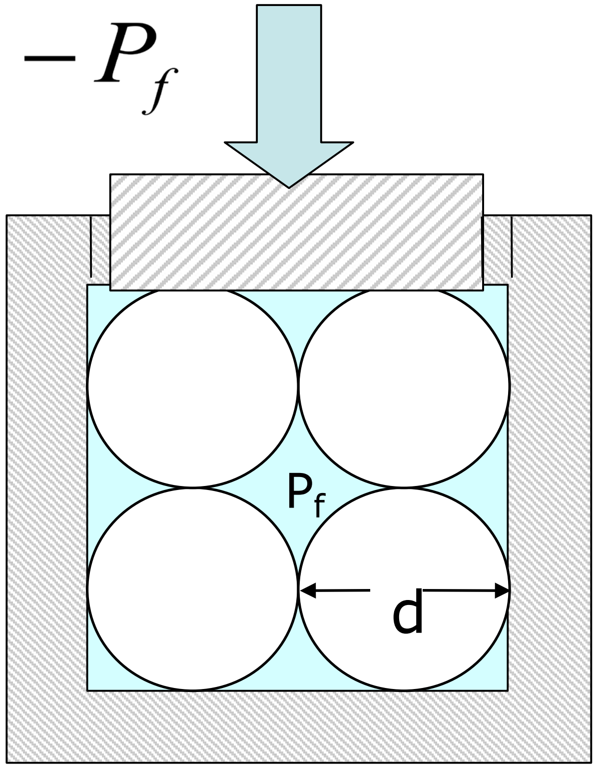
Urai, J.L., Spiers, C.J., Zwart, H.J., Lister, G.S., 1986. Weakening of rock salt by water during long-term creep. Nature 324, 554–557.

Pressure solution in rock salt: Compaction creep

Spiers et al., (1996- 2007)

$$\sigma_e = \sigma - P_f$$



Dissolution Control:

$$\dot{\varepsilon}_s = I_s \times \frac{\sigma_e}{d} \times f_s(\phi)$$

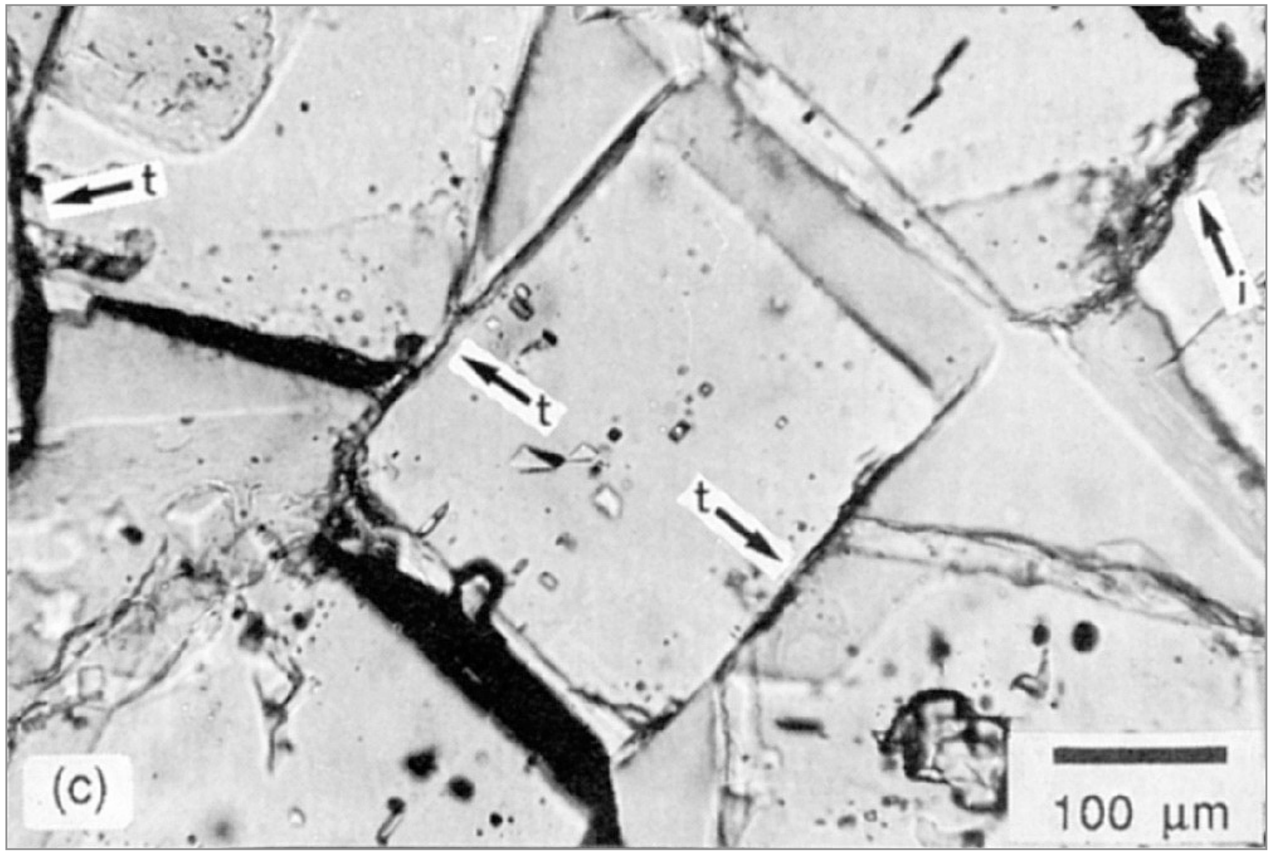
Diffusion Control:

$$\dot{\varepsilon}_d = [DCS] \times \frac{\sigma_e}{d^3} \times f_d(\phi)$$

Precipitation Control:

$$\dot{\varepsilon}_p = I_p \times \frac{\sigma_e}{d} \times f_p(\phi)$$

Pressure solution in wet rock salt has been studied extensively. This paper gives the **constitutive equations** for different steps of the process (dissolution, diffusion or precipitation) being the rate controlling step. These all have different kinetics and grain size sensitivities



Spiers, C.J., Schutjens, P.M.T.M., Brzesowsky, R.H., Peach, C.J., Liezenberg, J.L., Zwart, H.J., 1990. Experimental determination of constitutive parameters governing creep of rocksalt by pressure solution. In: Knipe, R.J., Rutter, E.H. (Eds.), Deformation Mechanisms, Rheology and Tectonics, Geological Society Special Publications.

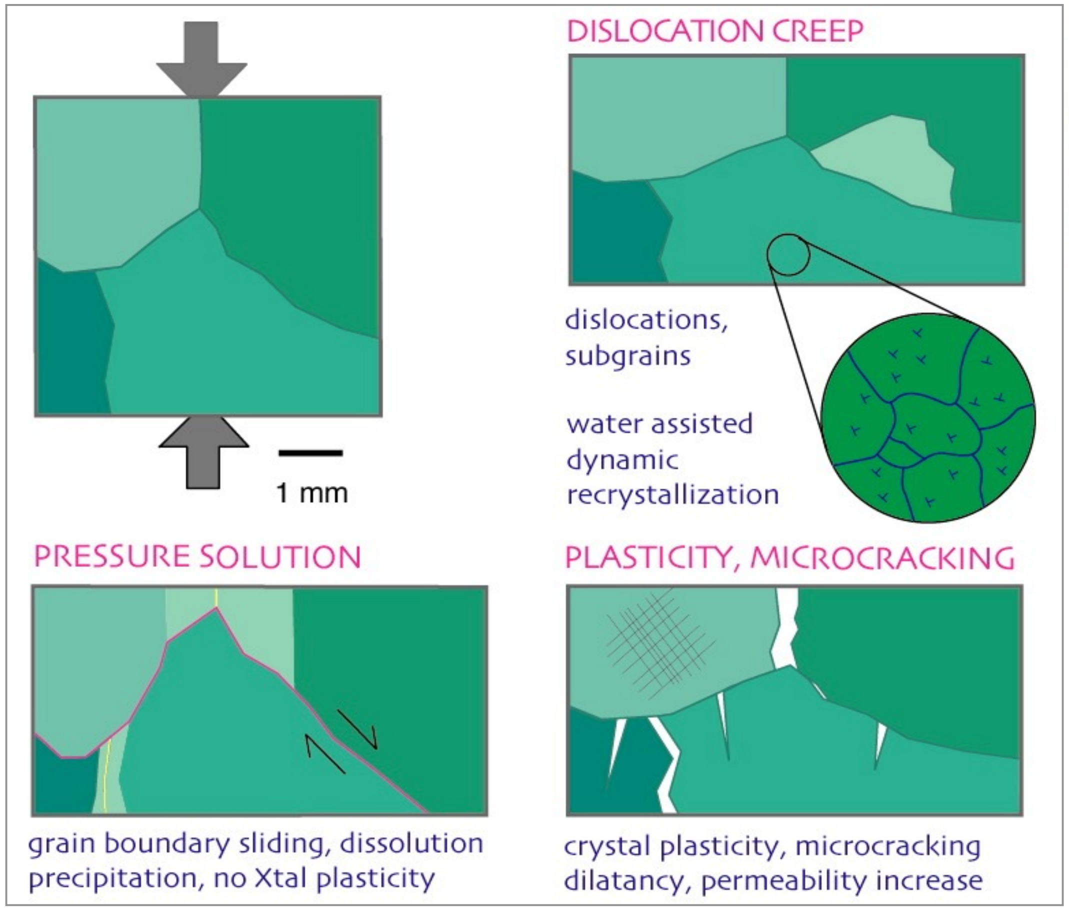
This is a summary of the main microphysical deformation mechanisms in rock salt. They can all operate simultaneously depending on micro fabric and physical conditions, and they are associated with very different mechanical and transport properties

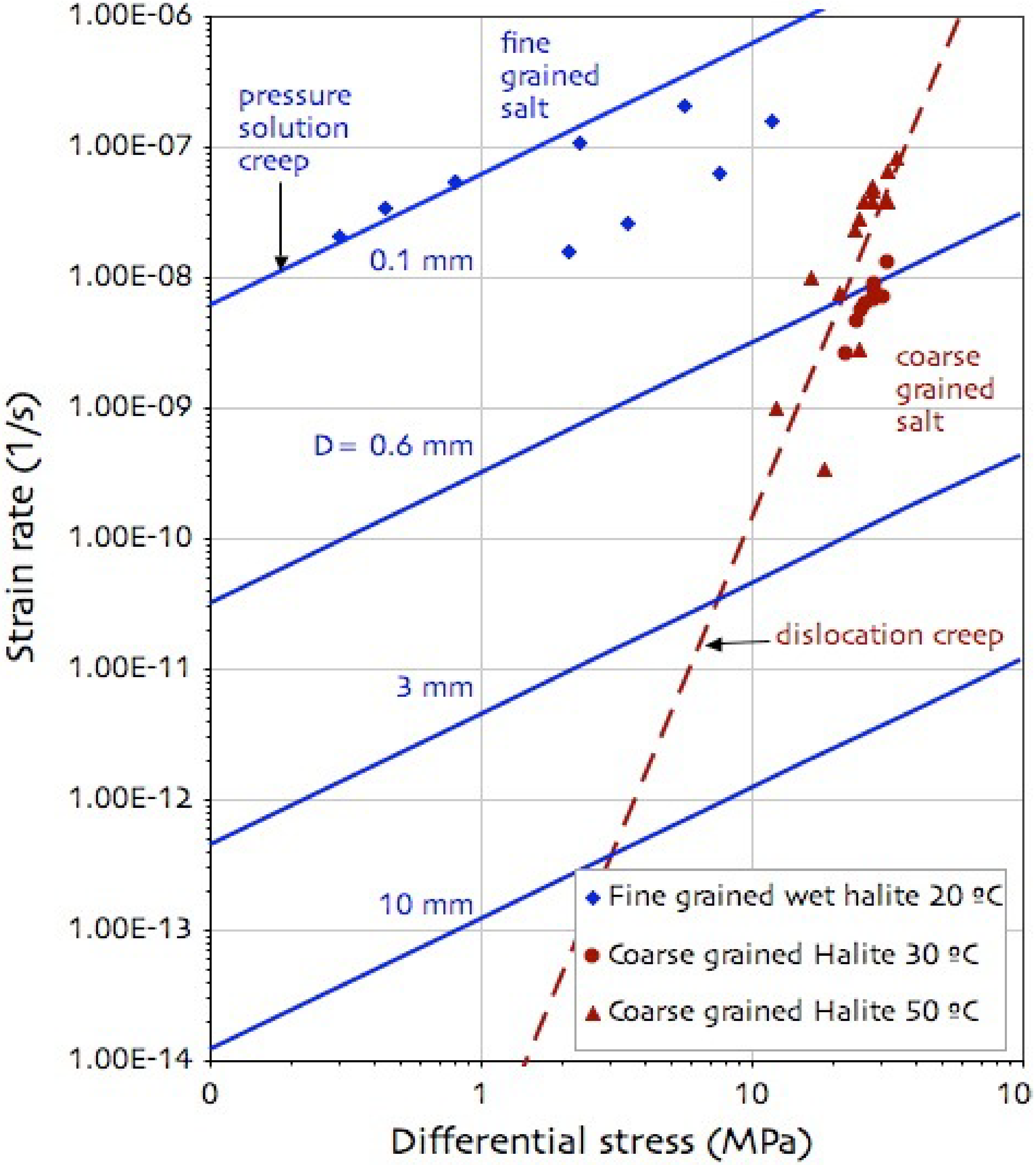
$$\dot{\varepsilon} = A(\Delta\sigma)^n = A_0 \exp\left(-\frac{Q}{RT}\right)(\sigma_1 - \sigma_3)^n$$

dislocation creep

$$\dot{\varepsilon} = B(\Delta\sigma^1) = B_0 \exp\left(-\frac{Q}{RT}\right)\left(\frac{(\sigma_1 - \sigma_3)^1}{TD^m}\right)$$

pressure solution creep

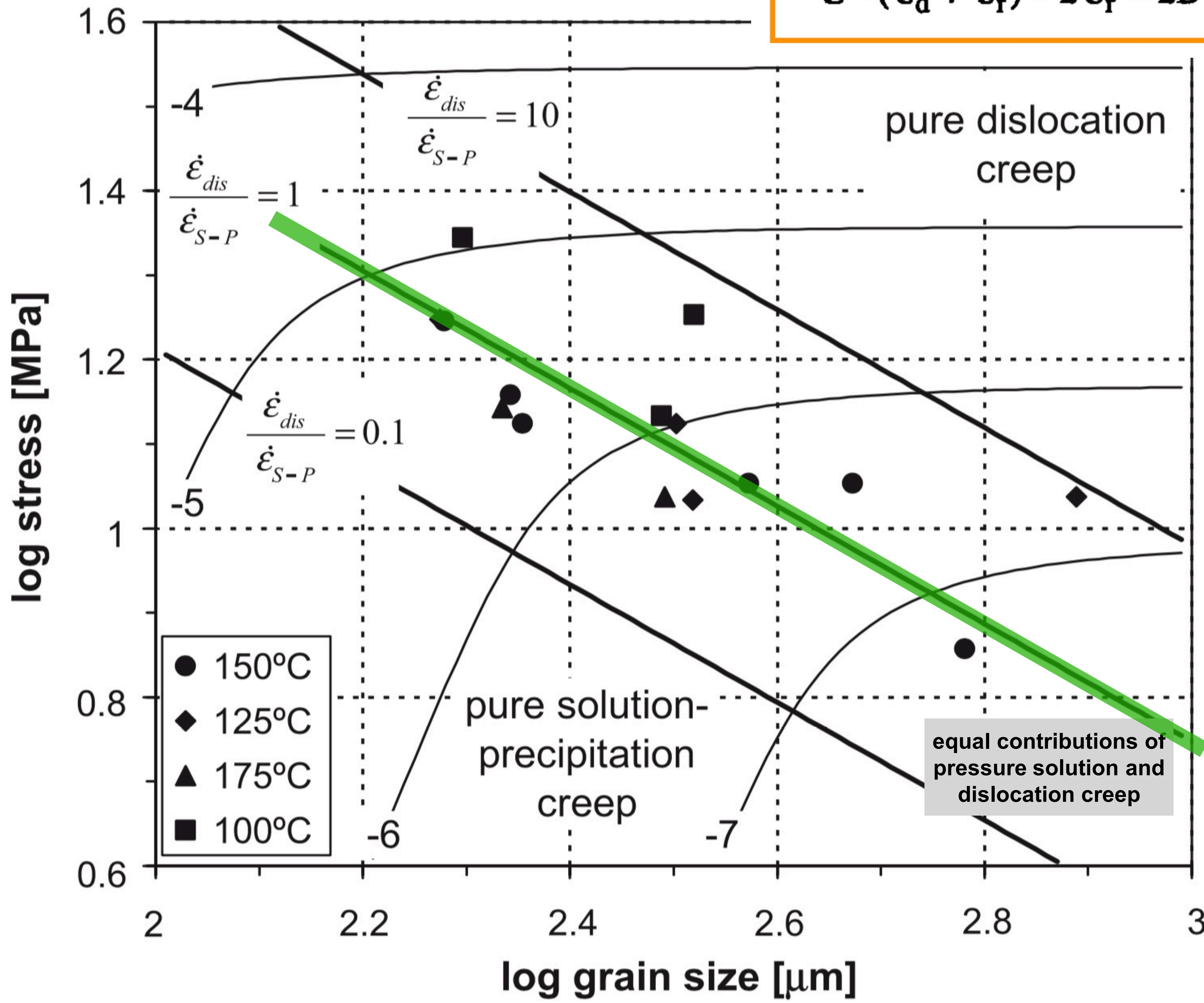




Urai, J.L., Schleder, Z., Spiers, C.J., Kukla, P.A., 2008. Flow and Transport Properties of Salt Rocks. Dynamics of Complex Intracontinental Basins: The Central European Basin System. Springer, 277–290.

field boundary model for deforming wet rock salt

$$\dot{\varepsilon} = (\dot{\varepsilon}_d + \dot{\varepsilon}_r) \approx 2\,\dot{\varepsilon}_r = 2B\,\sigma^n \exp(-Q_r/RT)$$



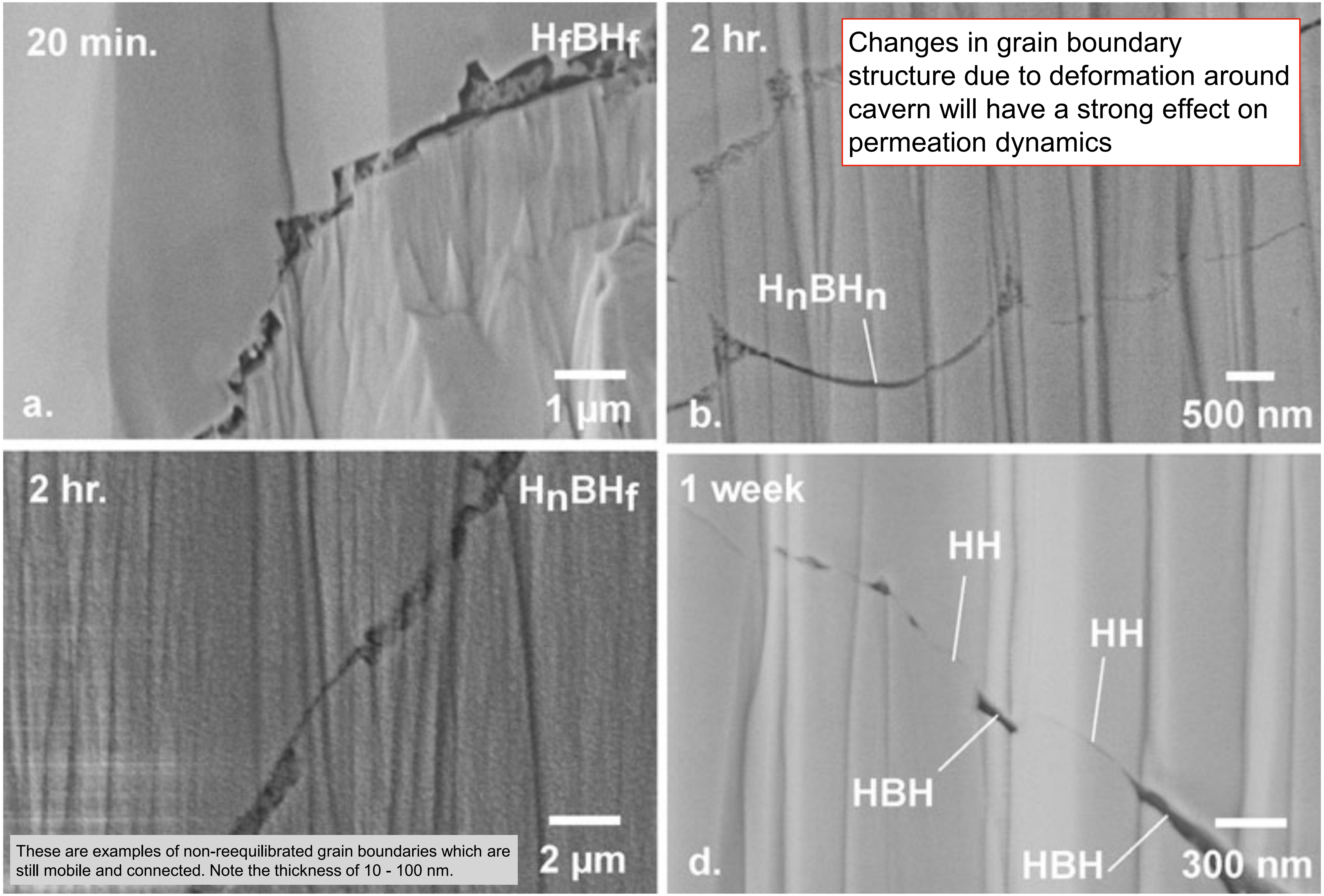
Dedicated studies of the effect of small amounts of water in rock salt (> 10 ppm, which is always present in nature) have shown a clear difference with artificially dried samples which creep about three orders of magnitude slower than wet samples under the same conditions.

There is extensive dynamic recrystallisation and the **samples deform by equal contributions of dislocation and pressure solution creep**. This is the basis of the well-known microphysical field boundary model.

The dynamically **recrystallized grainsize is a function of deviatoric stress:** the lower the deviatoric stress, the higher the dynamically recrystallized grainsize.

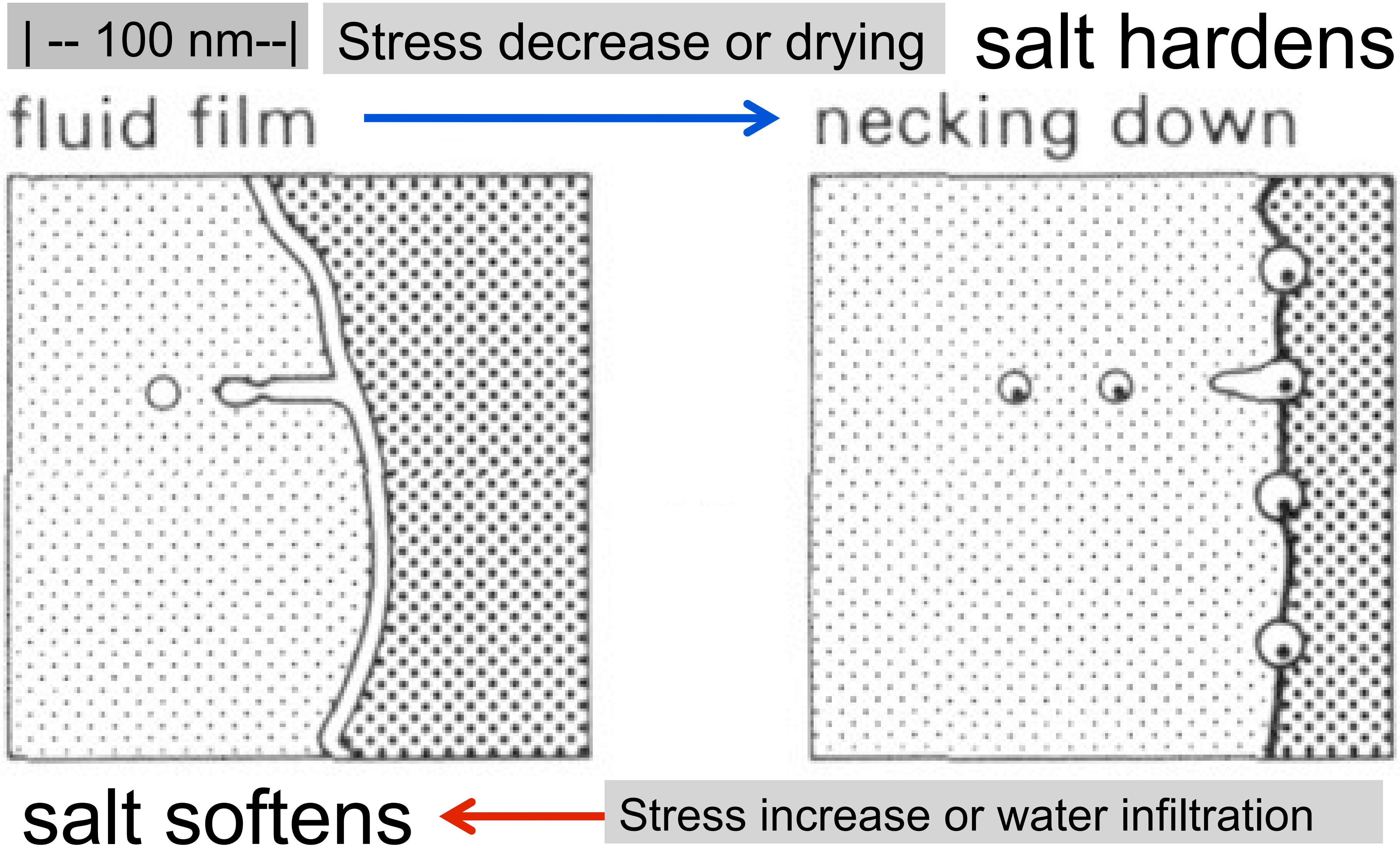
de Bresser, J.H.P., Ter Heege, J.H., Spiers, C.J., 2001. Grain size reduction by dynamic recrystallization: can it result in major rheological

Cryo-BIB-SEM of wet salt grain boundaries



Desbois, G., Urai, J.L., Kukla, P.A., Wollenberg, U., Pérez-Willard, F., Radí, Z., Riholm, S., 2012. Distribution of brine in grain boundaries during static recrystallization in wet, synthetic Halite: Insight from broad ion beam sectioning and SEM observation at cryogenic temperature. Contributions to Mineralogy and Petrology 163, 19–31.

The healing of of grain boundaries

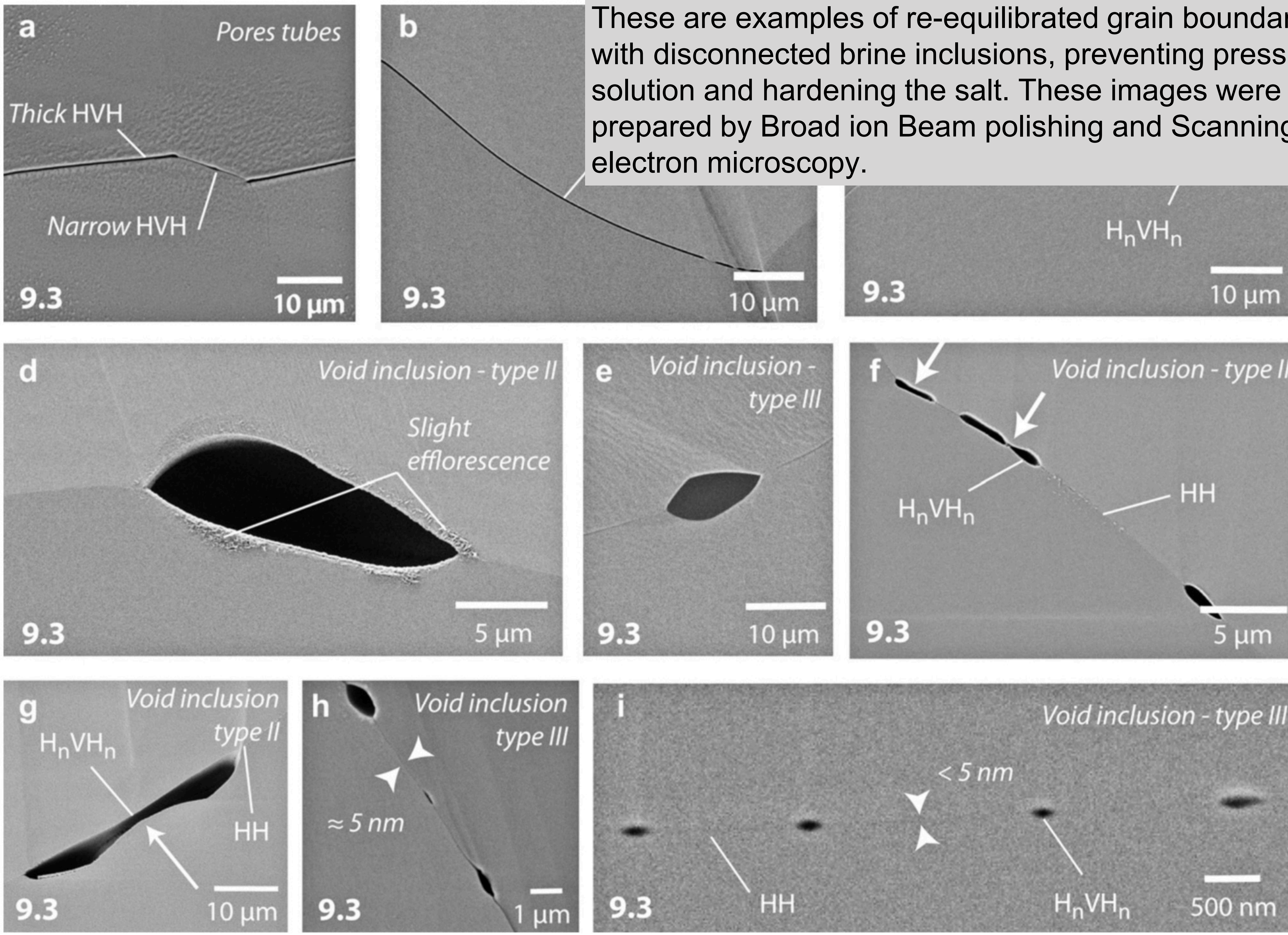


One interesting and important property of grain boundary fluid films in rock salt is that they are only stable when the deviatoric stress is sufficiently high. If the rock salt is recrystallized and deviatoric stress decreases, the films re-equilibrate into disconnected fluid inclusions, potentially stopping pressure solution and hardening the salt. this process is reversible

Drury, M.R., Urai, J.L., 1990. Deformation-related recrystallization processes. Tectonophysics 172, 235–253.

grain boundary fluid distribution in natural rock salt (BIB-SEM)

These are examples of re-equilibrated grain boundaries with disconnected brine inclusions, preventing pressure solution and hardening the salt. These images were prepared by Broad ion Beam polishing and Scanning electron microscopy.



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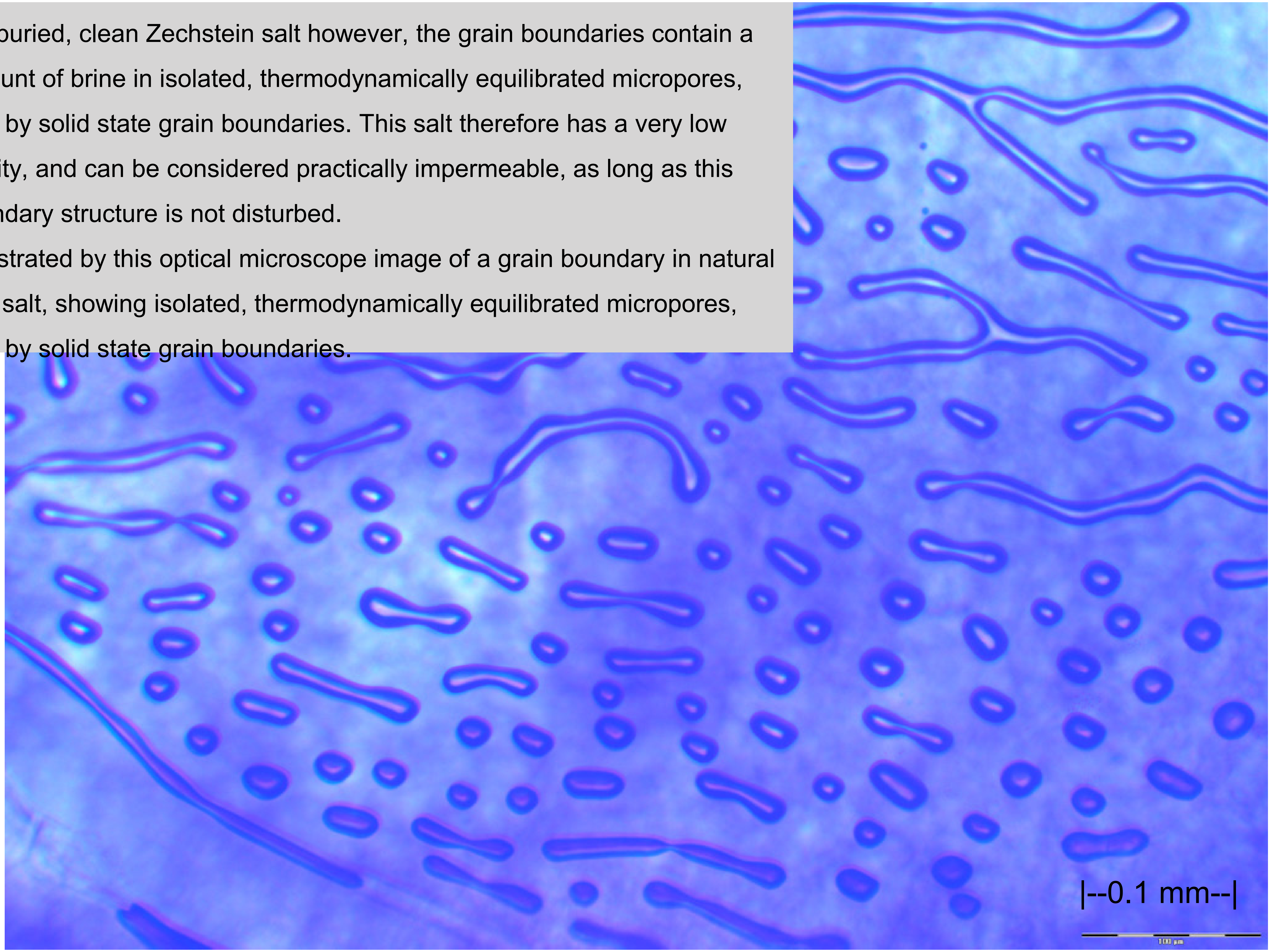
Desbois, G., Urai, J.L., de Bresser, J.H.P., 2012. Fluid distribution in grain boundaries of natural fine-grained rock salt deformed at low differential stress (Qom Kuh Salt Fountain, Central Iran): Implications for rheology and transport properties. Journal of Structural Geology 43, 128–143.

Grain Boundary fluid inclusions in Rock salt

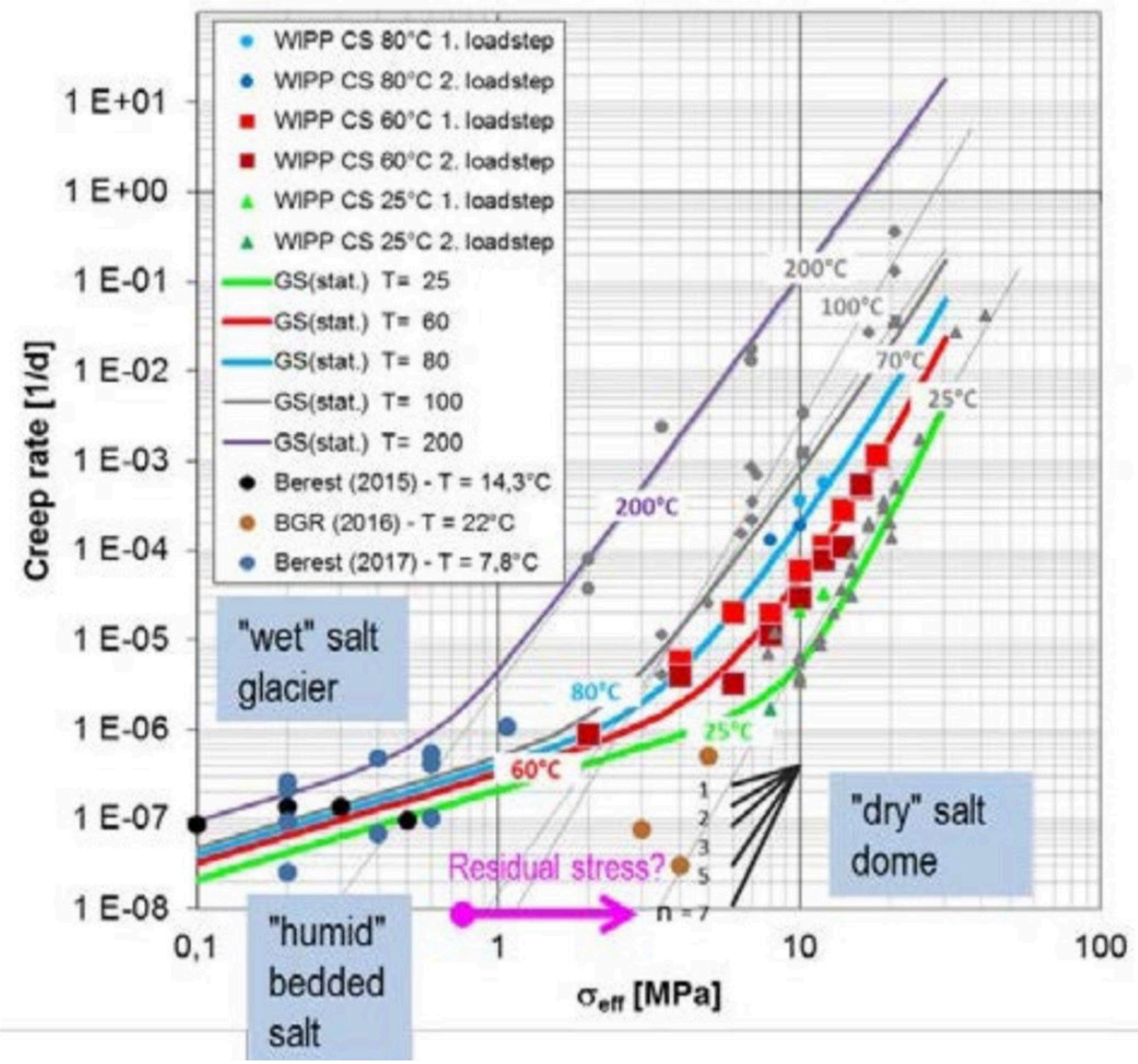
In deeply buried, clean Zechstein salt however, the grain boundaries contain a small amount of brine in isolated, thermodynamically equilibrated micropores, separated by solid state grain boundaries. This salt therefore has a very low permeability, and can be considered practically impermeable, as long as this grain boundary structure is not disturbed.

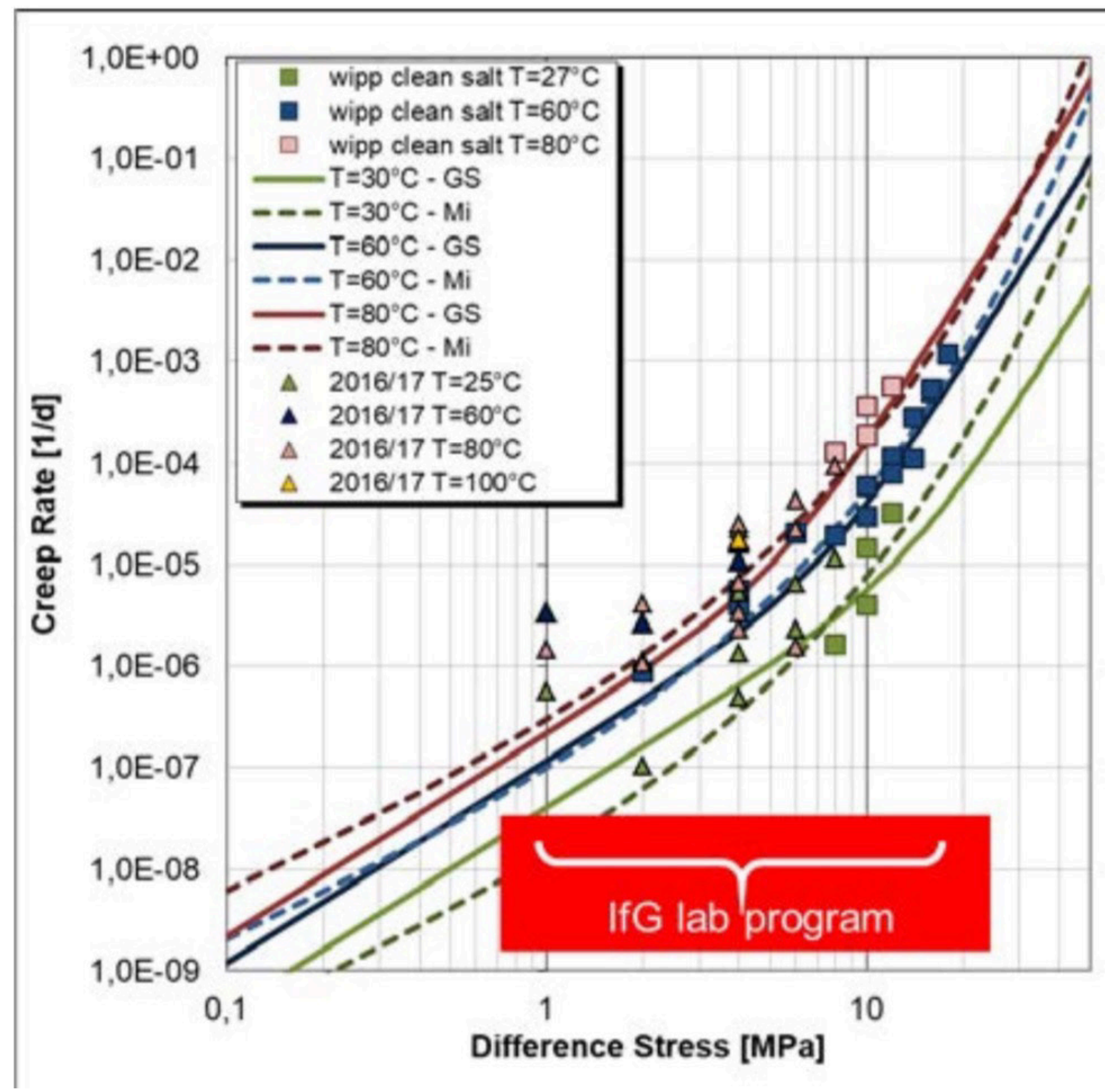
This is illustrated by this optical microscope image of a grain boundary in natural Zechstein salt, showing isolated, thermodynamically equilibrated micropores, separated by solid state grain boundaries.



State of the art, engineering flow laws for rock salt

“Professor Pierre Bérest, who along with a group of colleagues from the Solution Mining Research Institute, has executed some **clever low-deviatoric creep tests** with control of temperature and stress to tight specifications (SMRI Research Report RR2017-1, available at SMRI) The data base obtained from in situ observations and laboratory tests on salt indicates that both dislocation (e.g., Carter & Hansen, 1983) and diffusional creep mechanisms (Urai & Spiers, 2007; Spiers et al., 1990) can be important in salt under long-term conditions.”

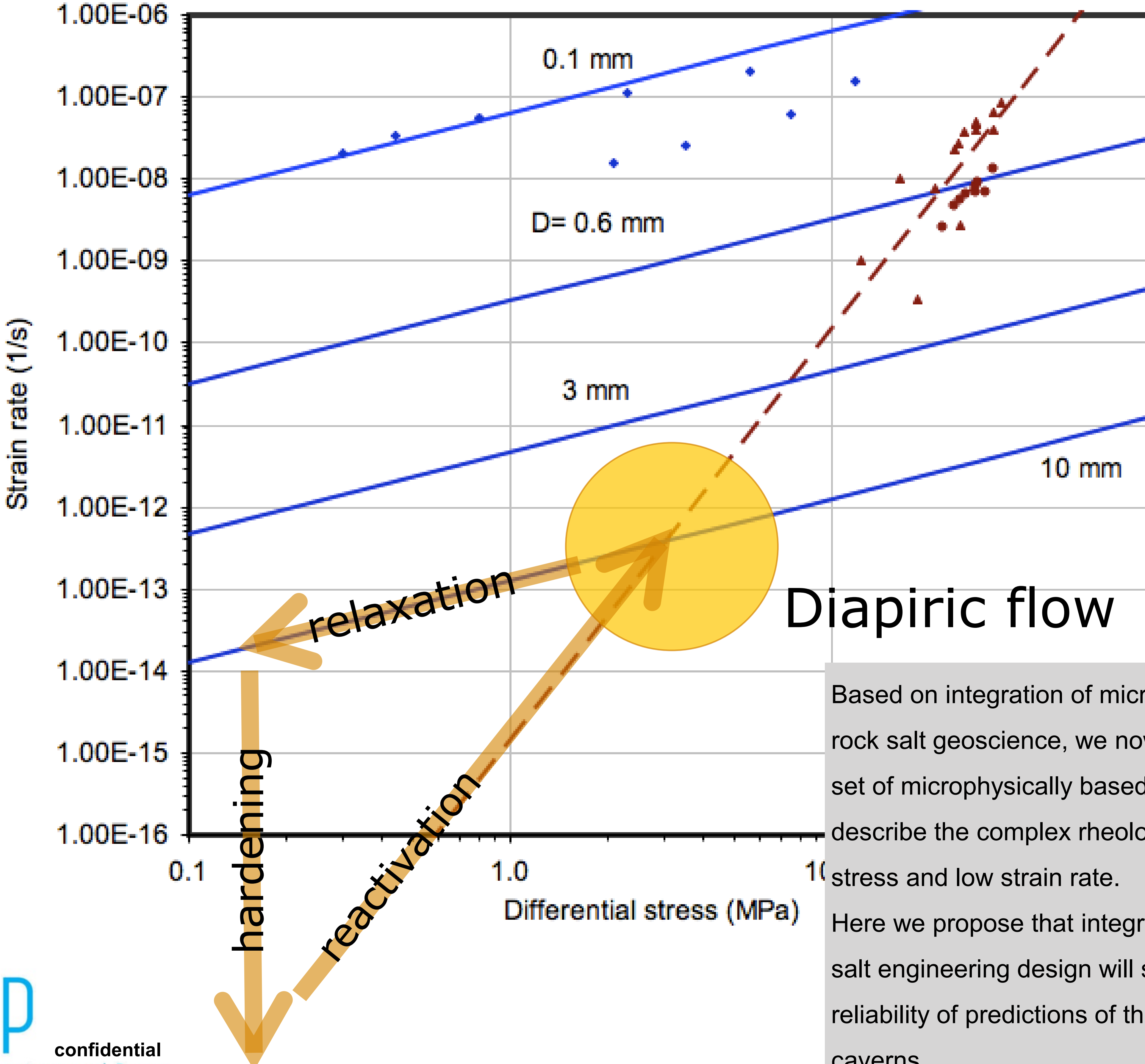




MAP Microstructure and Pores confidential

Bérest, 2018: CREEP AT LOW DEVIATORIC STRESS. In: Proceedings of the 8th US/German Workshop on Salt Repository Research, Design, and Operation - Spent Fuel and Waste Disposition - Prepared for US Department of Energy - Spent Fuel and Waste Science and Technology - Francis D. Hansen, RESPEC, Walter Steininger, Project Management Agency Karlsruhe, Wilhelm Bollingerfehr, DBE TECHNOLOGY GmbH, Kristopher Kuhlman, Sean Dunagan, Sandia National Laboratories - February 7, 2018 - SFWD-SFWST-2018-000485

Cyclic Halite rheology



Li, S., Abe, S., Urai, J.L., Strozyk, F., Kukla, P.A., van Gent, H.W., 2012. A method to evaluate long-term rheology of Zechstein salt in the Tertiary. SaltMech7 - The Mechanical Behaviour of Salt VII. Taylor & Francis Group, Paris, France, 215–220.

10^{19} Pa s

Based on integration of microphysics and rock mechanics in rock salt geoscience, we now have a reasonably complete set of microphysically based constitutive models which describe the complex rheology of rock salt at low deviatoric stress and low strain rate.

Here we propose that integrating this understanding in the salt engineering design will significantly increase the reliability of predictions of the evolution of abandoned caverns.

Microstructure - based salt property determination

Materials science

Microscale attributes (state variables)

Dislocations: distribution, density

Microcracks: density, connectivity

Grain boundaries: structure, porosity, fluid content

Grain size, grain shape

Subgrain size, subgrain distribution

Solid solution impurities

Second phase impurities, fraction, distribution

Pore fluid, pore pressure

30 Parameters

⟺

Salt rock engineering

Macroscale attributes (constitutive equations)

Elastic properties

Tensile failure

Brittle Triaxial

Primary creep

Steady state creep

Dilatancy

Permeation, Permeability

30 Parameters

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connection - microphysics and machine learning

permeation in an abandoned salt cavern

the conventional view

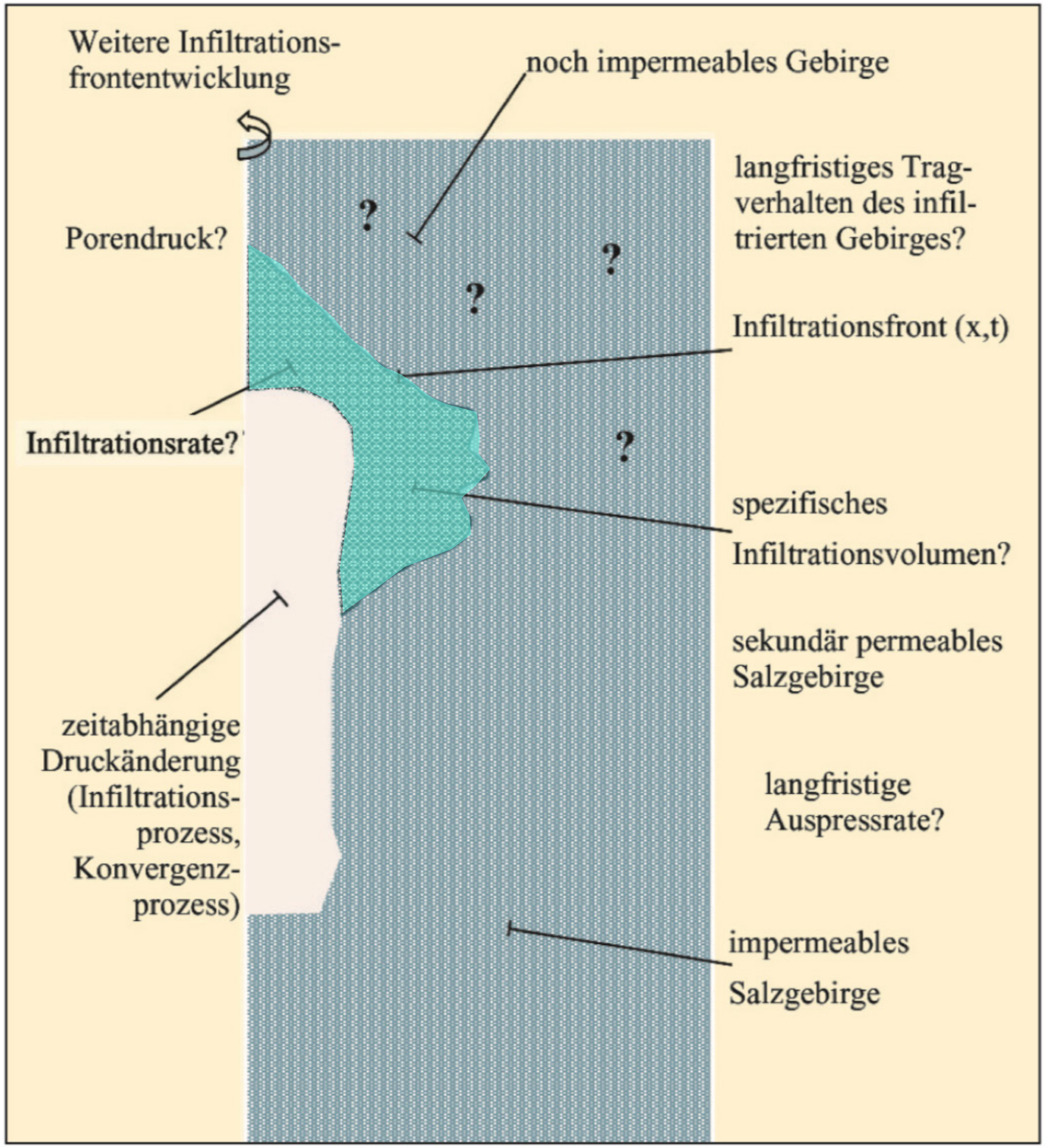


Abb. 2 Einige Fragestellungen zum Langzeitverhalten einer verschlossenen fluidgefüllten Salzkaverne

the view of this report

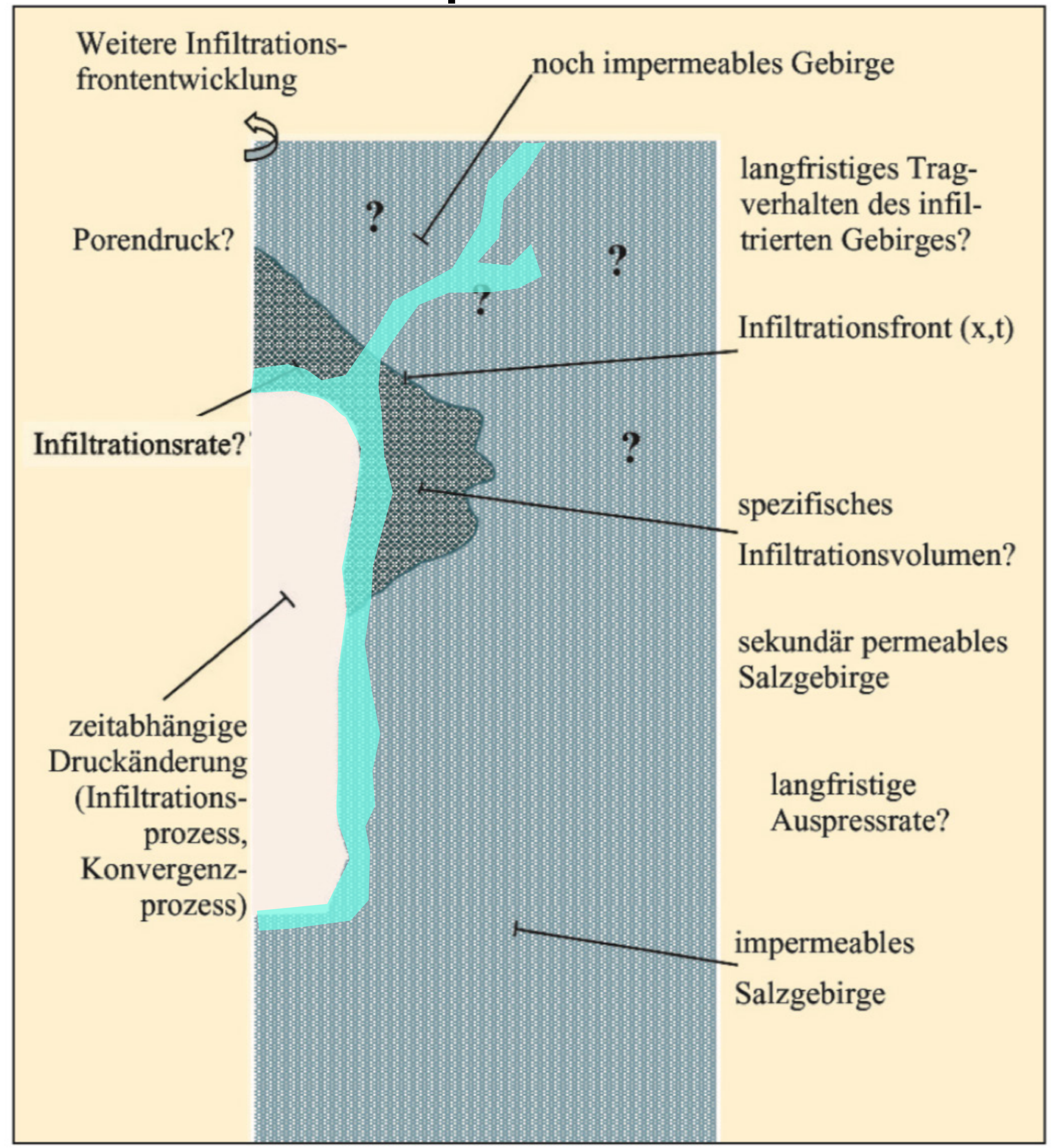
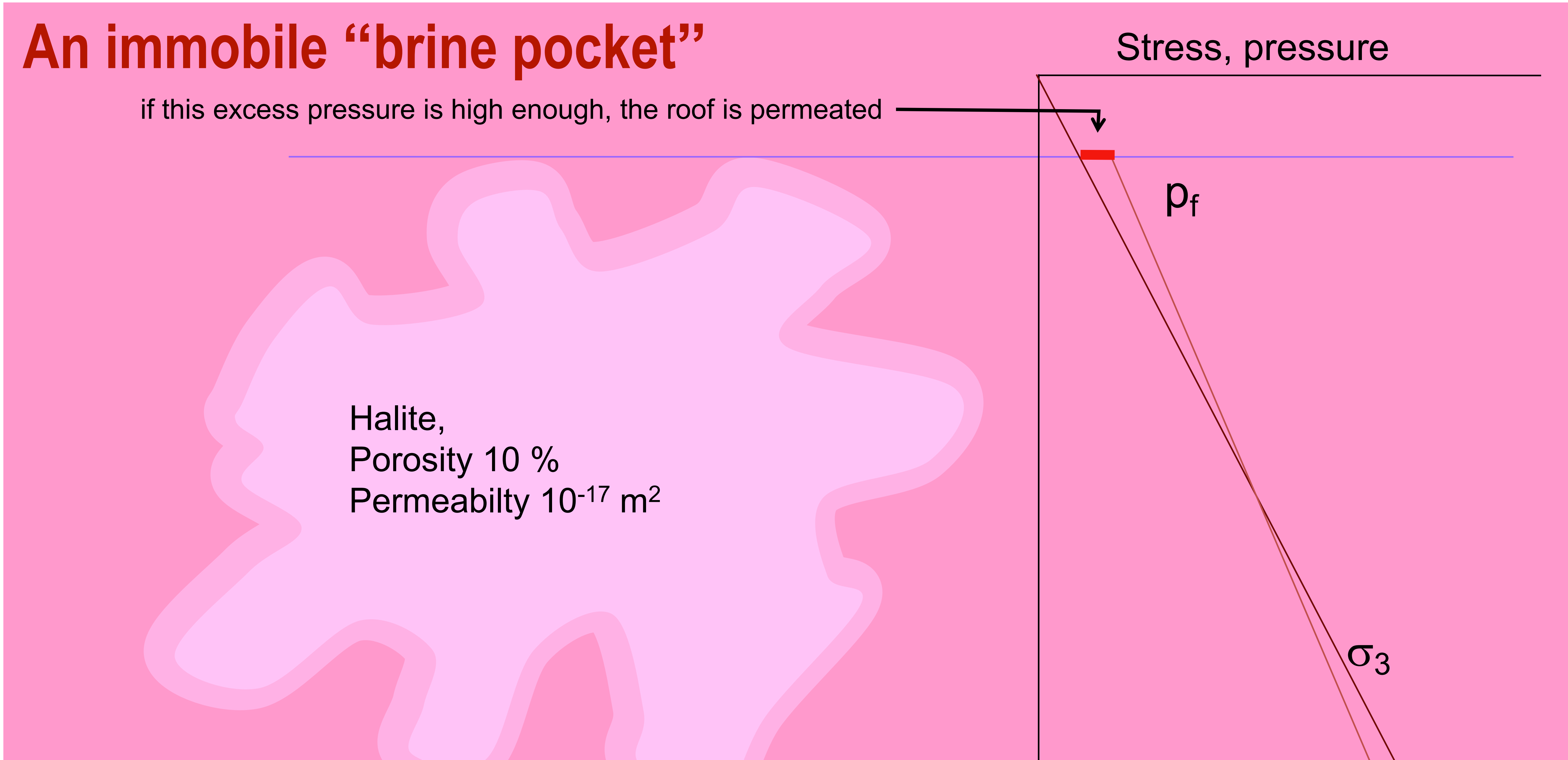


Abb. 2 Einige Fragestellungen zum Langzeitverhalten einer verschlossenen fluidgefüllten Salzkaverne

Lux, K.-H., 2006. Zum langfristigen Tragverhalten von verschlossenen solegefüllten Salzkavernen – ein neuer Ansatz zu physikalischer Modellierung und numerischer Simulation: Rechnerische Analysen und grundlegende Erkenntnisse [Long-term behaviour of sealed liquid-filled salt cavities – a new approach for physical modelling

An immobile “brine pocket”

An immobile “brine pocket”



Halite,
Porosity 0.01 %
Permeabilty 10^{-21} m^2

Potential for upward movement depends on vertical dimension

In drilling Zechstein salt, “brine pockets” can pose major problems. These are thought to consist of slightly porous and permeable volumes of rock salt, containing brine at close to lithostatic pressure. However, when the vertical dimension of these pockets is small enough, the pockets can be geologically stable, even though the pressure at the top is slightly larger than the minimum principal stress in the salt.

These brine pockets are natural analogues of brine migrating upwards from abandoned caverns.

NB: In principle, in the presence of a thermal gradient, convection can take place leading to dissolution at the warmer face of the pocket

MAP
Microstructure and Pores

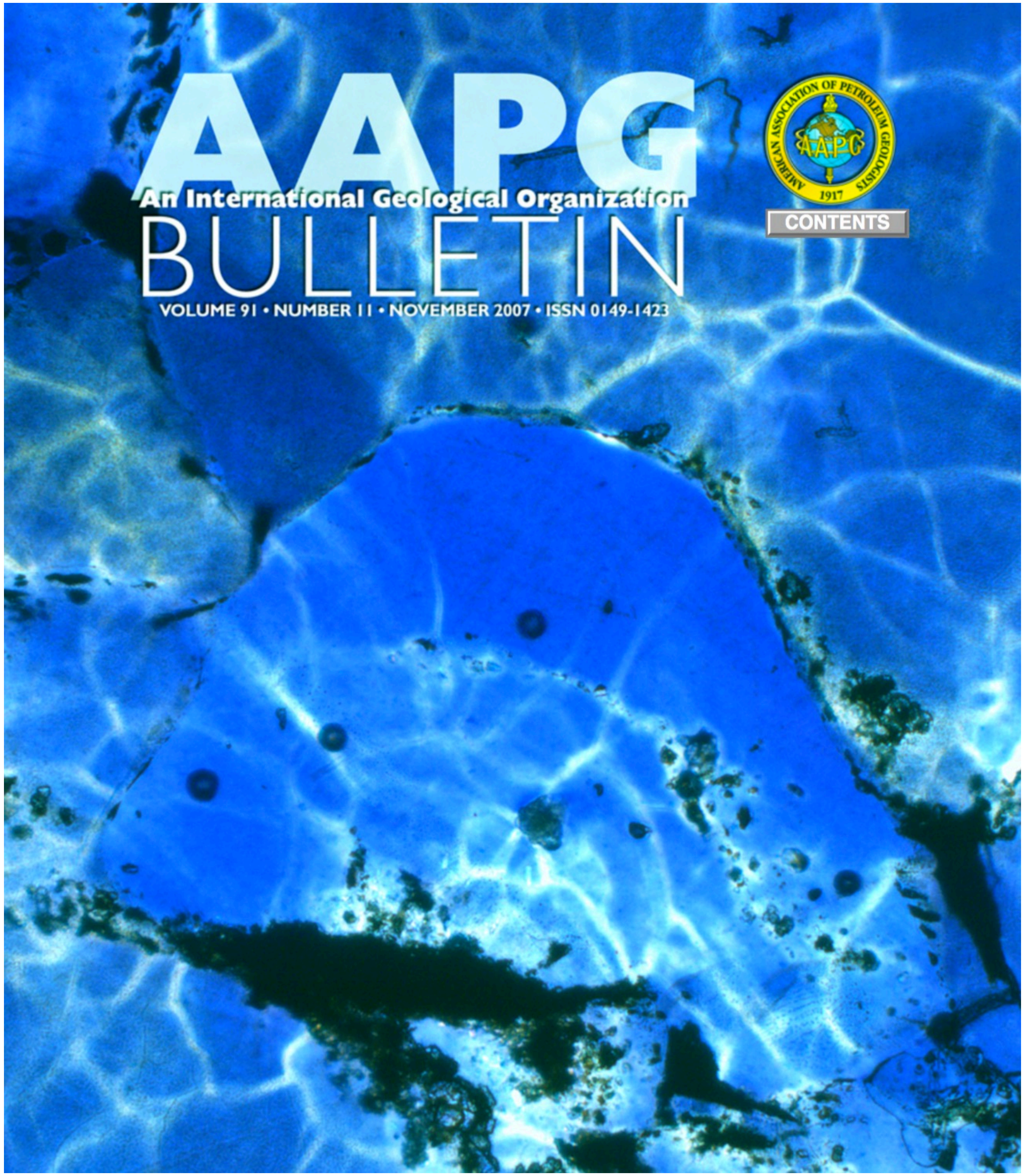
a natural example of infiltration

Limits to the sealing capacity of rock salt: A case study of the infra-Cambrian Ara Salt from the South Oman salt basin

Johannes Schoenherr, Janos L. Urai, Peter A. Kukla, Ralf Littke, Zsolt Schléder, Jean-Michel Larroque, Mark J. Newall, Nadia Al-Abry, Hisham A. Al-Siyabi, and Zuwena Rawahi

The study concludes that when fluid pressure (in this case oil) exceeds the minimum principal stress, grain boundaries in rock salt dilate and the rock salt becomes permeable.

This is a natural example of permeation, which is this case is visible by the oil films along grain boundaries.



Schoenherr, J., Urai, J.L., Kukla, P.A., Littke, R., Schleder, Z., Larroque, J.-M., Newall, M.J., Al-Abry, N., Al-Siyabi, H.A., Rawahi, Z., 2007. Limits to the sealing capacity of rock salt: A case study of the infra-Cambrian ara salt from the south Oman salt basin. AAPG Bulletin 91, 1541–1557.

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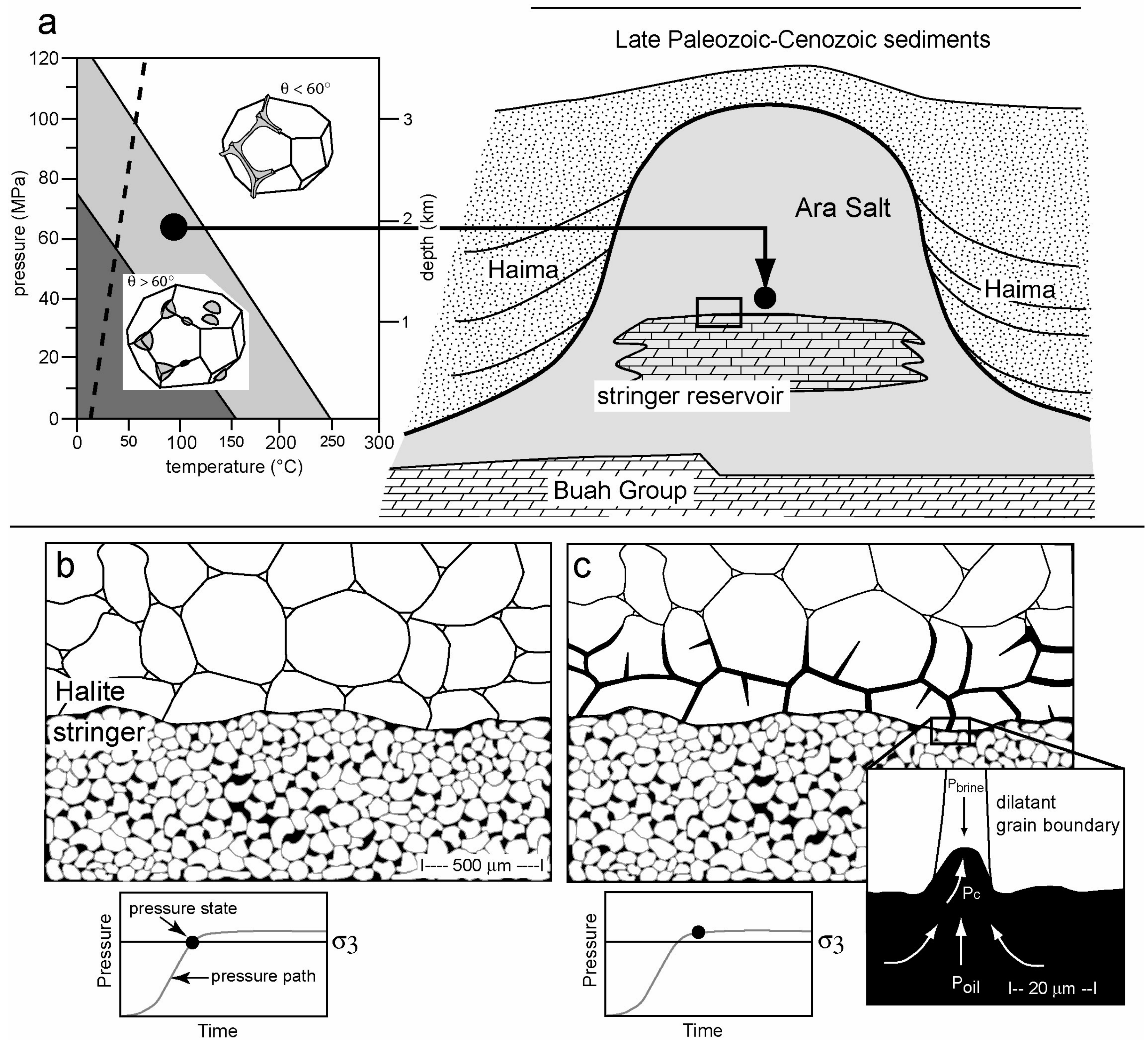

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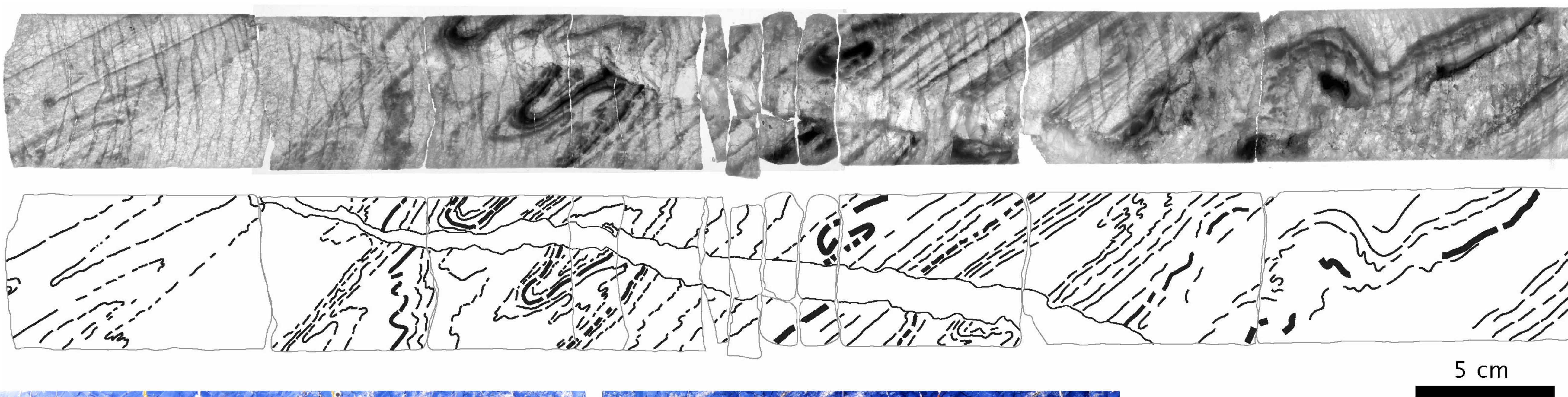
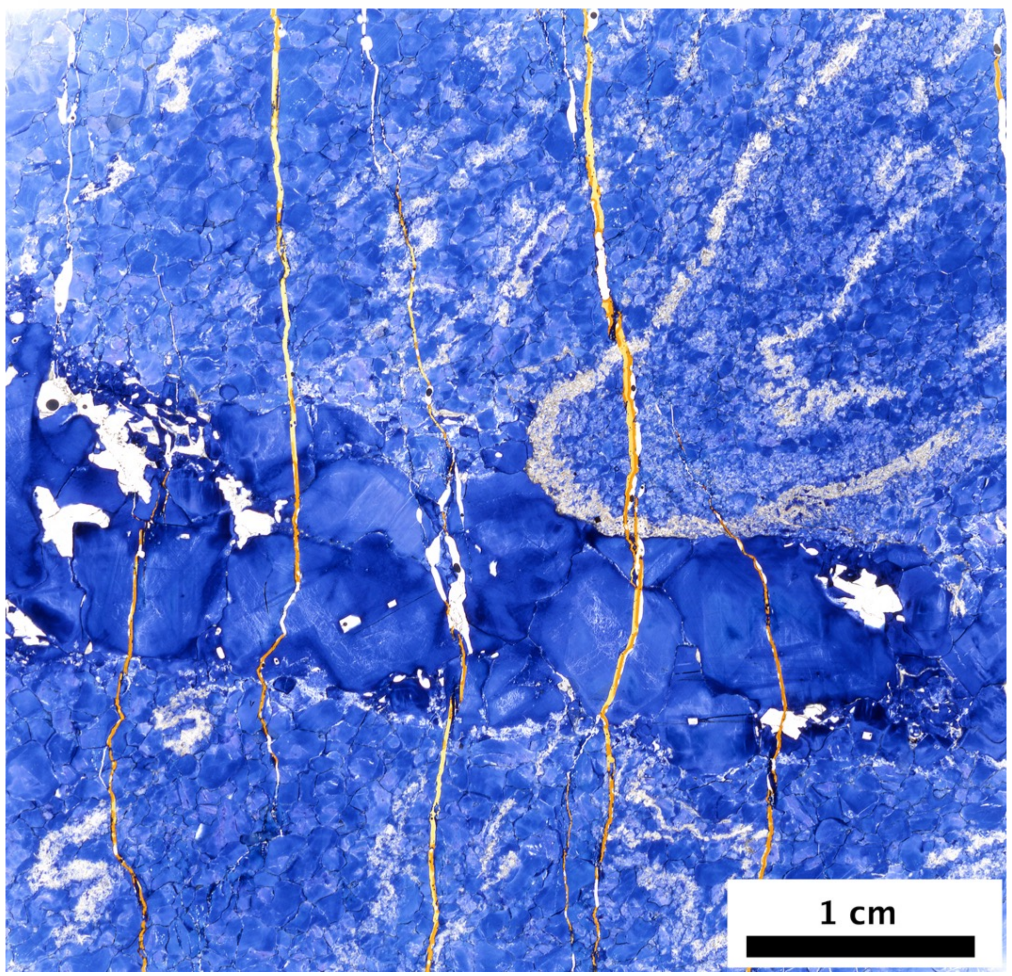
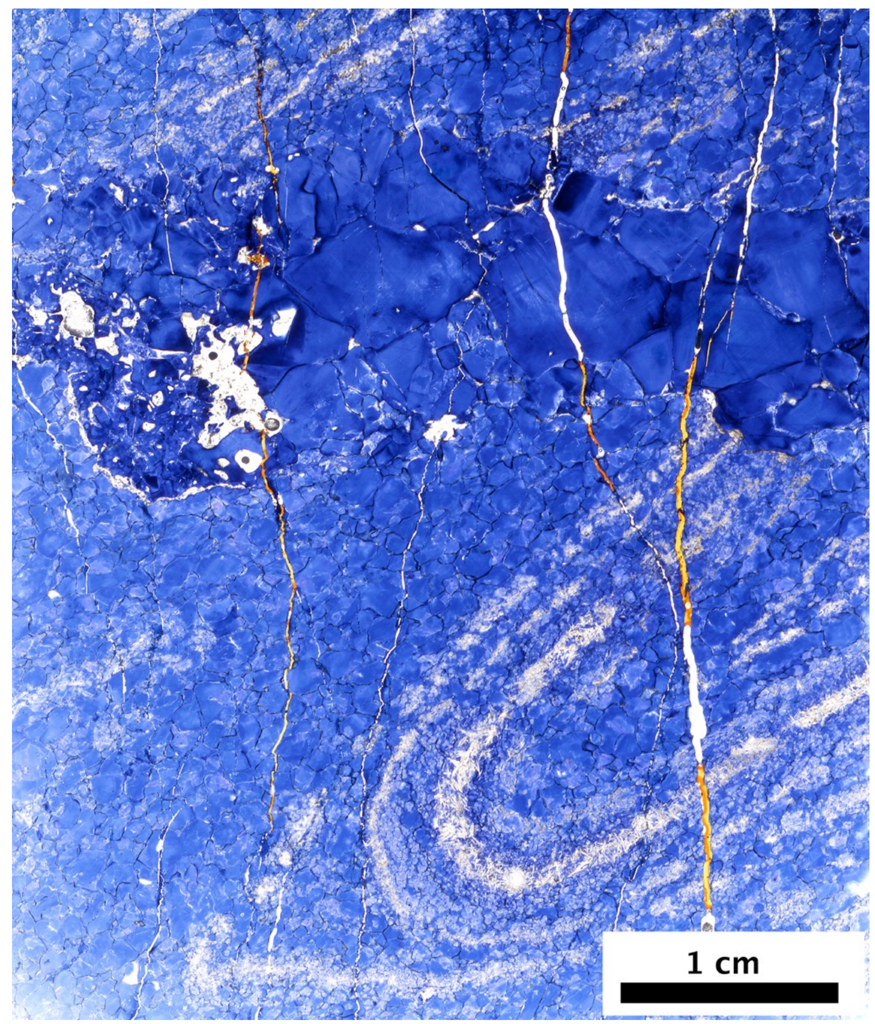
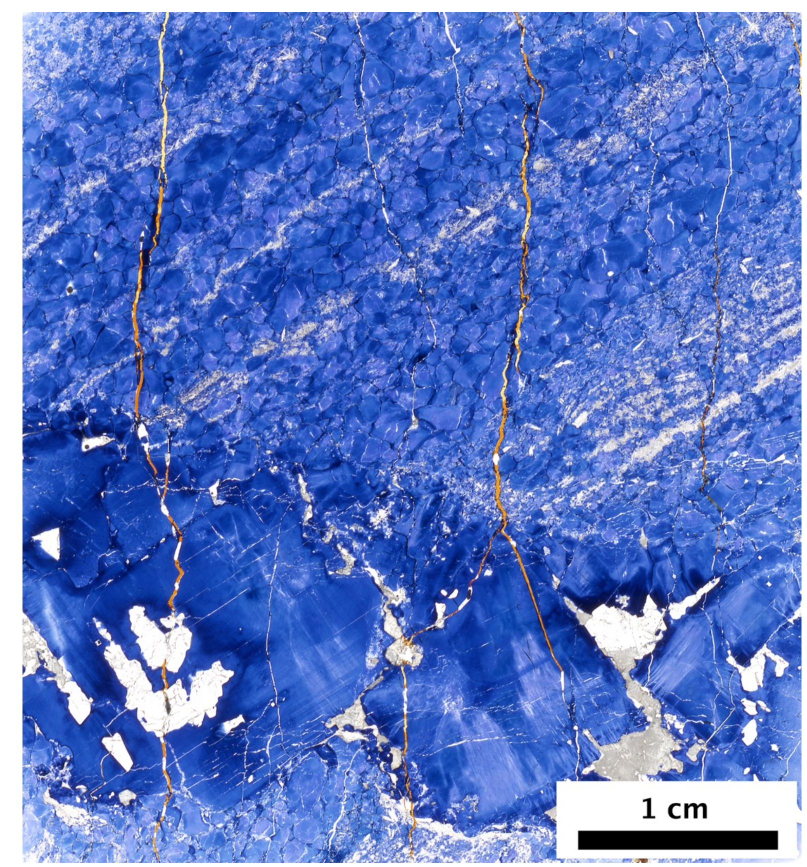
Schoenherr, J., Urai, J.L., Kukla, P.A., Littke, R., Schleder, Z., Larroque, J.-M., Newall, M.J., Al-Abry, N., Al-Siyabi, H.A., Rawahi, Z., 2007. Limits to the sealing capacity of rock salt: A case study of the infra-Cambrian ara salt from the south Oman salt basin. AAPG Bulletin 91, 1541–1557.

The folded halite layers contain sealed hydrofractures







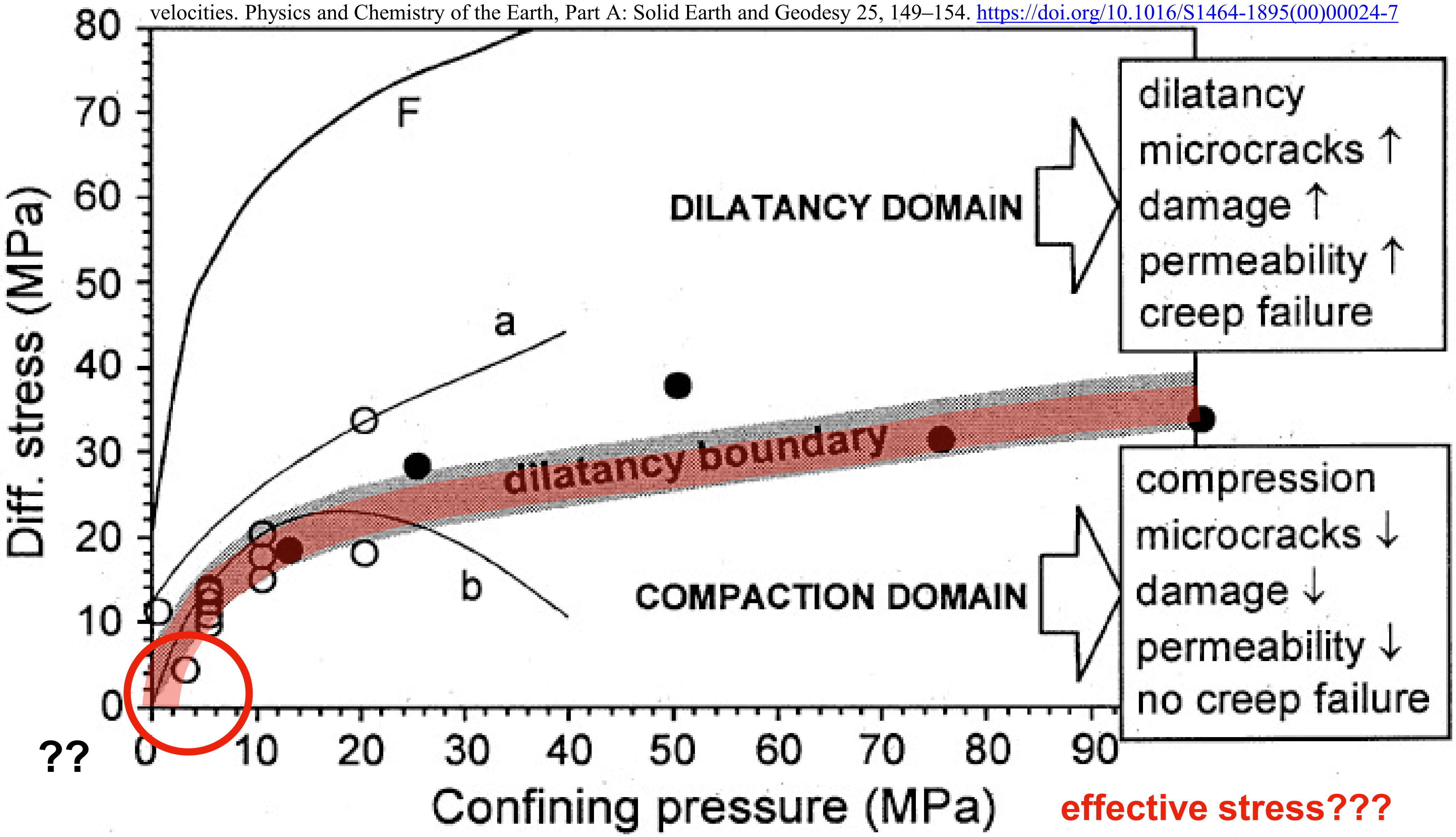


MaP confidential
Microstructure and Pores

Schleder, Z., Urai, J.L., Nollet, S., Hilgers, C., 2008. Solution-precipitation creep and fluid flow in Halite: A case study of Zechstein (Z1) rocksalt from Neuhof salt mine (germany). International Journal of Earth Sciences 97, 1045–1056.

Dilatancy boundary for salt

Popp, T., Kern, H., 2000. Monitoring the state of microfracturing in rock salt during deformation by combined measurements of permeability and P- and S- wave velocities. Physics and Chemistry of the Earth, Part A: Solid Earth and Geodesy 25, 149–154. https://doi.org/10.1016/S1464-1895(00)00024-7

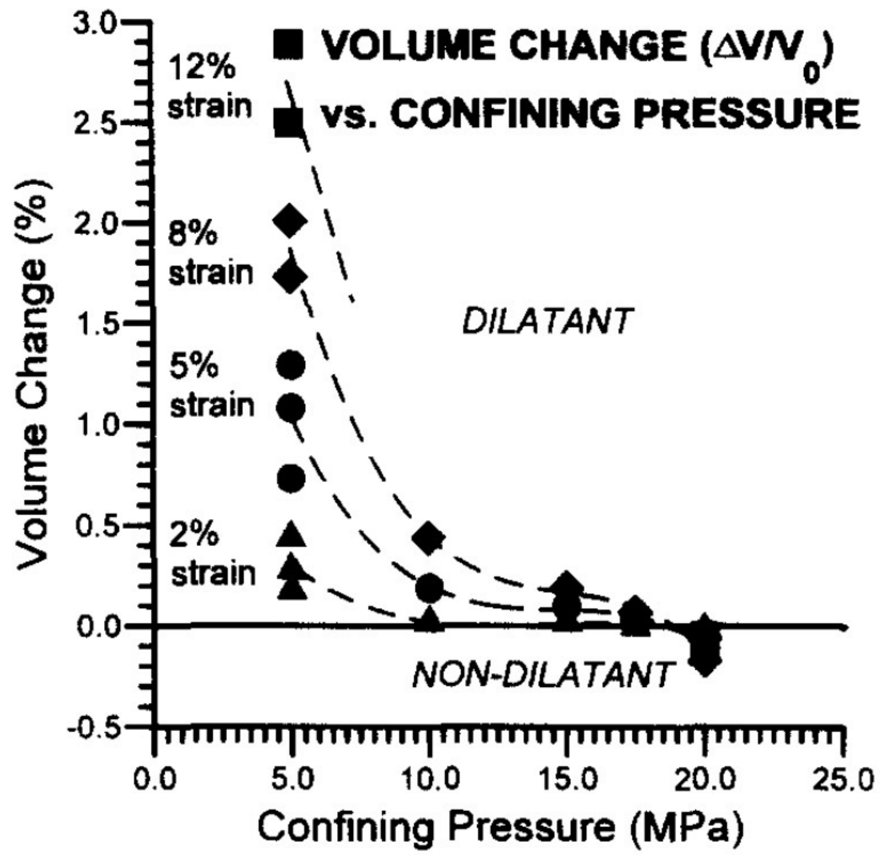


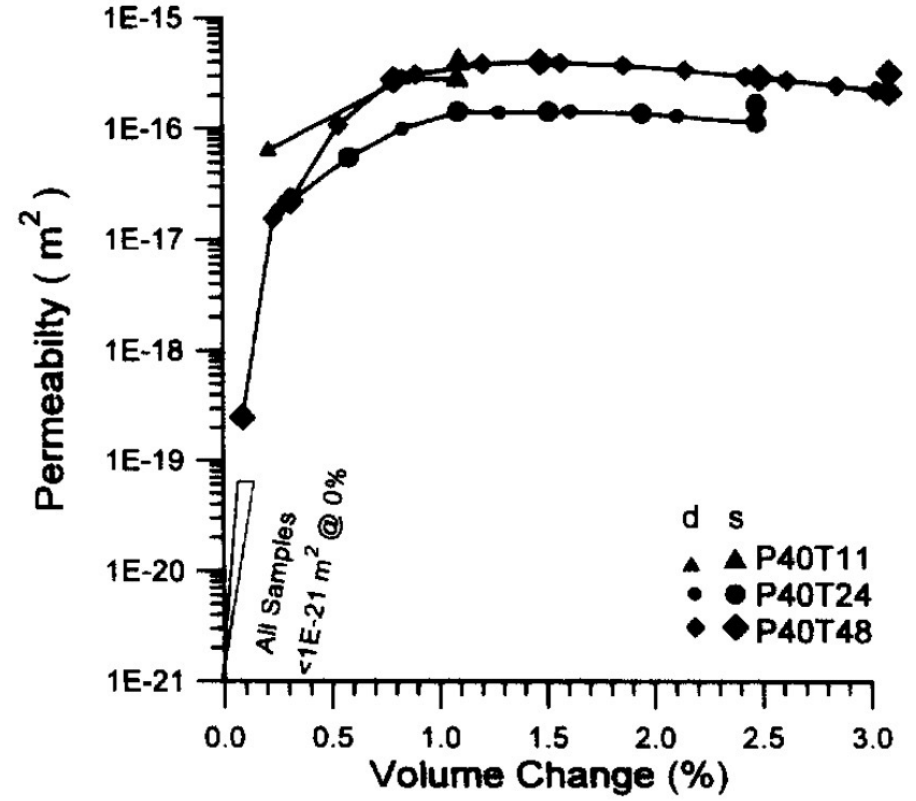
From many triaxial tests the dilatancy boundary in rock salt is reasonably known at high differential stress. However, the effects of dilatancy on microstructure mechanical properties are not well implemented in the salt engineering community. Here there are major effects if samples can lose their in-situ brine content by evaporation.

In addition, the relevant part of this diagram for the permeation problem considered here (low effective stress, low differential stress) is poorly known and needs more study, including microstructural investigations (see part 2 of this report).

Schoenherr, J., Urai, J.L., Kukla, P.A., Littke, R., Schléder, Z., Larroque, J.-M., Newall, M.J., Al-Abry, N., Al-Siyabi, H.A., Rawahi, Z., 2007. Limits to the sealing capacity of rock salt: A case study of the infra-Cambrian Ara Salt from the South Oman salt basin. AAPG Bulletin 91, 1541–1557.

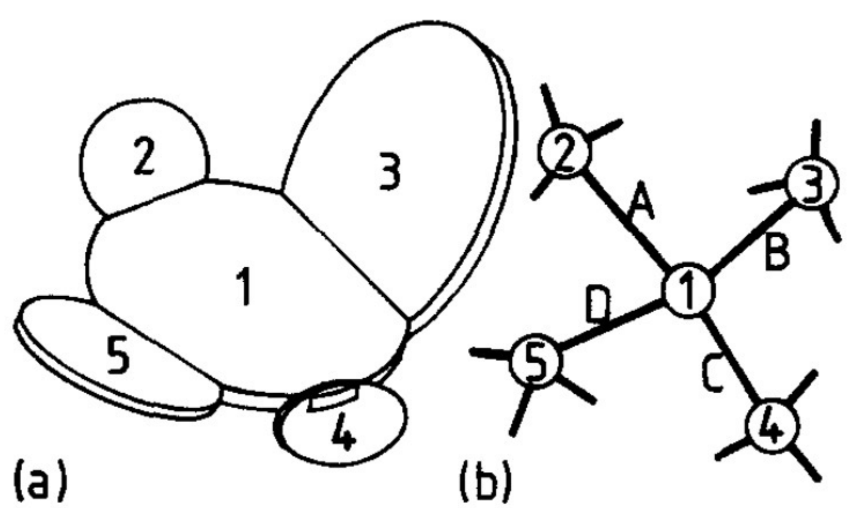
Dilatancy and Permeability evolution in salt @ 23 ºC



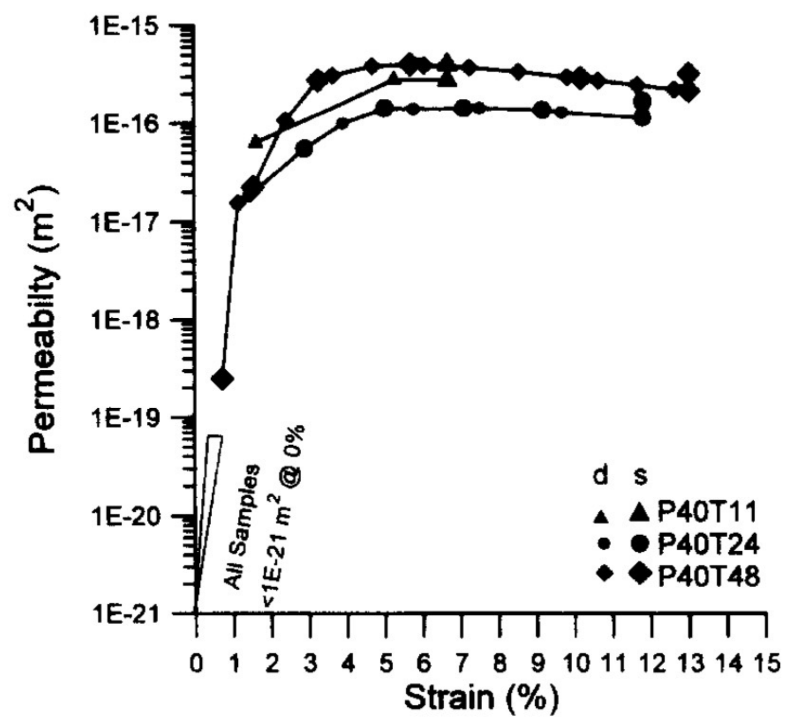


This paper is a good example of measurement of dilatancy, permeability integrated with microstructural observation and microphysical modelling using a connected microcrack model.

However the results are not directly applicable to the abandonment problem, because dilatancy is created by the imposed stress, and fluid flow is by gas, in dry rock salt.



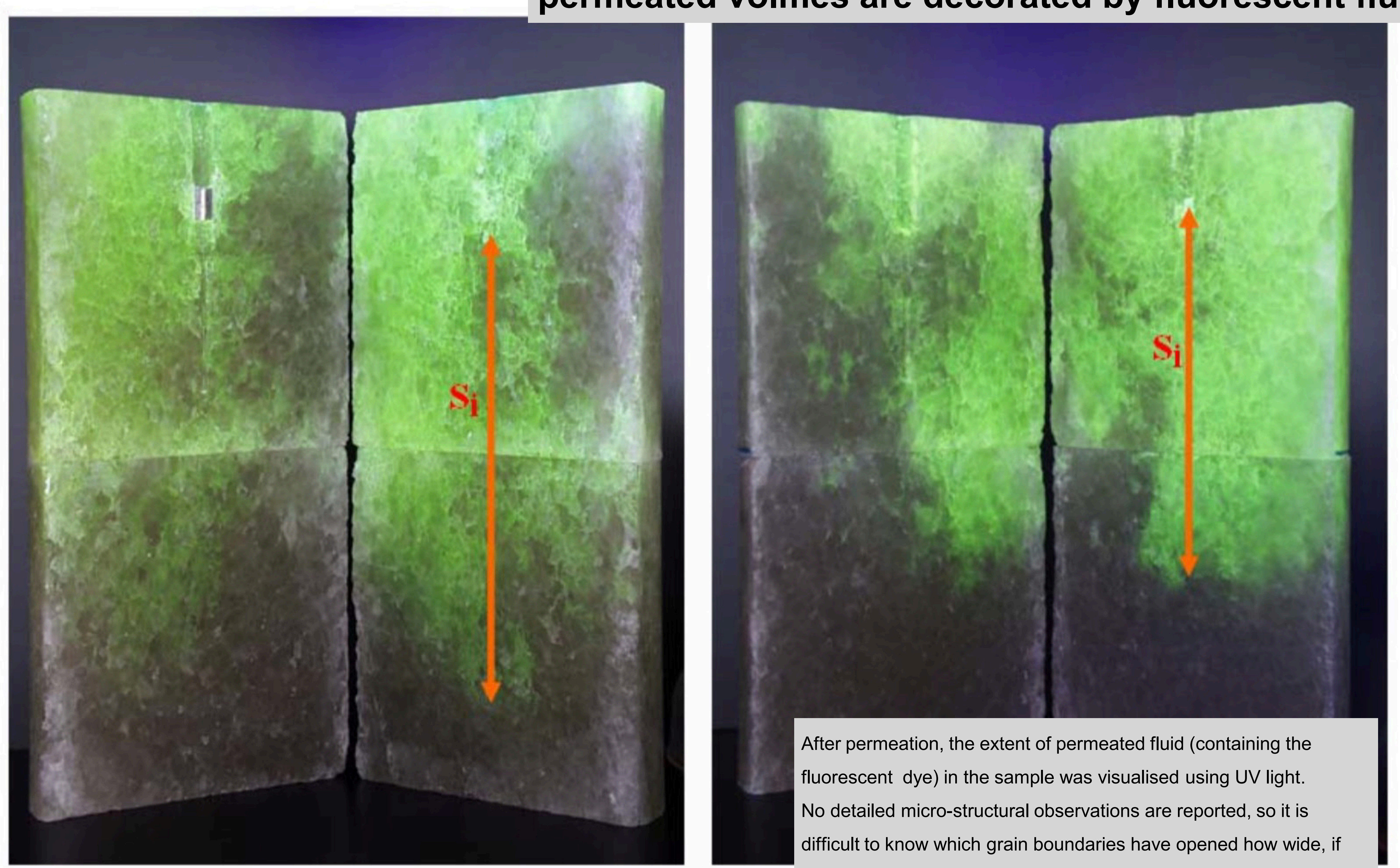
connected microcrack model



Peach, C.J., Spiers, C.J., 1996. Influence of crystal plastic deformation on dilatancy and permeability development in synthetic salt rock. Tectonophysics 256, 101–128. https://doi.org/10.1016/0040-1951(95)00170-0

Clausthal infiltration experiments

permeated volmes are decorated by fluorescent fluid



After permeation, the extent of permeated fluid (containing the fluorescent dye) in the sample was visualised using UV light. No detailed micro-structural observations are reported, so it is difficult to know which grain boundaries have opened how wide, if there were intra-crystalline microcracks, what the grain size was, etc.

Lux, K.-H., 2005. Zum langfristigen Tragverhalten von verschlossenen solegefüllten Salzkavernen – ein neuer Ansatz zu physikalischer Modellierung und

numerischer Simulation: Theoretische und laborative Grundlagen [Long-term behaviour of sealed liquid-filled salt cavities – a new approach for physical modelling
Wolters, R., Lux, K.-H., Düsterloh, U., 2011. Fluid infiltration processes into rock salt barriers resulting from fluid pressure build-up due to
and numerical simulation – basics from theory and lab investigations]. Erdöl, Erdgas, Kohle 121, 414–422.
convergence, thermal expansion and gas generation. In: Li, X., Jing, L., Blaser, P. (Eds.), Impact of Thermo-Hydro-Mechanical Chemical

(THMC) Processes on the Safety of Underground Radioactive Waste Repositories. European Commission, Luxembourg, 209–218.

Infiltration rate increases rapidly with excess pressure

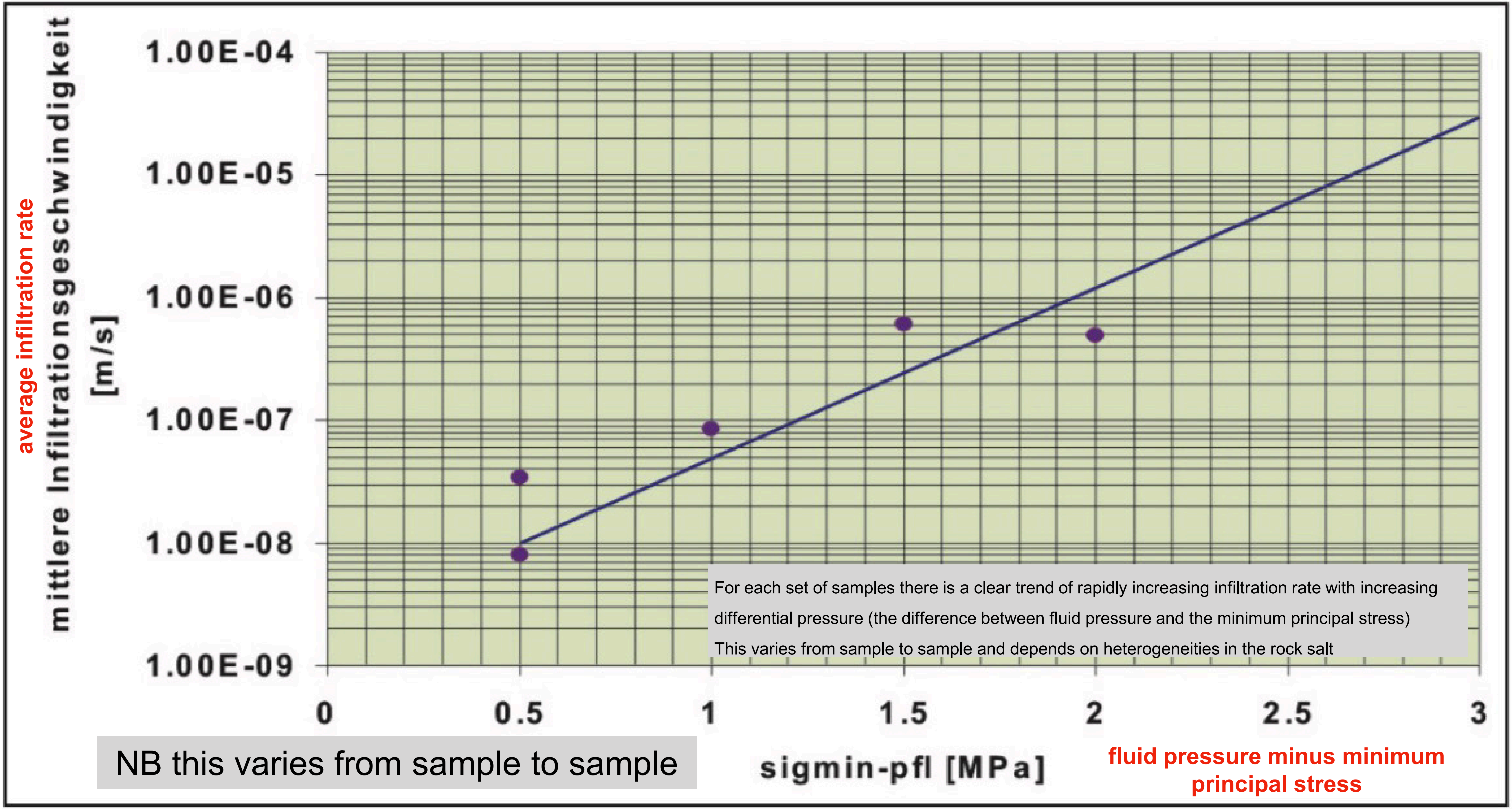
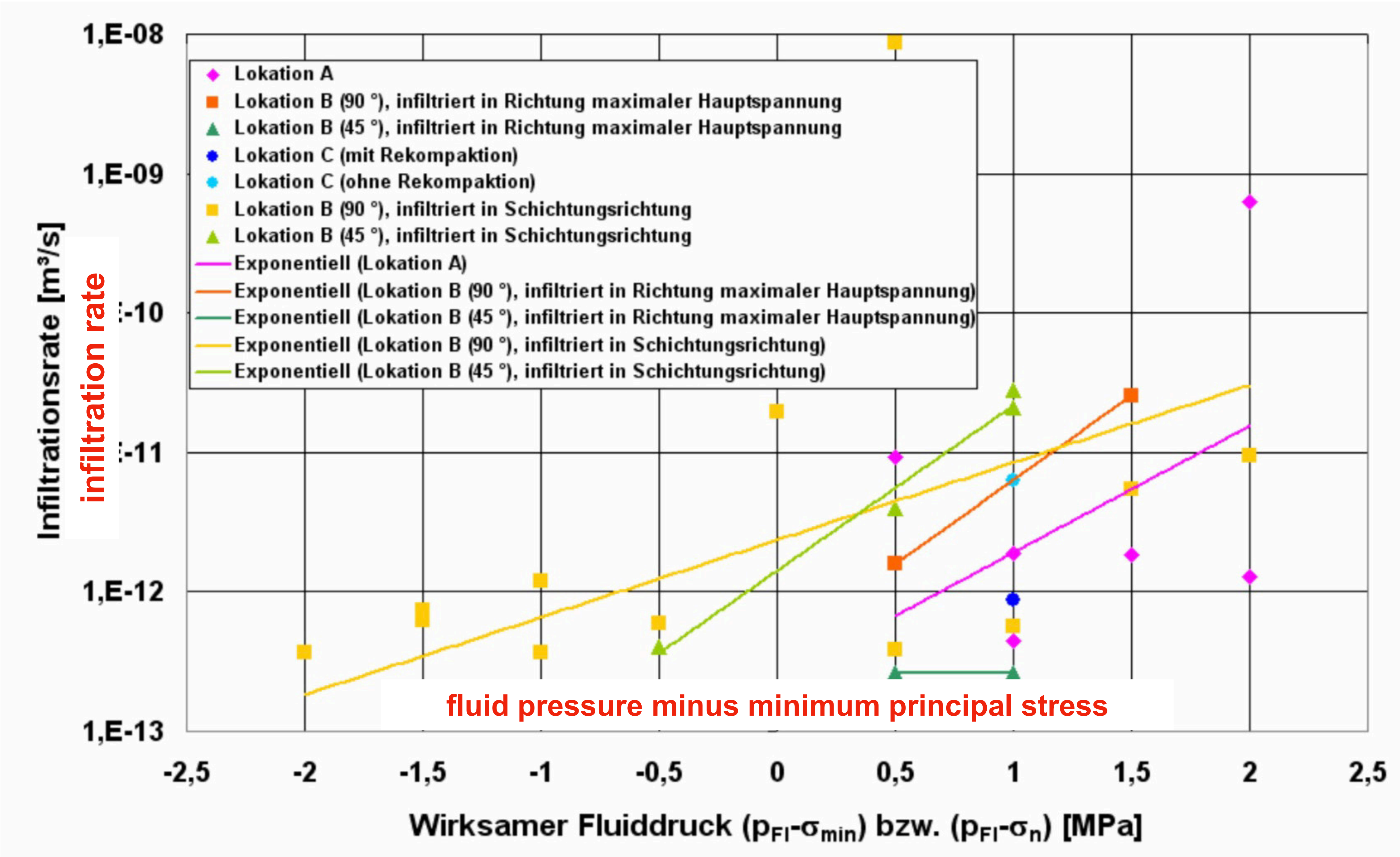


Abb. 9 Mittlere Infiltrationsrate in Abhängigkeit vom Differenzdruck

Lux, K.-H., 2005. Zum langfristigen Tragverhalten von verschlossenen solegefüllten Salzkavernen – ein neuer Ansatz zu physikalischer Modellierung und numerischer Simulation: Theoretische und

Infiltration rate depends on the sample (MICROSTRUCTURE)



The results show that the kinetics of permeation vary with microstructure and impurities. In this plot the large differences between samples are summarised. In some cases, infiltration is possible at excess pressure below zero.

Although this project does not aim to model this process at the cavern scale, here we propose that therefore permeation after cavern abandonment will be strongly heterogeneous and localised.

Wolters, R., Düsterloh, U., 2009. Weiterentwicklung der EDV-Software INFIL zur Simulation des druckgetriebenen Infiltrationsprozesses von Fluiden in ein nicht permeables Barrieren-Gebirge (Salinar). Final Report No. 02C1355. Technical University Clausthal, Clausthal, Germany.

Current numerical modelling appears to use homogeneous material properties, resulting in a far too large infiltrated volume

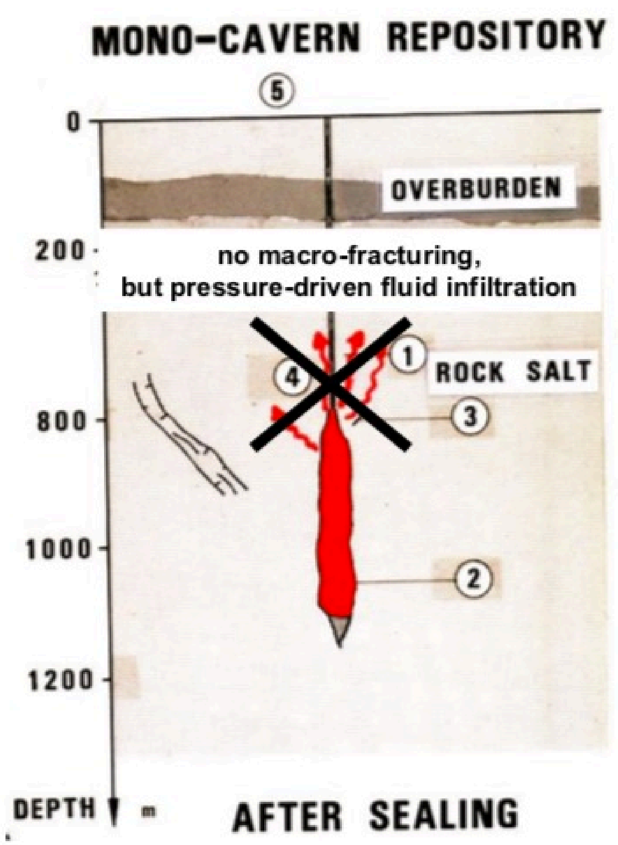


Figure 3. Abandoned and sealed liquid-filled salt cavity (Lux 2009)

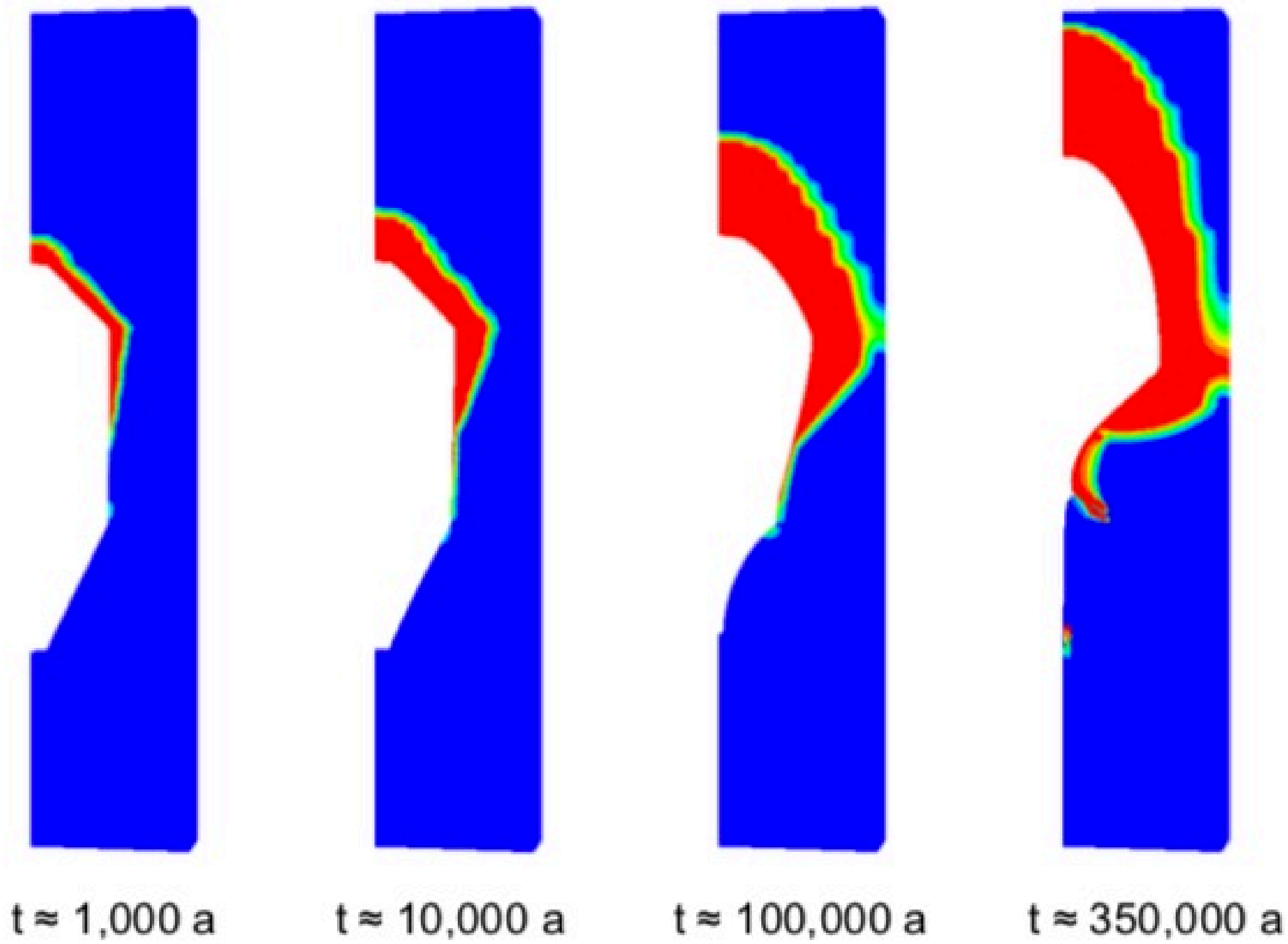


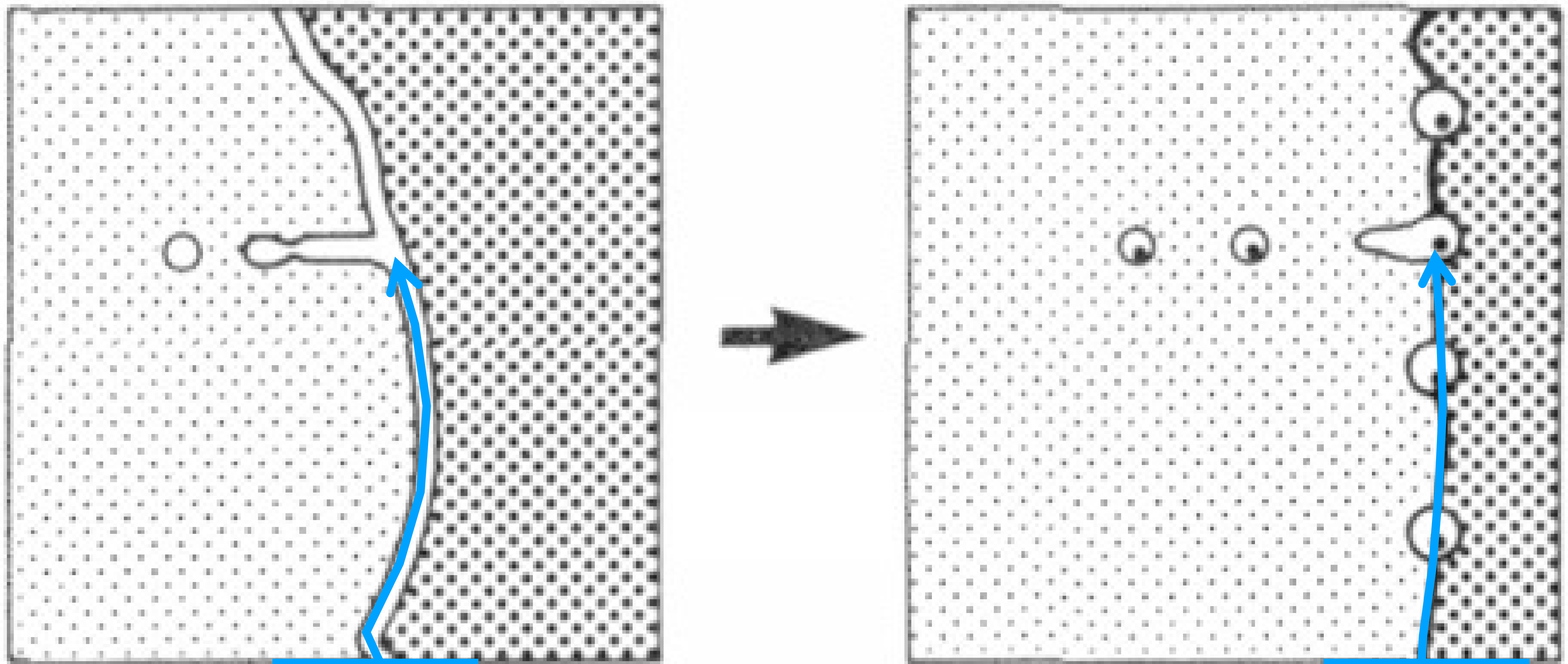
Figure 16. Pressure-driven fluid infiltration processes around a sealed brine-filled cavern in a rock salt mass

Although this part of the project does not aim to model this process at the cavern scale, (this will be done in the second part of the project) here we propose that permeation after cavern abandonment will be much more heterogeneous and localised.

Wolters, R., Lux, K.-H., Düsterloh, U., 2017. Rock-mechanical investigations regarding the proof of long-term safety of abandoned salt production cavities using hazardous waste as backfill material. Presented at the SMRI Spring 2017 Technical Conference, 23–26 April 2017, Solution Mining Research Institute, Albuquerque, New Mexico, USA, 1–19.

The infiltration of of grain boundaries

polycrystalline rock salt at anisotropic stress state



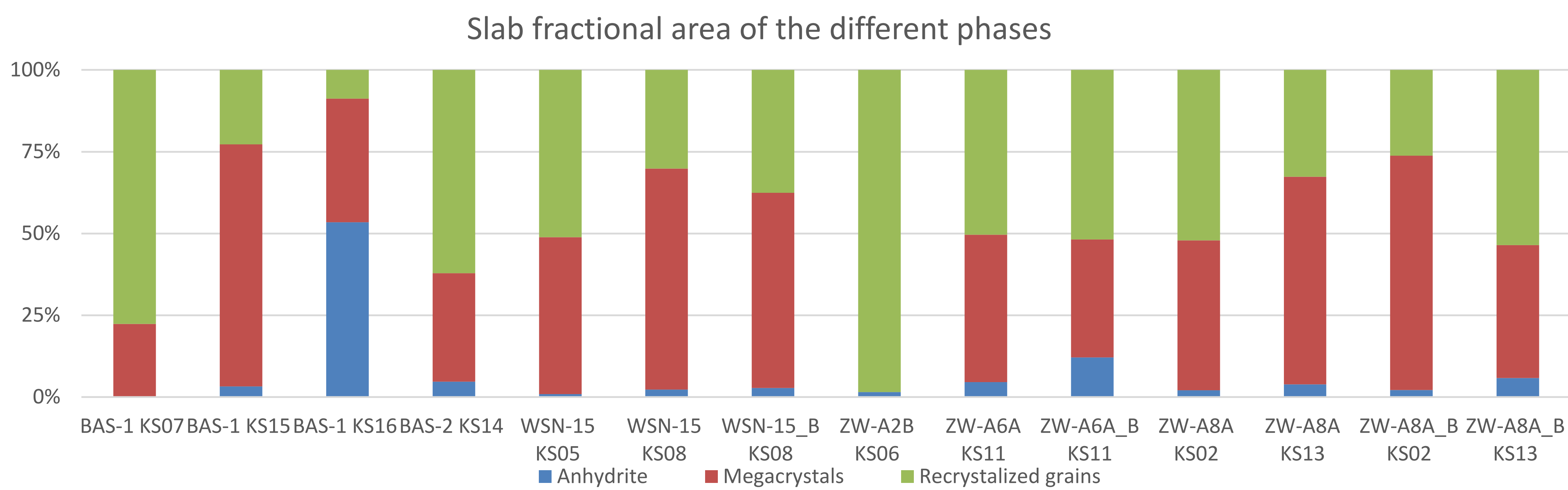
local fluid reservoir at pressure slightly higher than the local normal stress on the grain boundary (? = minimum stress in the rock salt ?)

Site locations core material

Source: https://www.nlog.nl/kaart-boringen, Vergunningen steenzout 02/01/2019

Macroscale: Slab segmentation and analysis

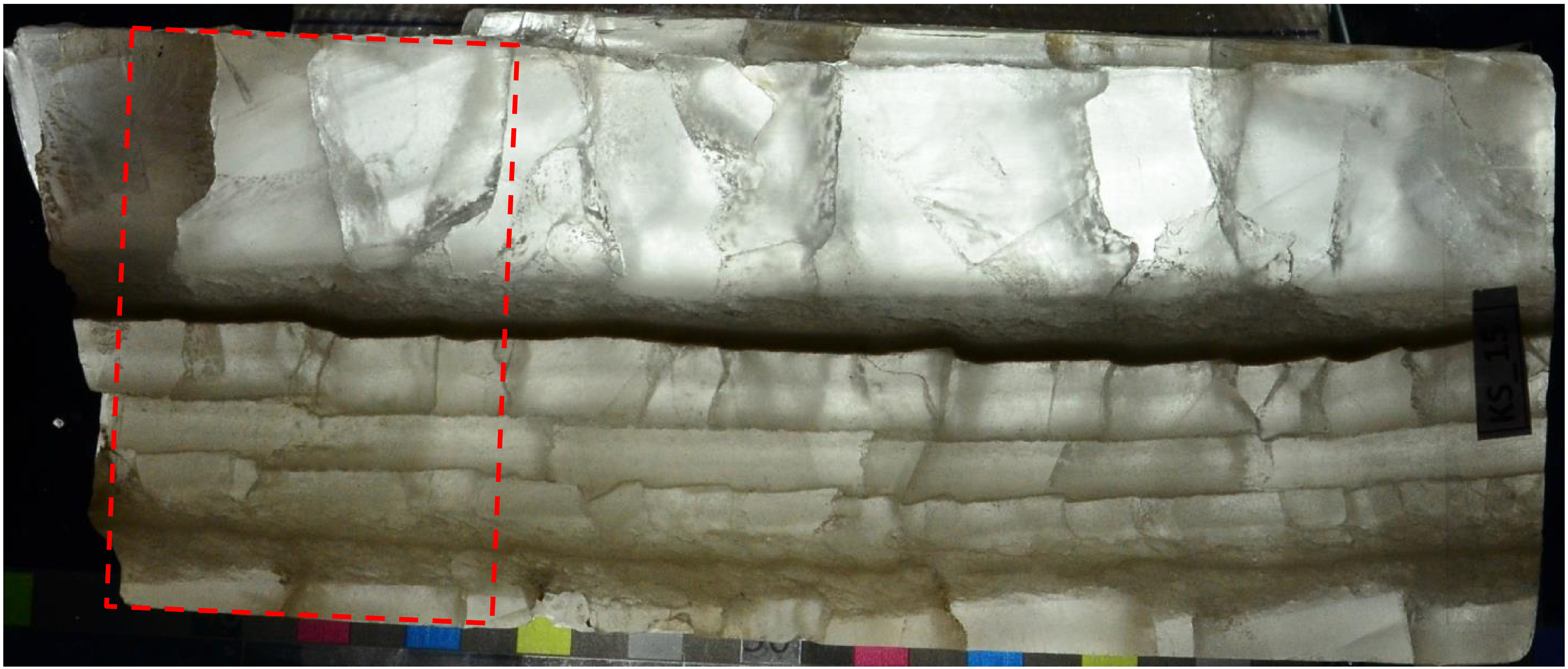
Phase mapping of core slab photos showed that the core samples were heterogeneous and predominantly consisting of three phases: Halite megacrystals alternating with recrystallized Halite polycrystalline layers and with Anhydrite layers. This causes an overall bimodal grain size distribution of the Halite fraction. While hundreds of recrystallized grains were measured in the polycrystalline layers, only a few, but large single crystals (megacrystals) were found. The size of the megacrystals was not measured as it exceeded in most cases the size of the thin section and even the slab.



	Barradel				Winschoten			Zuidwending				
SAMPLE	BAS-1 KS07	BAS-1 KS15	BAS-1 KS16	BAS-2 KS14	WSN-15 KS05	WSN-15 KS08	WSN-15_B KS08	ZW-A2B KS06	ZW-A6A KS11	ZW-A6A_B KS11	ZW-A8A KS02	ZW-A8A K
Anhydrite	0.0%	3.2%	53.4%	4.7%	0.9%	2.3%	2.8%	1.5%	4.5%	12.1%	2.1%	3.9%
Single Crystals	22.3%	74.0%	37.8%	33.2%	48.0%	67.6%	59.7%	0.0%	45.1%	36.1%	45.8%	63.5
Recrystallized grains	77.7%	22.8%	8.8%	62.1%	51.1%	30.2%	37.6%	98.5%	50.4%	51.8%	52.1%	32.7

Barradeel, KS_15 BAS-1, 2845.0 m

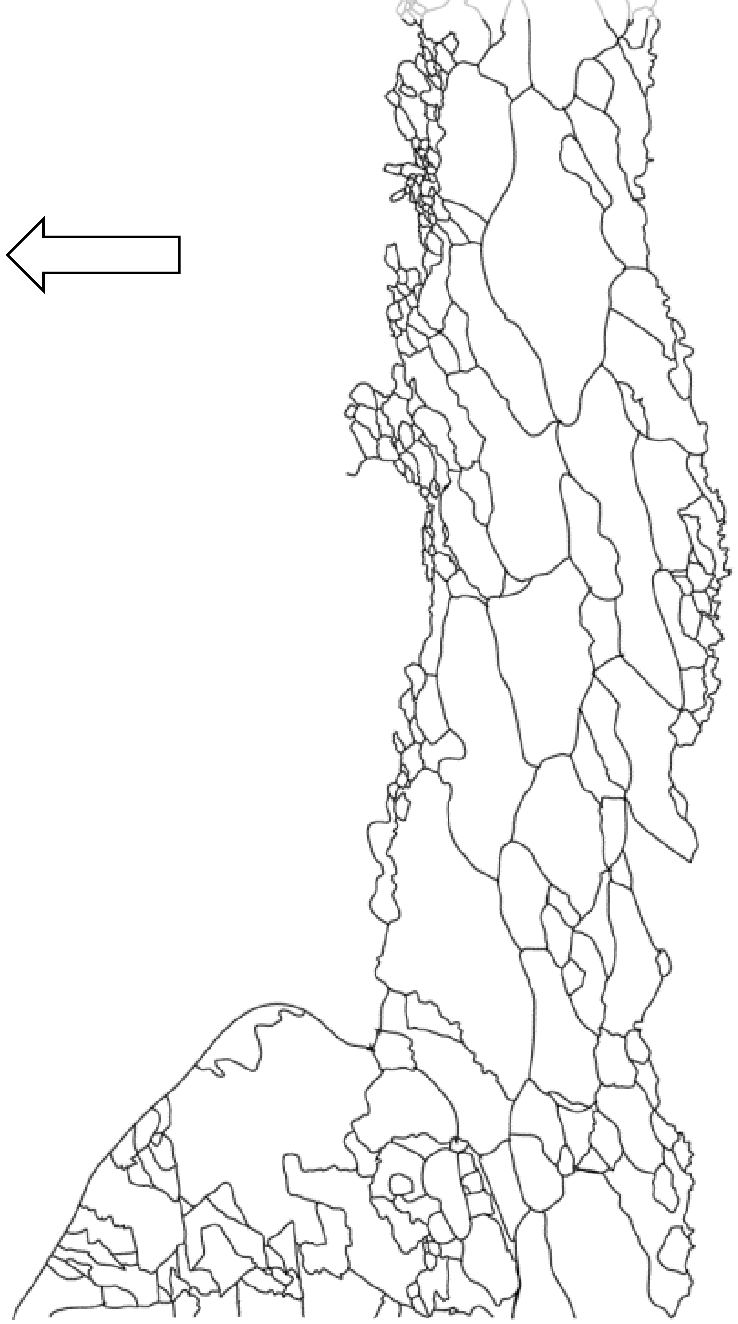
Core slab (KS_15) of well BAS-1 from 2845 m depth in transmitted light showing deformed (layer parallel shear, foliation) Anhydrite layers bounding large to very large crystals (>cm scale). Red frame indicating thin section location.

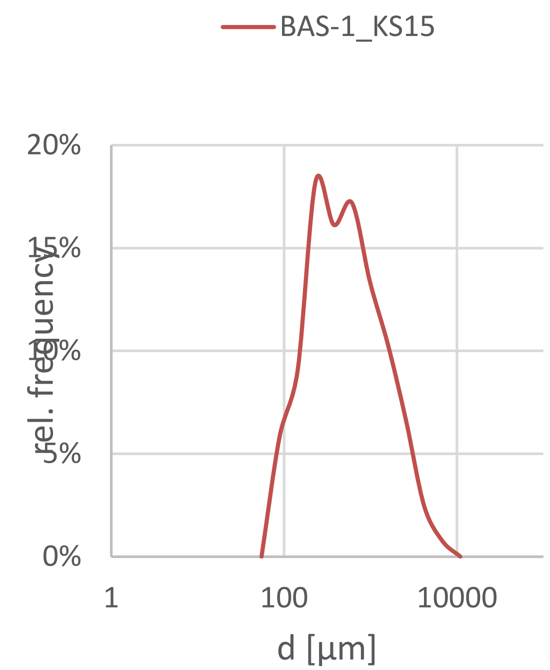


5 cm

Barradeel, KS_15 BAS-1, 2845.0 m, Grain Size analysis

Grain size distribution of the recrystallized areas was measured using micrographs acquired in reflected light. The grain boundaries were exposed using chemical etching facilitating semi-automated image analysis. The recrystallized grain size was log-normally (with visible influence of small grain size) distributed with decreasing grain size towards the interbedded anhydrate layers and close to Halite single crystals. Halite megacrystals (one crystal in this thin section) were excluded from the measurement as they cross-cut the thin section.





Equivalent diameter d [µm]	
Mean	659.6
Standard Error	29.5
Median	381.2
Mode	66.6
Standard Deviation	753.6
Minimum	66.6

1.5 cm

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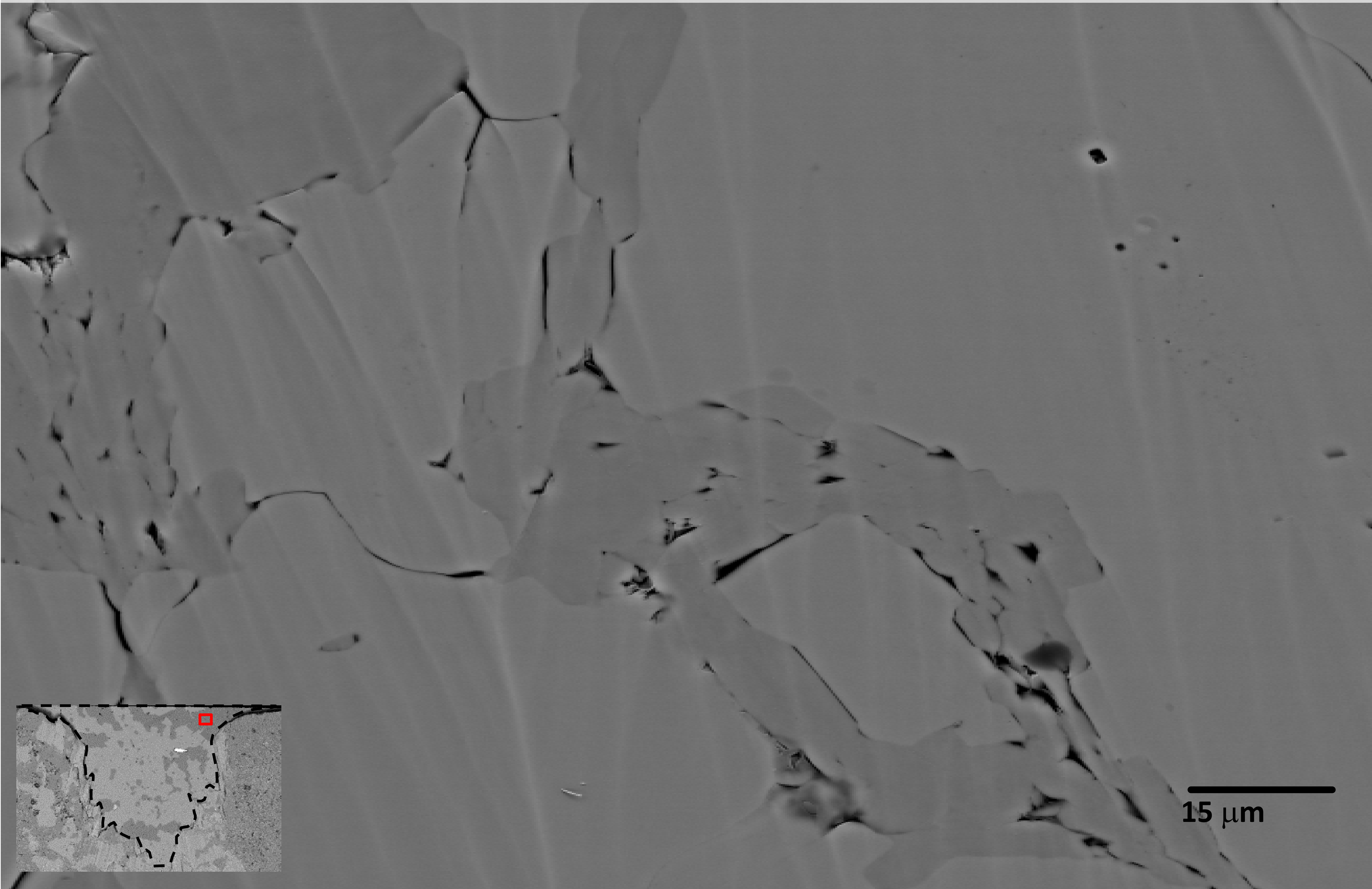
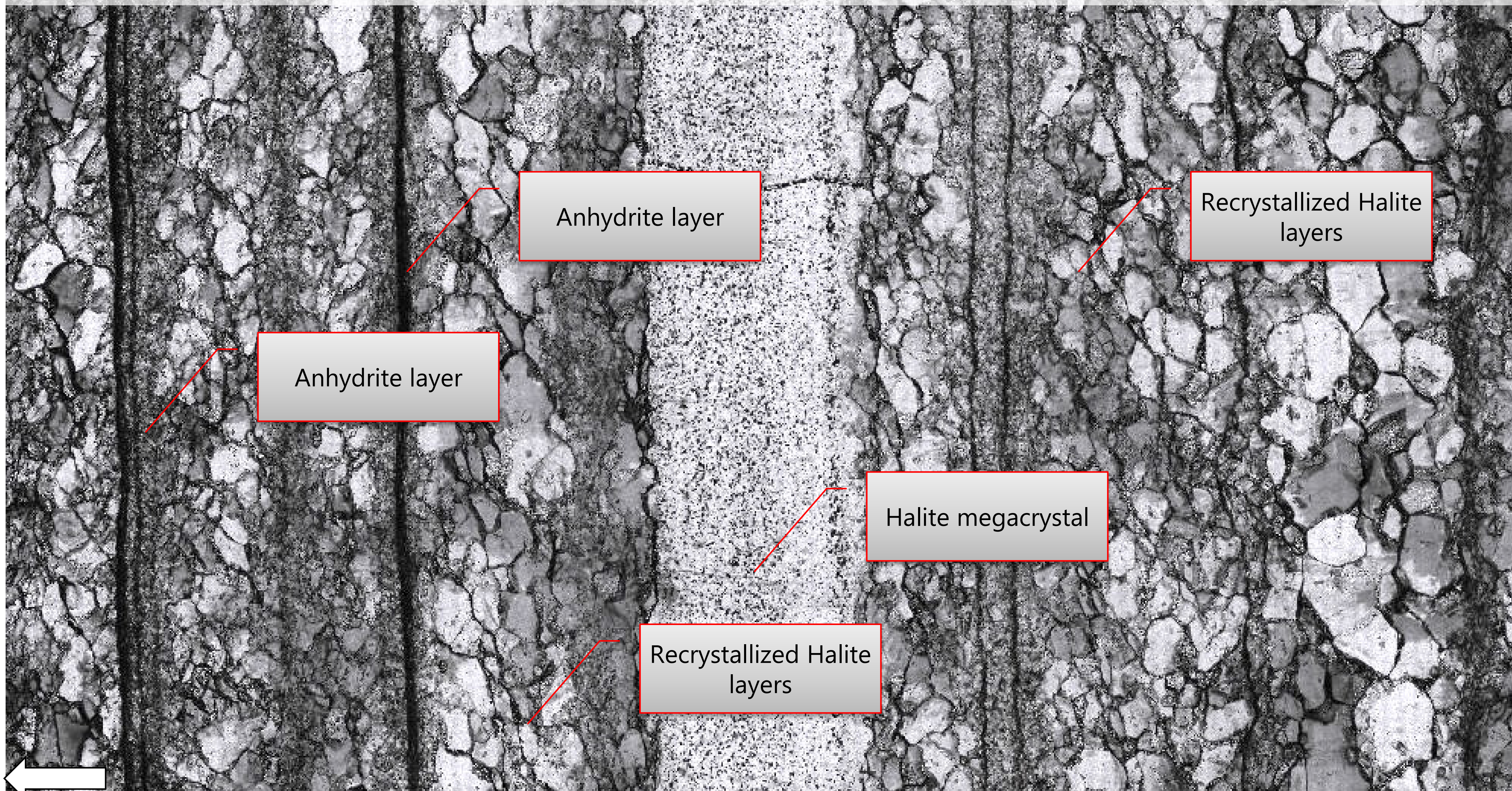
Barradeel, KS_15 BAS-1, 2845.0 m, BIB-SEM analysis

SEM (BSE) micrograph showing overview on BIB-milled cross section showing porosity in between the three different phases.



Barradeel, KS_07 BAS-1, 2680.4 m, ViP – ppol

ViP micrograph acquired in transmitted plane polarized light was used to investigate the grain boundary fluid morphology, as well as the nature and distribution of second phase impurities such as Anhydrite grains, enclosed to Halite grains and grain boundaries. This overview of the entire cross-section allows fast discrimination of Halite single crystals and recrystallized areas.



MaP Microstructure and Pores

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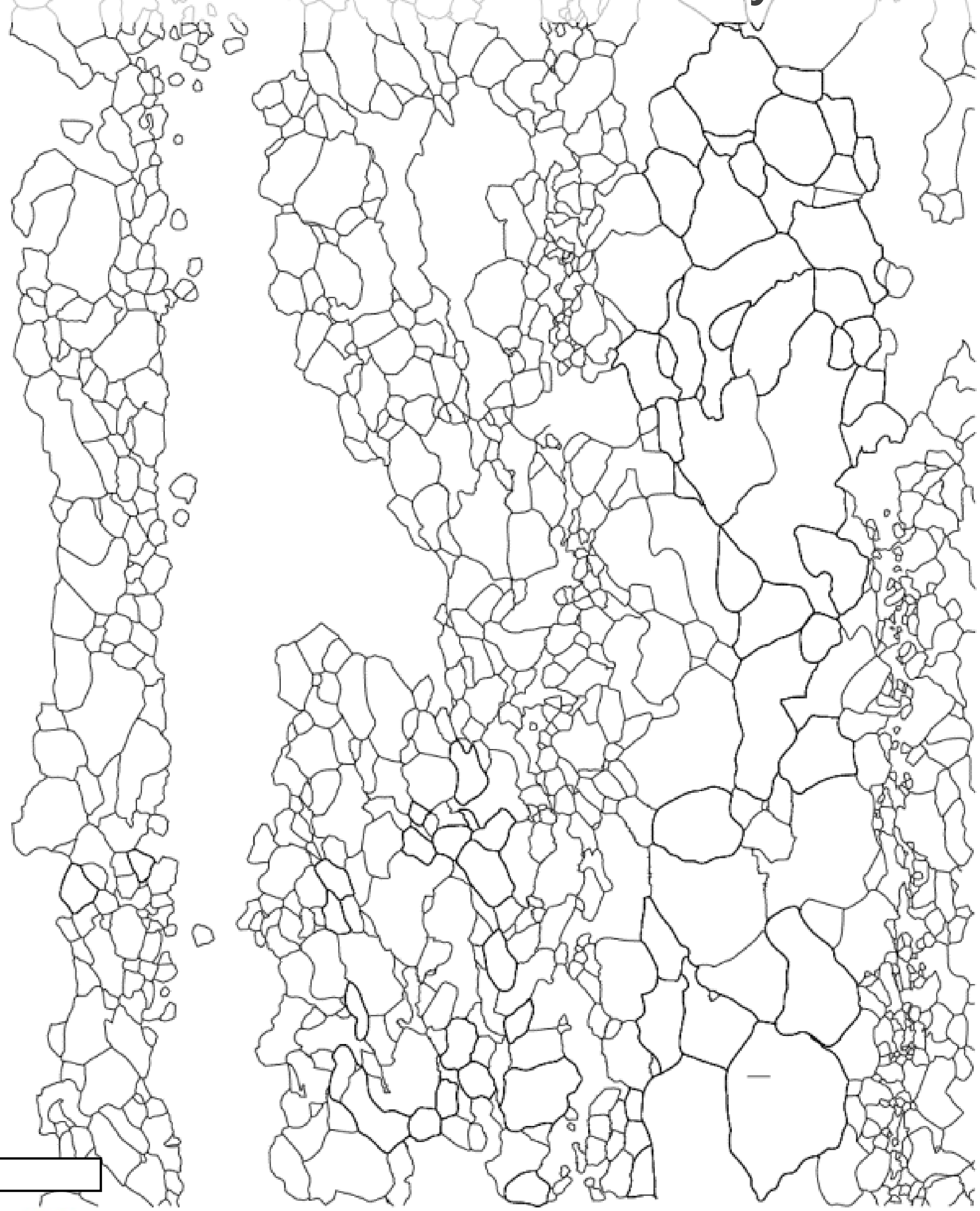
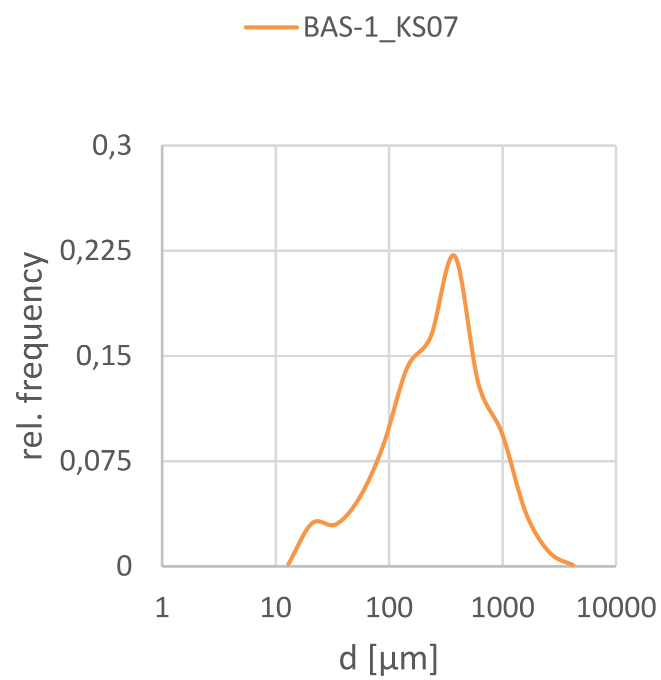
Barradeel, KS_07 BAS-1, 2680.4 m, grain size analysis

Grain size distribution of the recrystallized areas was measured using micrographs acquired in reflected light. The grain boundaries were exposed using chemical etching facilitating semi-automated image analysis. The recrystallized grain size was log-normally distributed with decreasing grain size towards the interbedded anhydrate layers and close to Halite megacrystals. Halite megacrystals (one crystal in this thin section) were excluded from the measurement as they cross-cut the thin section.





Equivalent diameter d [µm]	
Mean	855
Standard Error	25
Median	604
Mode	38
Standard Deviation	880
Minimum	32
Maximum	8885
Count	1201

1.5 cm

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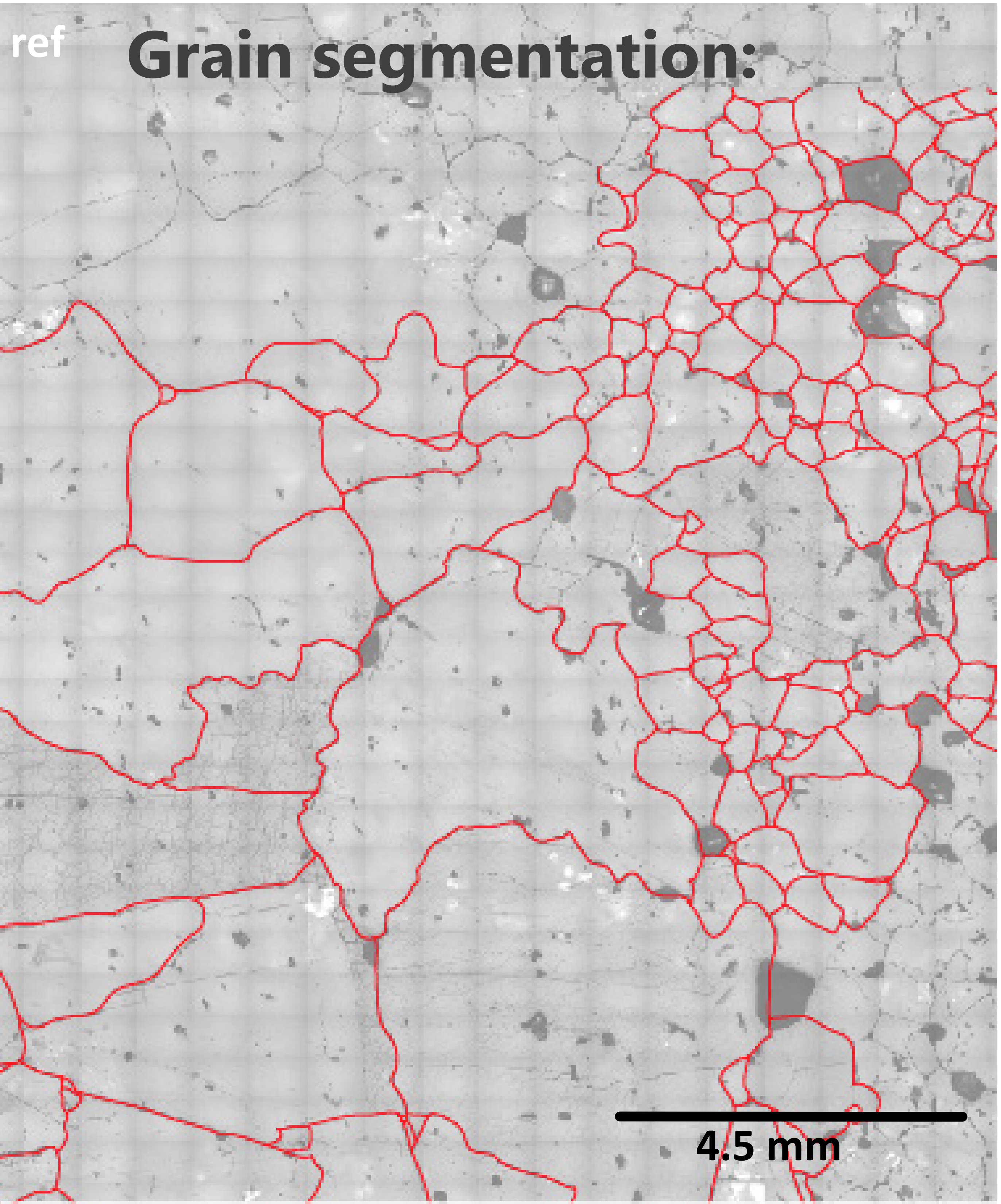
Microstructure and Pores

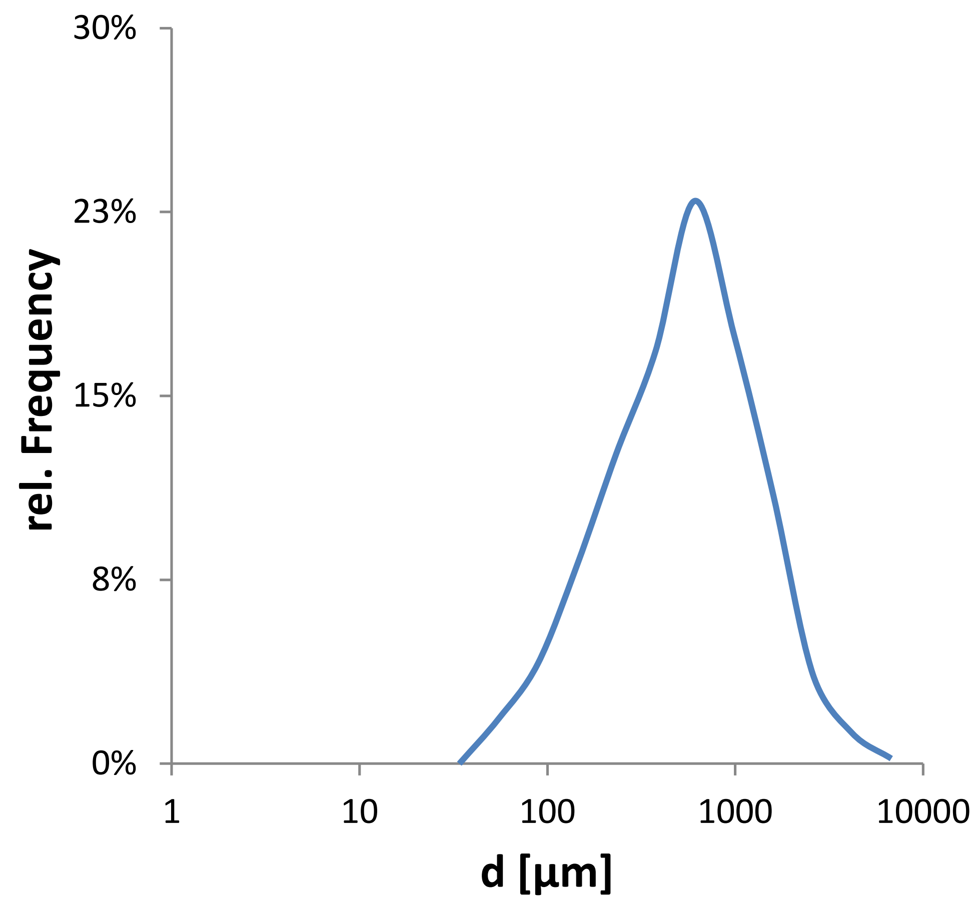
Slab photography and analysis

Photographs of slabs using incident and transmitted light (upper picture) were used to estimate areal fractions of the different phases and to select thin section locations. The slab photos were segmented (lower image) using semi-automated image processing and analysis to derive the area fraction of Anhydrite, recrystallized Halite, and Halite megacrystals.

ViP – Grain size Analysis

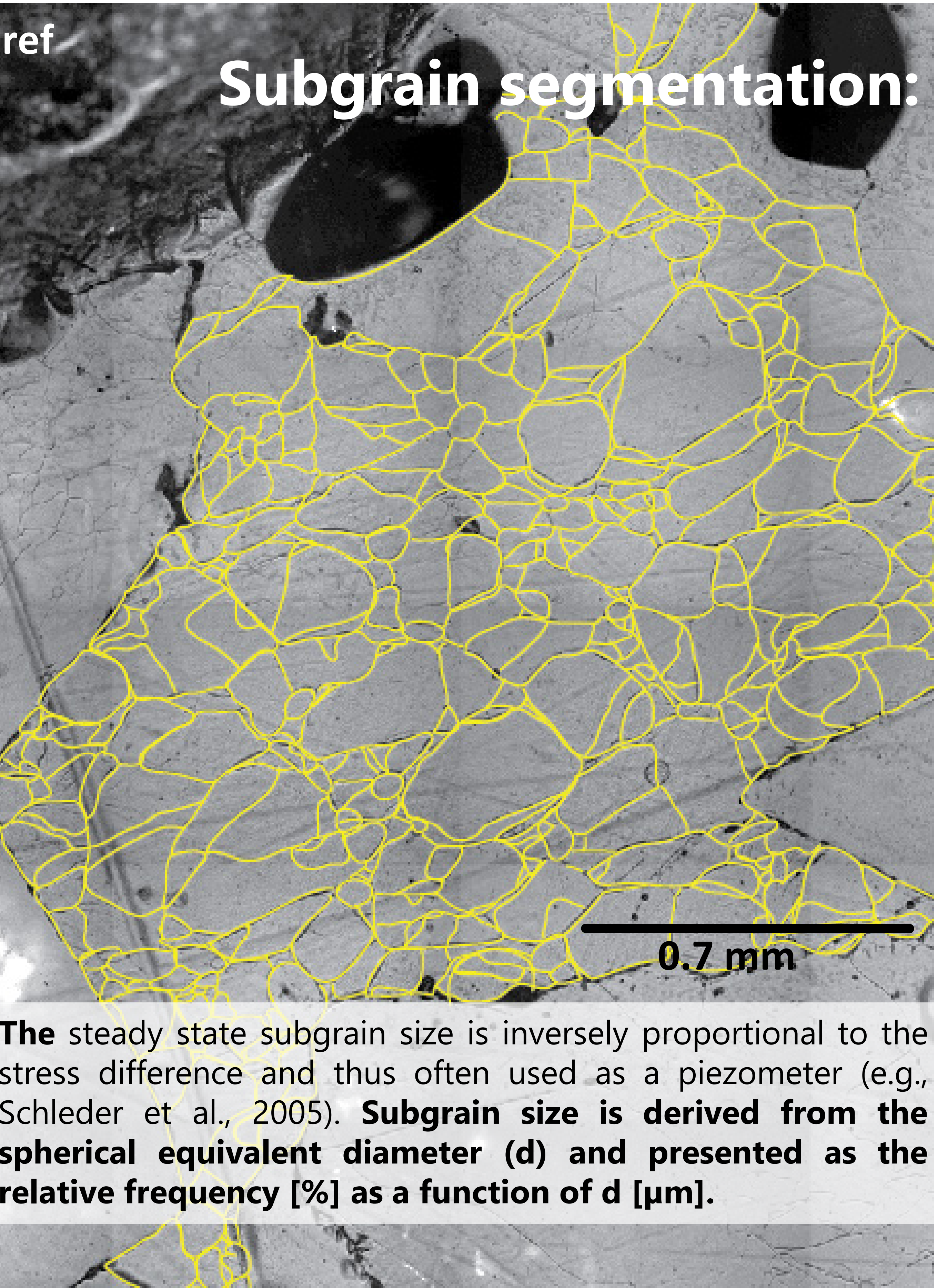


Grain size is derived from the spherical equivalent diameter (d) of the traced area and presented as the relative frequency [%] as a function of d [µm].

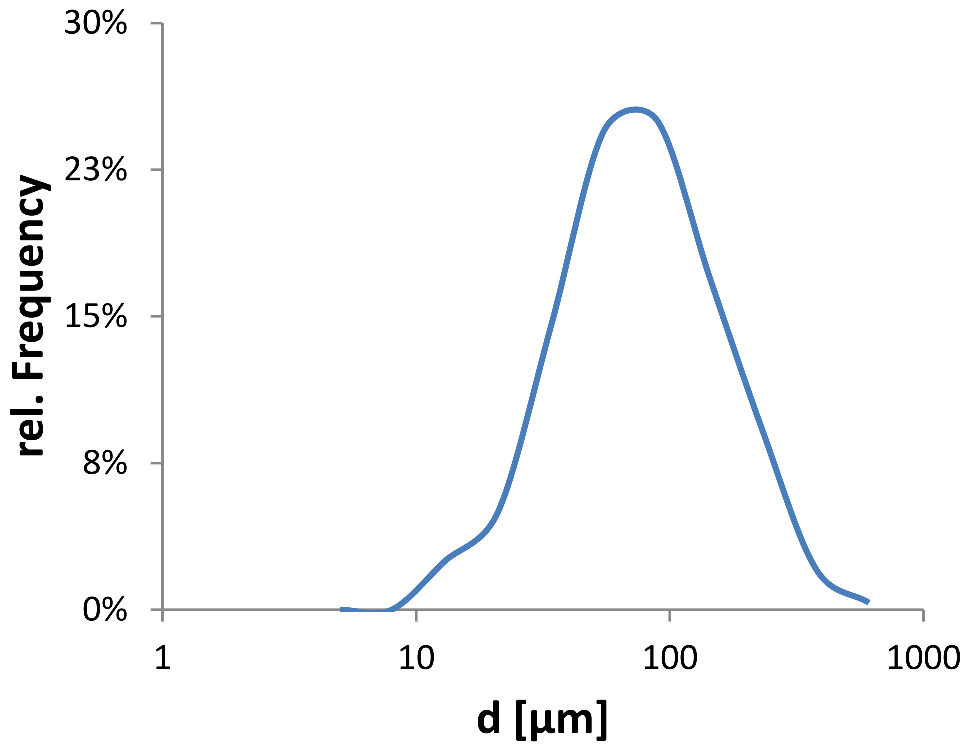


Equivalent diameter d [µm]	
Mean	593
Standard Error	13
Median	430
Mode	43
Standard Deviation	579
Minimum	43
Maximum	4769
Count	1912

ViP – Subgrain Analysis



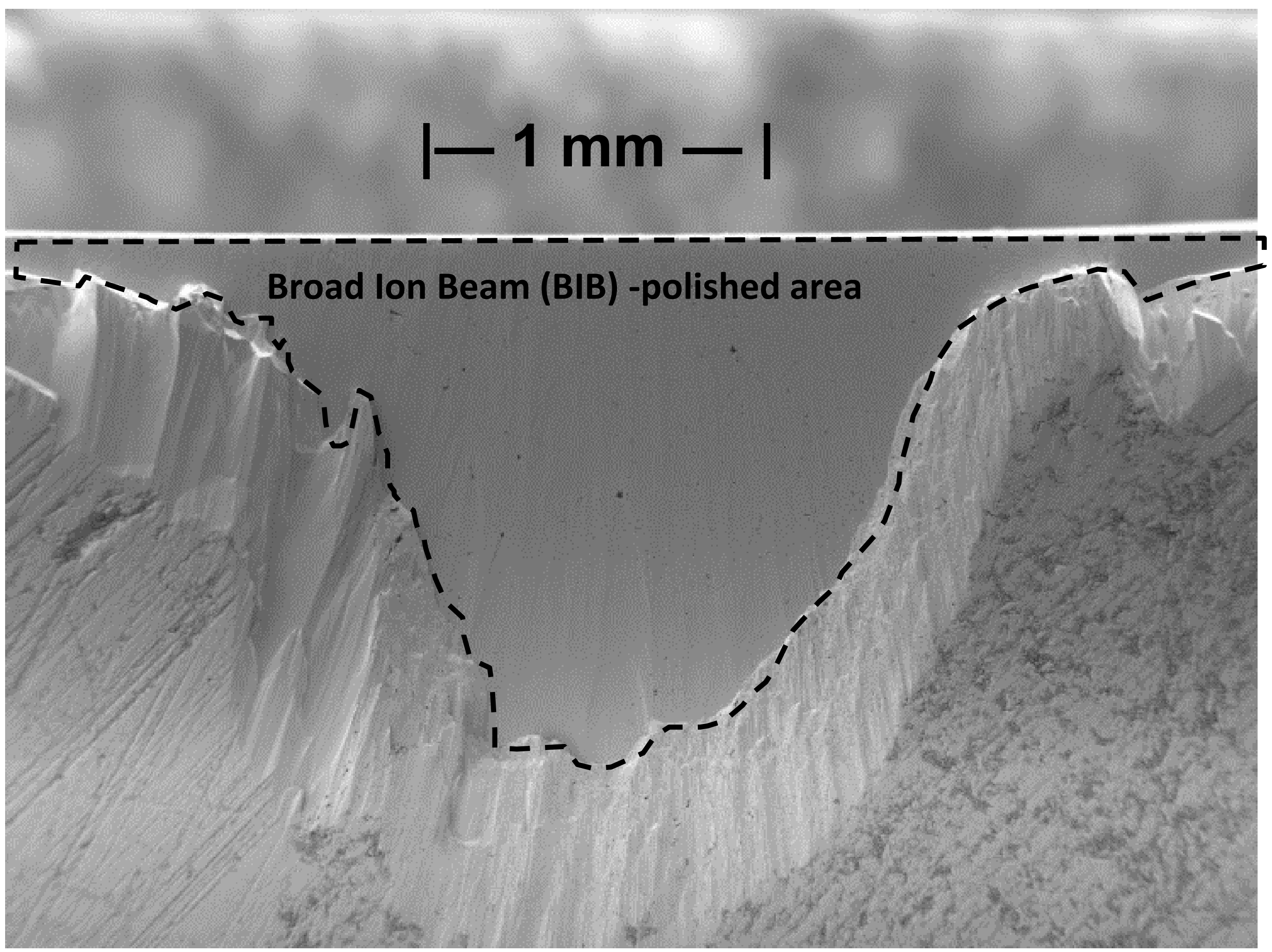
The steady state subgrain size is inversely proportional to the stress difference and thus often used as a piezometer (e.g., Schleder et al., 2005). **Subgrain size is derived from the spherical equivalent diameter (d) and presented as the relative frequency [%] as a function of d [µm].**

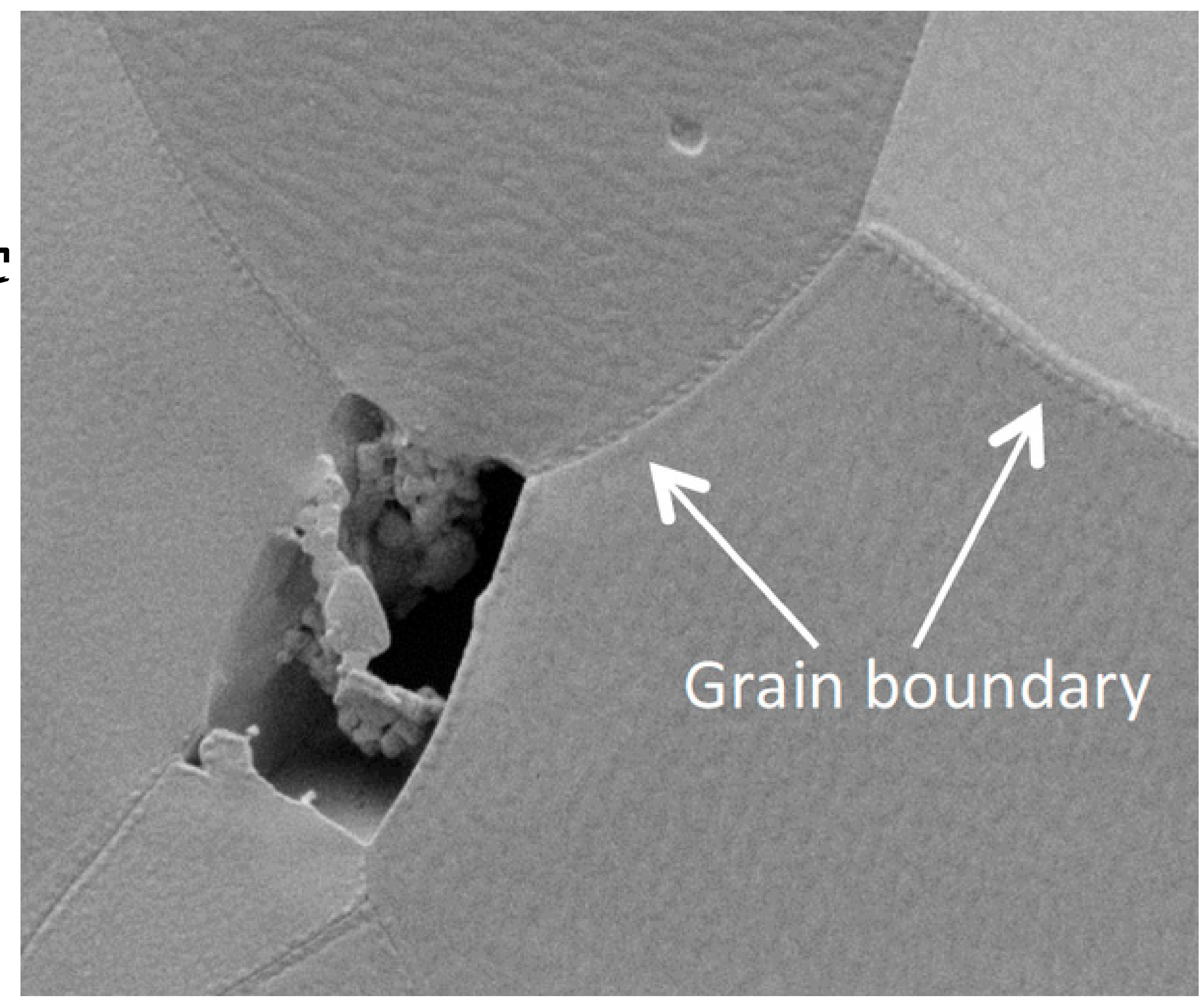


Equivalent diameter d [µm]	
Mean	76.3
Standard Error	1.5
Median	58.7
Mode	9.0
Standard Deviation	60.0
Minimum	9.0
Maximum	516.4
Count	1682

BIB-SEM

Combining Broad Ion Beam (BIB) milling with Scanning Electron Microscopy (SEM) enables topography-, phase-, and chemical- mapping using Secondary Electrons (SE), Backscattered Electrons (BSE) and Energy Dispersive X-ray Spectroscopy (EDS). The argon ion sputtered surface can be imaged at nano-scale resolution to investigate the pore geometries and grain boundaries.





Zuidwending, KS_11 ZW-A6A, 599.4 m, microstructural observations

ViP micrograph showing triple junction of coarse, recrystallized Halite grains exposing fluid inclusion arrays on the grain surfaces.