Engineering Geology 239 (2018) 206–213

Contents lists available at ScienceDirect

Engineering Geology

journal homepage: www.elsevier.com/locate/enggeo

Micromechanical processes in consolidated granular salt

Melissa M. Mills[a,b,*], John C. Stormont[a], Stephen J. Bauer[b]

[a] Department of Civil Engineering, University of New Mexico, MSC01 1070, 1 University of New Mexico, Albuquerque, NM 87131-0001, USA
[b] Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185, USA

ARTICLE INFO

Keywords:
Granular salt consolidation
Deformation mechanism
Microstructural investigations
Geologic nuclear waste repository applications

ABSTRACT

Granular salt is likely to be used as backfill material and a seal system component within geologic salt formations serving as a repository for long-term isolation of nuclear waste. Pressure from closure of the surrounding salt formation will promote consolidation of granular salt, eventually resulting in properties comparable to native salt. Understanding dependence of consolidation processes on stress state, moisture availability, temperature, and time is important for demonstrating sealing functions and long-term repository performance. This study characterizes laboratory-consolidated granular salt by means of microstructural observations. Granular salt material from mining operations was obtained from the bedded Salado Formation hosting the Waste Isolation Pilot Plant and the Avery Island salt dome. Laboratory test conditions included hydrostatic consolidation of jacketed granular salt with varying conditions of confining isochoric stress to 38 MPa, temperature to 250 °C, moisture additions of 1% by weight, time duration, and vented and non-vented states. Resultant porosities ranged between 1% and 22%. Optical and scanning electron microscopic techniques were used to ascertain consolidation mechanisms. From these investigations, samples with 1% added moisture or unvented during consolidation, exhibit clear pressure solution processes with tightly cohered grain boundaries and occluded fluid pores. Samples with only natural moisture content consolidated by a combination of brittle, cataclastic, and crystal plastic deformation. Recrystallization at 250 °C irrespective of moisture conditions was also observed. The range and variability of conditions applied in this study, combined with the techniques used to display microstructural features, are unique, and provide insight into an important area of governing deformation mechanism(s) occurring within salt repository applications.

1. Introduction

1.1. Background

Rock salt is a viable geologic repository host for disposal of waste from nuclear energy and nuclear weapon by-products, owing to its location in stable geologic areas, plastic material behavior, and low permeability (National Research Council, 1957). Germany, The Netherlands, Poland, and the United States are actively advancing salt disposal research, design, and operations (NEA Salt Club, 2015) in bedded and domal formations. Disposal of chemotoxic and radioactive waste in salt has been active in Germany for decades, for example at Herfa-Neurode and Morsleben mines, and in the USA at the Waste Isolation Pilot Plant (WIPP). After waste has been transported and placed in the underground, granular salt is commonly used as backfill material for mined underground openings at these facilities. Planning for future salt repositories, including heat-generating nuclear waste, relies on re-consolidation of granular salt to affect sealing functions by way of several design features, such as backfilled rooms, engineered seal systems, and fill around waste packages for encasing waste completely (Fuenkajorn and Daemen, 1996). Granular salt is expected to consolidate to a condition comparable to host rock; that is, achieve low porosity and permeability under a variety of scenarios (Stührenberg, 2007; Kröhn et al., 2015; Hansen et al., 2015). Documenting deformation mechanisms as granular salt consolidates bestows a further degree of confidence regarding operational and long-term repository performance.

Granular salt consolidation is essentially pore volume reduction, where the amount and nature of final pore volume depends on multiple factors. Granular salt consolidation varies as a function of stress, temperature, moisture conditions, time, and quantity of impurities (e.g., clay). Typical salt consolidation initiates by compaction processes accomplished by brittle processes, grain rearrangement, and cataclastic flow. As void space is eliminated, grain boundary processes and crystal-plastic mechanisms control additional porosity reduction. Signatures of isochoric deformation mechanisms have been documented as

* Corresponding author at: Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185, USA.
E-mail addresses: mmmills@sandia.gov (M.M. Mills), jcstorm@unm.edu (J.C. Stormont), sjbauer@sandia.gov (S.J. Bauer).

https://doi.org/10.1016/j.enggeo.2018.03.024
Received 17 October 2017; Received in revised form 9 March 2018; Accepted 24 March 2018
Available online 27 March 2018
0013-7952/ Published by Elsevier B.V.

functionally controlled by increasing temperature and stress include dislocation multiplication, glide, cross slip, climb, fluid-assisted creep, and annealing/recrystallization. These microprocesses are described specifically in natural salt by Hansen (2014) and Urai and Spiers (2007), among others, and provide reference examples for observations made here.

A brief description of deformation mechanisms pertaining to rock salt is as follows. Deformation mechanisms in salt have been broadly investigated through microstructural observations on naturally and/or mechanically deformed samples by a variety of researchers (e.g. Urai et al., 1986a,b; Spiers and Brzesowsky, 1993; Desbois et al., 2010; Hansen et al., 2012; Broome et al., 2014). Natural intact salt deforms initially by strain hardening (although, this is currently under discussion and reinvestigation) attributed to dislocation density increase, a process observed in natural intact rock salt by Carter et al. (1982), Urai et al. (1986b), and Hunsche and Hampel (1999), among others. Hardening occurs via lattice distortion and obstructed movement lacking thermal activation for recovery processes. In salt, dislocation motion is predominately by glide or slip along the dodecahedral family of crystallographic planes where shear stresses exceed the critical resolved shear stress. Glide produces observable bands, which highlight primary slip systems (Carter and Heard, 1970). When barriers exist in the crystal lattice, glide can be aided by thermal activation, which promotes cross slip and climb (Senseny et al., 1992). Cross slip is apparent at modest temperatures and involves screw dislocation movement from one glide plane to another with the same Burger's vector (Skrotzki and Haasen, 1984). Climb occurs as dislocations move perpendicular to the glide plane at elevated temperatures. Each of these deformation mechanisms has been found to be a function of stress difference and temperature.

Brine content varies with respect to formation and impurity quantities, which impacts the behavior of rock salt. Bedded salt formations tend to have elevated impurities thereby increasing brine content from a few thousandth to several weight percent (Finley et al., 1992). Domal formations nominally have less brine (0.01 to 0.1 wt%) (Roedder and Bassett, 1981) and minor amounts of impurities. Natural water content is vital for repository applications because small amounts of available moisture promote solution precipitation processes, which Urai and Spiers (2007) show reduce dislocation densities, influence grain boundary migration, and can lead to recrystallization. Fluid-assisted creep (pressure solution) comprises transfer of fluid along grain boundaries where portions of highly stressed grains dissolve owing to increased solubility and precipitate at lower stress interfaces. Fluid-assisted migration is capable of removing stored energy even at ambient temperatures and noticeably accelerates densification of granular salt (Spiers et al., 1990; Callahan et al., 1996, 1998).

Annealing/recrystallization is a thermally-activated recovery process that involves strain softening when work-hardening processes are unbalanced. Annealing/recrystallization has been observed in rock salt deformed naturally and in cores deformed experimentally (Spiers and Carter, 1998; Peach et al., 2001; Schléder and Urai, 2005; Desbois et al., 2010). Only a handful of studies observed recrystallization in experiments on consolidated granular rock salt (Carter et al., 1982; Hansen et al., 2012). This is perhaps because of limited test duration and conditions (e.g., temperature, added moisture, etc.), and/or excluded observational work altogether. The aforementioned investigations provide examples of substructural observations and procedures which are employed here.

1.2. Previous studies on consolidation

Laboratory consolidation of natural granular rock salt has been widely studied (Munson and Dawson, 1984; Holcomb and Shields, 1987; Case et al., 1987; Brodsky, 1994; Hunsche and Hampel, 1999; Stührenberg and Schulze, 2012; Bérest, 2013; Bauer et al., 2015). Previous studies conclude deformation mechanisms and consolidation rate depend on temperature, stress, and moisture availability. Without available moisture, salt consolidation at room temperature requires greater stress as void space reduces by cataclastic fractures, grain boundary sliding, and minimal crystallographic distortion via glide mechanisms, in order to achieve significant consolidation. At increased temperatures, extensive glide enables grain deformation accompanied by thermally-activated climb recovery, as observed in samples by Broome et al. (2014) where dried granular salt was consolidated at temperatures to 250 °C and 20 MPa confining pressure. Broome et al. (2014) also observed internal fluid inclusions migrating toward grain boundaries, promoted by elevated temperature, where consolidation continued by plasticity-aided pressure solution processes.

Microscopic examination of pressure solution processes has primarily been investigated by consolidation or compaction experiments on synthetic salt, such as table salt or analytical grade NaCl granulates, as an analogue for natural granular rock salt (Spiers and Brzesowsky, 1993; Pennock et al., 2007; Schutjens and Spiers, 1999; Ter Heege et al., 2005). Added moisture in synthetic salt enables rapid recrystallization at grain boundaries from fluid-assisted creep (Urai and Spiers, 2007); however, there have been few (e.g., Urai et al., 1986b; Brodsky et al., 1996) microstructural observations on consolidated natural granular rock salt with an amount of water added consistent with potential repository applications (i.e. not saturated). Spiers and Brzesowsky (1993) investigated dry and brine-saturated NaCl granulates after uniaxial compaction to 8 MPa and ambient temperature, resulting in porosities between 17% and 30%. They observed clear evidence of densification by pressure solution processes and lower porosities in brine-saturated samples compared to non-saturated samples. After rapid oedometer compaction (rates of 10^{-5}–10^{-6} s^{-1}) of dry synthetic NaCl powder (grain size 100 to 600 μm) at room temperature to 5% porosity, Pennock et al. (2007) investigated etched impregnated thin sections under SEM electron backscattered diffraction. They found a blend of strain free and deformed grain substructures, indicating several mechanisms had occurred including (unexpectedly) recrystallization by fluid-assisted grain boundary migration. The given explanation for fluid in the system was from small amounts of water vapor in laboratory air used to flush samples during compaction. Schutjens and Spiers (1999) examined by optical microscopy intergranular pressure solution at grain contacts of synthetic halite crystals with applied unconfined effective normal stresses to 7 MPa on oriented single crystals in the presence of saturated brine. They observed time-dependent dissolution of material at the contact of grains without plastic or brittle deformation, concluding intergranular pressure solution was the driving mechanism for healing. Although the material and conditions used in their investigations of pressure solution processes are not directly applicable to anticipated field conditions, they confirm the importance of pressure solution processes to consolidation of salt.

While many experiments have been conducted on consolidating granular salt, these studies often excluded microstructural observations, used synthetic salt, omitted supplementary added moisture conditions, and/or had limited temperature, pressure, and time ranges. Therefore, this study aims to provide an integrated experimental and investigative research of natural granular salt consolidation by introducing moisture under variable temperatures, stresses, and durations. A principal objective of this work is to document the relationship between deformation mechanisms and resulting consolidation over a range of conditions, producing an array of end state porosities of less than 10%. Profound effects of adding moisture are emphasized because pressure solution processes provide potential to affect functional sealing conditions by enhanced densification and lower permeability.

2. Material and methods

2.1. Consolidation experiments

Granular salt from mining operations at WIPP, located in a bedded salt formation, and the Avery Island mine, located in a domal salt

Table 1
Consolidated samples with associated conditions. Samples not pre-conditioned had a starting porosity between 60% and 70%. W = WIPP, A = Avery Island.

Sample name	Temperature (°C)	Hydrostatic stress (MPa)	Moisture added	Elapsed consolidation time (days)	Pre-conditioned	Core porosity (%)	Point counting porosity (%)
W90-1	90	20	None	111	No	8.0	8.33
W90-2	90	20	1%	16	No	2.0	1.89
W90-4	90	20	1%	5	Yes	3.5	4.45
W90-7	90	20	1%	21	Yes	2.0	2.67
W90-8	90	38	None	3	Yes	3.6	3.44
W175-1	175	20	None	1	Yes	5.5	4.55
W250-1	250	20	None	14	No	1.0	1.55
W250-2	250	20	None, Unvented	12	No	1.0	1.67
A250-1	250	20	None	7	No	1.0	1.78
A250-2	250	20	1%	7	No	1.0	0.78

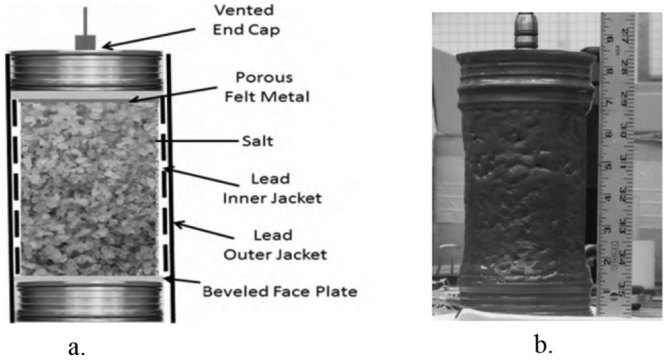

Fig. 1. a. Example of sample assemblage for consolidation. b. Sample after consolidation.

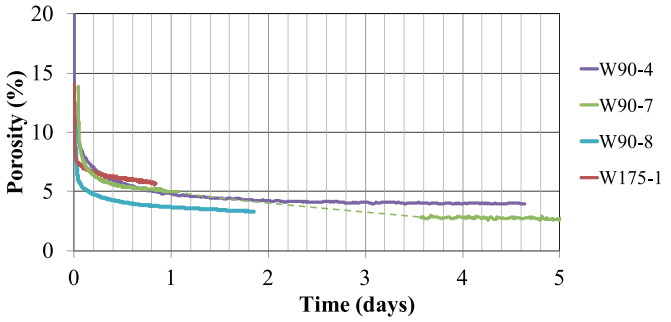

Fig. 2. Porosity vs time for pre-conditioned samples at different moisture, stress, and temperature conditions and time durations. Test conditions are given in Table 1.

formation, was consolidated in laboratory hydrostatic creep tests with temperatures to 250 °C and confining pressures to 38 MPa, to produce deformation. The test matrix is shown in Table 1. Granular salt was screened to exclude particles larger than 9.5 mm, while further sieving yielded a well-graded distribution with about 60% finer than 2.8 mm (Paneru, 2016), and dried at 110 °C. To produce material for wetted samples, granular salt was spread in a single layer and exposed to 100% humidity until 1% moisture content by weight was achieved. Cylindrical samples, as shown in Fig. 1, nominally 105 mm diameter and 122 mm tall, were created by placing the prepared granular salt into copper and malleable soldered lead tubes fitted with end caps.

In early testing, difficulties arose in measuring large and rapid volumetric strains during the initial portion of the consolidation testing, which necessitated development of a pre-conditioning stage. For the pre-conditioning stage, jacketed samples, which had an initial porosity between 30% and 40%, were placed in a pressure vessel within a load frame and loaded to hydrostatic pressures of 19 to 38 MPa for 10–15 min at ambient temperature. Pre-conditioning produced samples with a porosity of about 20%. Six of the ten samples in this study did not undergo pre-conditioning, detailed in Table 1. During testing, band heaters on the pressure vessel were used to achieve and maintain test temperature. Hydrostatic stresses were generated by pressurizing hydraulic oil in the pressure vessel. Principal strains during consolidation were determined from axial displacement LVDT's (linear variable differential transformer), lateral displacements with Schuler gages (Schuler, 1978), with additional volume strain measured from confining fluid changes. A more complete description of consolidation tests can be found in Bauer et al. (2015) and Broome et al. (2014). Test durations ranged from a few days to a few months. Final porosities were below 10%, determined by post-test gas porosimetry measurements on cores and point counting impregnated thin sections, which are agreeable (see Table 1).

A graph of porosity response during testing over five days compares four pre-conditioned samples under different conditions is shown in Fig. 2. A superimposed dashed line for W90-7 was generated due to interrupted data logging. Curves are becoming more or less straight lines, which may be extrapolated to a final state where complete compaction is reached. At 175 °C, W175-1, consolidated for a short duration, did not achieve porosity as low as those with added moisture, higher stress, and/or longer durations. At higher stress (38 MPa), W90-8 reduces porosity initially, but added moisture samples (W90-4 and W90-7, subject to 20 MPa hydrostatic pressure) develop a similar or lower porosity, given a longer duration.

2.2. Sub-sample preparation and observational techniques

A suite of observational techniques utilized multiple polished thick petrographic sections and freshly broken surfaces as shown in Fig. 3. First, ends of consolidated samples were cut using a low-damage diamond wire saw to create sub-samples. A number of sub-samples were commercially vacuum impregnated with blue stained RF 1366 resin and thin sectioned, while others were vacuum impregnated with rhodamine-B doped Spurr Low-Viscosity Embedding Kit and cut using a Buehler IsoMet™ low-speed saw. Fresh aggregate fragments were carefully broken from sub-samples by hand to expose clean surfaces that reveal sample cohesiveness, grain boundary characteristics, and other evidence of consolidation processes.

Observational devices included optical and scanning electron microscopes (SEM). A Leitz Ortholux II optical microscope equipped with a Leica Microsystems camera and Leica Application Suite software was used to examine unetched/etched thin sections, point count, and capture images. Two SEM's, a FEI Nova 200 Nanolab and Tescan Vega3 LM, were used to view etched thin sections and freshly broken aggregate surfaces. Etching highlights grain microstructure and is completed by agitating a specimen in a solution of methanol saturated with $PbCl_2$ and stopped by transferring to butanol to discontinue the etch.

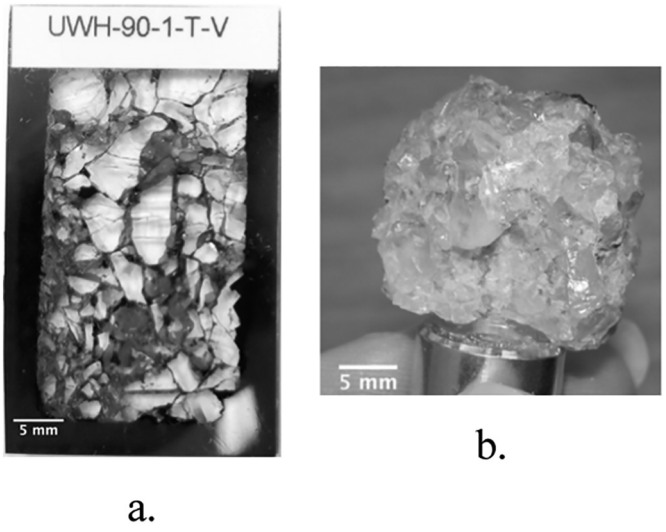

Fig. 3. Sample types examined a. Thick-thin section of standard dimensions. b. Broken aggregate.

3. Results

3.1. Plastic mechanisms and moisture aided processes

Without addition of moisture, samples at temperatures of 90 °C and 175 °C consolidated by processes of mechanical abrasion and minor plastic deformation, as captured in photomicrographs of broken aggregate surfaces presented in Fig. 4a–c. These samples retain visible pore space, unhealed grain boundaries, and mechanical rearrangement processes. Abraded surfaces, shown prominently in W90-8 (4b.), signify grinding along boundaries, as grains shear past one another. Emergent and distorted {110} planes are indicative of glide deformation as interpreted by circled areas of samples W90-1 and W175-1 (Fig. 4a and c). In W90-1 (Fig. 4a), curved planes are discernable in the upper portion near the grain boundary, an example of cataclastic and plastic processes of consolidation. Linear planes of the original crystal structure are seen in the lower portion of the image. Plastic deformation is also displayed in Fig. 4c., with the lower grain impinging against the upper grain, distorting {110} dodecahedron planes within the upper grain (see annotations). Substructure of W90-8, the only sample that experienced 38 MPa was directly compared to W90-1, which consolidated at 20 MPa for a longer time. While the end state porosity of W90-8 was 5% less than W90-1, no significant difference in their microstructure could be seen. With respect to only varying stress conditions, no evident differences were observed in consolidation processes.

Samples with added moisture or unvented conditions were observed to have cohered or healed grain boundaries, facilitated by pressure solution processes, and occluded fluid pore spaces along grain boundaries. These features can be seen throughout images in Fig. 5, in which pressure solution processes produced tight grain boundaries, which promote tensile breaks along crystallographic planes. Cleaved surfaces are created by breaking through the crystal structure rather than along grain boundaries, signifying a high level of healing and cohesion compared to abraded surfaces of samples without added moisture in Fig. 4. Pressure solution processes are rampant as evidenced by ubiquitous rounded cubic grain features (Fig. 5a–c).

Isolated and occluded fluid pore spaces were widespread along grain boundaries (noted with arrows) among the 1% added moisture and unvented samples. The array of these inhomogeneous features, most likely formed by the breakdown of brine films during deformation or by means of additional solution-precipitation processes from added moisture, are displayed prominently in Fig. 5d–h. Between magnifications of 500× to 1,500×, arrays of droplets and canals of residual moisture are apparent along grain boundaries and surfaces. The unvented sample, W250-2 (Fig. 5g and h), retained all natural moisture, therefore altering the morphology of remnant isolated and occluded fluid pore space while promoting fluid aided processes and achieving a high degree of consolidation. Evidently, pore pressure does not inhibit consolidation because sample W250-2 consolidated to extremely low porosity. A vented equivalent sample not shown, W250-1, also experienced a high degree of consolidation evident by tight grain boundaries and cleaved fracture surfaces. Moisture present as intragranular and impurity fluid vented from this sample during consolidation. These results suggest that consolidation at 250 °C achieves low porosities regardless of whether or not there was additional moisture.

3.2. Recrystallization

At temperatures of 250 °C, recrystallization (possibly both dynamic and fluid-assisted) was evident in etched thin sections as seen under the optical microscope in Fig. 6b–e. A grain showing polygonized subgrains from a sample consolidated at 90 °C with added moisture (W90-2) is displayed in Fig. 6a for comparison. It is important to note that all other samples consolidated with or without added moisture at 90 °C and even at a slightly elevated temperature of 175 °C had virtually no evidence of recrystallization. All samples consolidated at 250 °C exhibited zones of recrystallization, marked with X's in optical photomicrographs in Fig. 6b–e. Recrystallization fronts with no internal structure demonstrate internal grain recovery, and are prevalent in several grains of the 250 °C samples, surrounded by internal subgrain structures. Even though domal salt has a low initial brine content (0.01 to 0.1 wt%), the Avery Island sample without added moisture (A250-1) still experienced recrystallization, which indicates only thermally activated recovery processes were experienced.

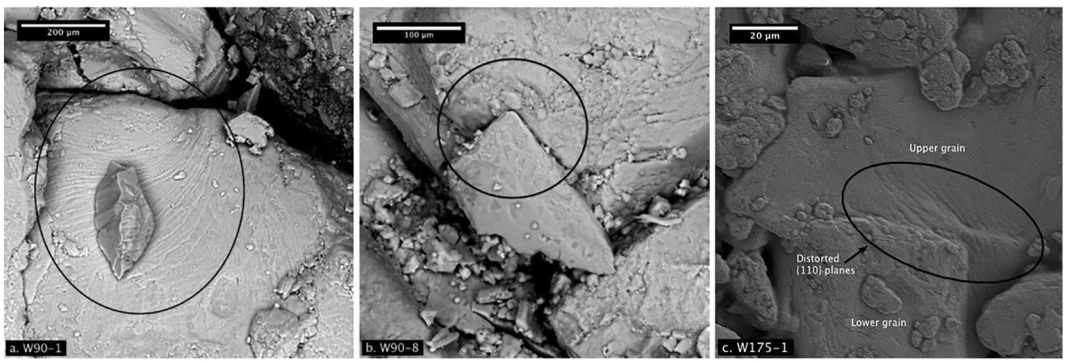

Fig. 4. SEM photomicrographs of mechanically broken aggregate pieces without added moisture. a. W90-1: Curved {110} planes experiencing minor glide along top portion of center grain while adjacent to linear {110} planes in lower grain (250×). b. W90-8: Grain boundaries lacking cohesion and rough, abraded surfaces (500×). c. W175-1: Plastic deformation at grain contact and open pore space (1,300×).

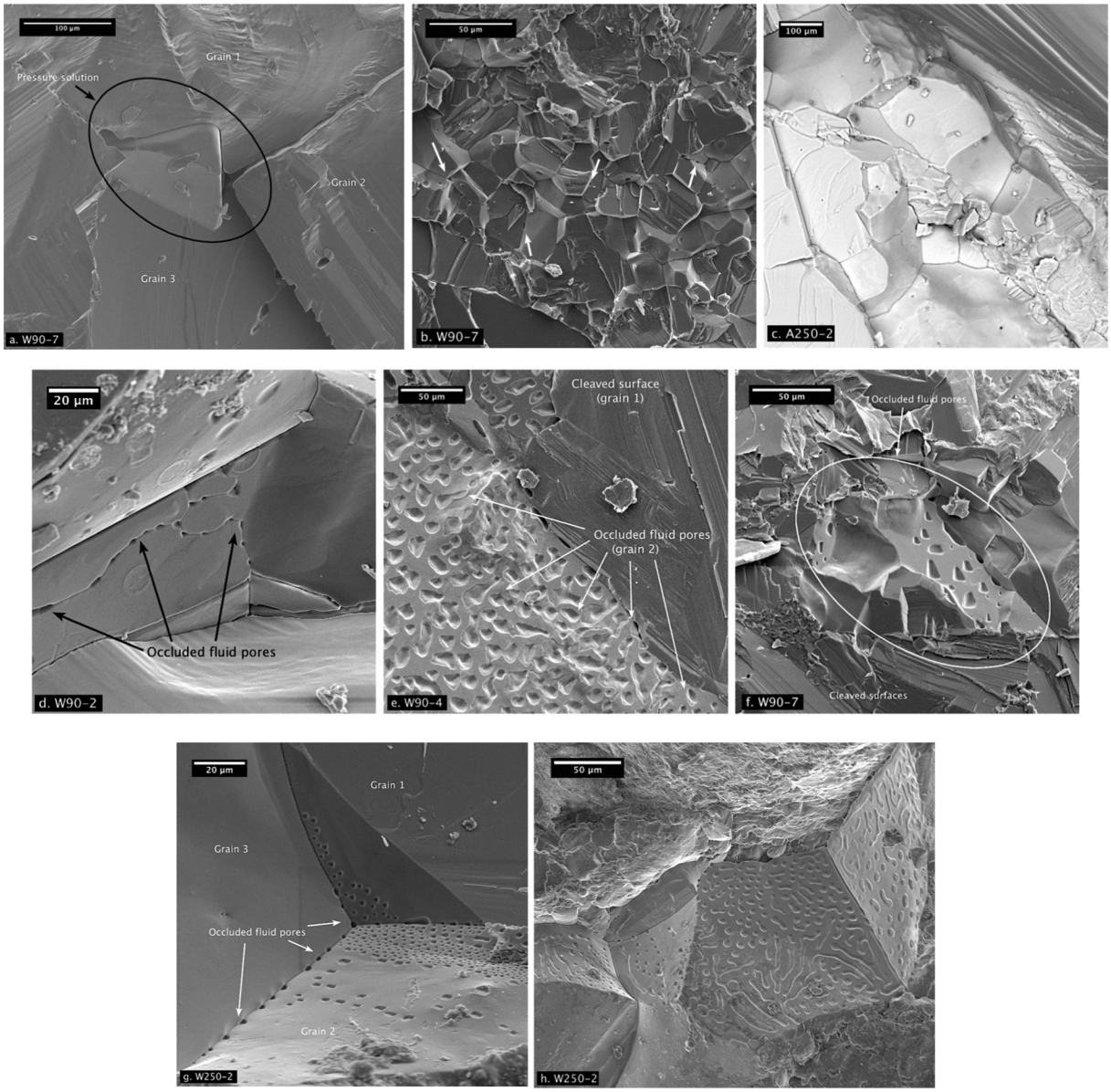

Fig. 5. Pressure solution processes and occluded fluid pore space in freshly broken aggregate surfaces of samples with 1% added moisture under SEM. a. W90-7: Highly deformed grain at center by pressure solution, surrounded by distorted grains from glide on {110} (650×). b. W90-7: Ubiquitous pressure solution processes indicated by nearly all eliminated void space producing tightly cohered grain boundaries (1,000×). c. A250-2: Rounded cubic grains encompassed by cleaved surfaces (250×). d. W90-2: Occluded fluid pores at center along grain boundaries and crystallographic cleaved surfaces (1,000×). e. W90-4: Array of fluid canals and pore space imprinted on grain boundary surface (750×). f. W90-7: Area of occluded fluid pore spaces at center surrounded by cleaved faces (950×). g. W250-2: Tight triple junction with canals of residual moisture displaying occluded pores (1,500×). h. W250-2: Occluded fluid droplets and canals on a planar grain boundary (500×).

3.3. Discussion

The results presented in this study explore and improve the understanding of micromechanic processes in granular salt associated with thermal, mechanical, and hydrologic conditions during consolidation. The photomicrographs provide direct observations of multiple deformation mechanisms, specifically when moisture is present and/or temperatures are elevated to 250 °C. A number of deformation mechanisms were identified that are consistent with those seen in previous research on salt such as plastic behavior, pressure solution processes, and recrystallization (e.g. Urai et al., 1986a,b; Spiers and Brzesowsky, 1993; Desbois et al., 2010; Hansen et al., 2012, 2015; Broome et al., 2014).

The deformation processes observed here follow the sequence given in Hansen et al. (2015), shown in Fig. 7, where acting deformation mechanisms related to stress, moisture, and time induced (creep) effects exist in series and parallel. An increase in stress induces plastic deformation, but without abundant moisture and significantly elevated temperatures, only grain rearrangement and cataclastic processes are produced (see substructures of W90-1, W90-8, and W175-1 in Fig. 4) (Carter et al., 1982; Senseny et al., 1992; Hunsche and Hampel, 1999).

Only minimal consolidation testing along with observational work has been conducted at elevated temperatures, which is especially important for disposal concepts of heat-generating waste. Increase of temperature elevates the diffusion rate and dislocation creep mechanisms, thus reducing the flow strength and increasing the compaction rate (Ter Heege et al., 2005; Urai et al., 2008). The effects of temperature in Fig. 6 agree with results from the few past experiments

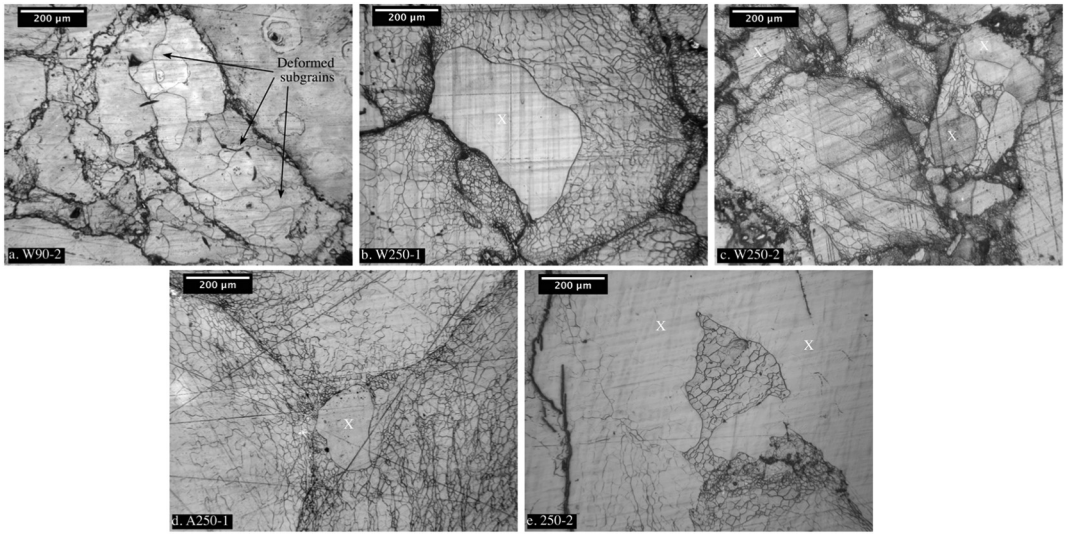

Fig. 6. Etched thin section images under reflected and transmitted light microscope. a. W90-2: Center grain with deformed subgrain structure indicating polygonization (10×). b. W250-1: Large recrystallized area at center as well as high-energy grain boundaries (10×). c. W250-2: Simultaneous recrystallization, internal grain recovery, and high energy grain boundaries (10×). d. A250-1: Triple-junction grain boundary with recrystallized area in center and small subgrains decorating tight grain boundaries (10×). e. A250-2: Congested substructure of tangled dislocation pile-up at center, inferred as possessing more internal strain energy than surrounding areas of nearly complete recrystallization (10×).

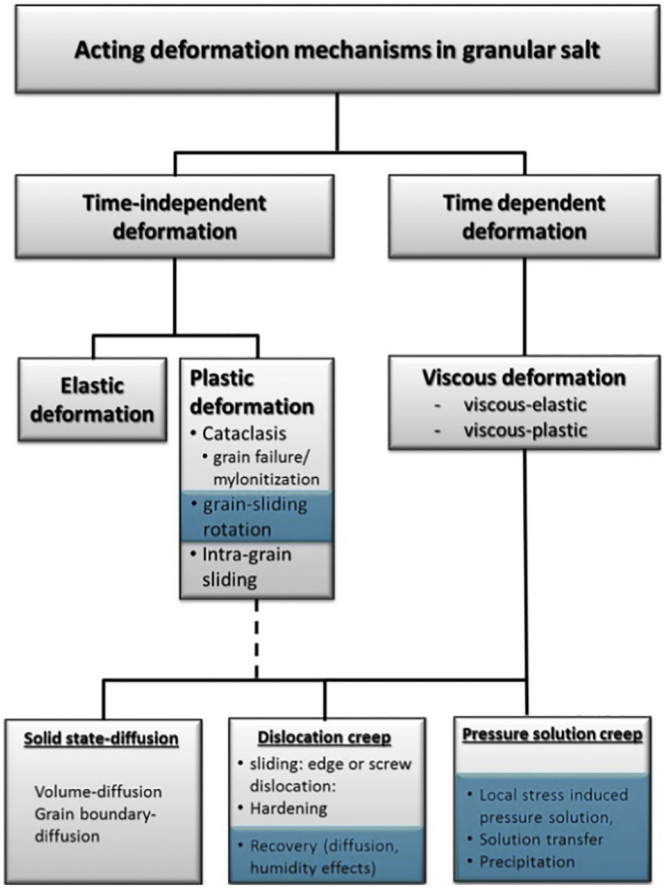

Fig. 7. Sequence of deformation mechanisms in granular salt from Hansen et al. (2015), where moisture effects are given in blue. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

conducted (Urai et al., 1986b; Hansen et al., 2012; Broome et al., 2014), where elevated temperatures enable considerable grain deformation. Results are consistent with the concept that fluid, when present along grain boundaries and liberated from inclusions, increases migration and promotes recrystallization (Urai and Spiers, 2007). Similar features of recrystallization on deformed Asse mine core samples were observed in Urai et al. (1986b), with conditions of 150 °C, 10 MPa, and an unknown amount of added brine. However, observations of recrystallization in granular salt consolidated at 250 °C has not been presented in previous research. There is a possibility added moisture at 175 °C may also promote recrystallization, but this was not included in this study and could be further investigated. It is difficult to precisely distinguish the dominating mechanism within the 250 °C samples since increasing temperature enhances dislocation creep, liberating fluid inclusions and moisture from impurities, thereby also introducing pressure solution processes to aid in deformation.

We found the presence of water in the microstructure of salt aggregates to be the most significant condition to consolidation, being beneficial at low and elevated temperatures. Moisture facilitates the diffusion controlled deformation mechanisms (i.e., pressure solution processes), again significantly reducing the flow strength of the salt (Peach et al., 2001; Ter Heege et al., 2005), and therefore, amplifying compaction of crushed salt. A limit exists for amount of added moisture and its effectiveness, but additional experiments should be conducted. Effects of moisture presented in Fig. 5 are consistent with previous theories and studies on synthetic salt (Spiers and Brzesowsky, 1993; Pennock et al., 2007; Schutjens and Spiers, 1999), but the current photomicrographs add to the minimal observations made on consolidated natural granular salt. Residual fluid arrays were also found on deformed intact Asse mine core samples by Urai et al. (1986b) and on WIPP salt with 1% added moisture by Brodsky et al. (1996). Spiers et al. (1990) suggests that at lower stress ranges, pressure solution and recrystallization are significant, and further investigation is needed, as moisture availability (i.e. humidity) can affect a wide range of repository conditions.

4. Conclusions

Micromechanics of eleven laboratory consolidated granular salt samples were characterized using various observational methods to help understand effects of stress, temperature, and moisture conditions. The altering of these variables has a direct effect on deformation mechanisms leading to different end state conditions. Results confirm the

importance of relatively small amounts of moisture on consolidation. With only 1% added moisture or moisture available in an unvented sample, pressure solution-redeposition effectively enhanced consolidation, while producing occluded fluid pore spaces and a degree of cohesion along grain boundaries equal to the crystallographic tensile strength.

Samples consolidated without added moisture at 90 °C (W90-1 and W90-8) and 175 °C (W175-1) show no evidence of pressure solution processes, only plastic deformation. Effects of increasing confining pressure were not apparent in sample morphology within the microstructural observations when considering W90-1 (20 MPa) and W90-8 (38 MPa). Occluded fluid pore spaces found along grain boundary surfaces and intersections in the unvented and added moisture samples were unexpected features since documentation is scarce in literature. The 250 °C samples developed a high degree of cohesion along grain boundaries, regardless of moisture conditions, compared to samples consolidated at lower temperatures. Both W250-1 (vented) and W250-2 (unvented) consolidated to extremely low porosity in a short duration (days), an intriguing result, suggesting that consolidation processes were not impeded by pore pressure. This could be further investigated with more unvented tests. At elevated temperatures, the difference between the two salt types only appeared to be residual fluid features, as both consolidated to low porosities. Observations of recrystallization in etched thin sections consolidated at temperatures of 250 °C was compelling and significant, since rock salt recrystallization is most often observed in laboratory consolidation of synthetic salt and naturally deformed salt deposits. The observations imply a replication of natural process in the experiments.

In order to reliably predict the behavior of granular salt consolidation for the purpose of barrier and seal functions, a better understanding of micromechanical processes is necessary to form more complete constitutive models. The number and range of conditions accompanied with this testing are unique, along with the techniques used to display microstructural features, and provide some insight into an important area of unknown governing deformation mechanism(s). Still, additional experimental testing incorporating multiple conditions to achieve low end-state porosities is required to further enhance the knowledge base of micromechanical processes and generate greater confidence in the application of granular salt as backfill in repository settings.

Acknowledgements

We thank the Geomechanics testing laboratory at Sandia National Laboratories for use of testing equipment, data collection instruments, and resources. We extend our utmost gratitude for Francis Hansen for providing his excellent knowledge, experience, and skills dedicated to salt sciences. We also thank Brandon Lampe, Timothy Lynn, and Laxmi Paneru for their diligent efforts in producing useful samples for investigation. This material is based upon work supported under an Integrated University Program Graduate Fellowship and research was performed using funding received from the DOE Office of Nuclear Energy's Nuclear Energy University Program under Grant number 13-4834. The views expressed in the article do not necessarily represent the views of the U.S. Department of Energy or the United States Government. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration. With main facilities in Albuquerque, N.M., and Livermore, C.A., Sandia has major R&D responsibilities in national security, energy and environmental technologies, and economic competitiveness. SAND2018-3372J

References

Bauer, S.J., Broome, S.T., Hansen, F.D., Lampe, B., Mills, M.M., Stormont, J.C., 2015. Gas flow measurements of consolidating crushed salt. In: 49th US Rock Mechanics/Geomechanics Symposium, San Francisco, California.

Bérest, P., 2013. Mechanical behavior of salt and salt caverns. In: Kwaśniewski, M., Łydżba, D. (Eds.), Rock Mechanics for Resources, Energy, and Environment. Taylor & Francis Group, London, pp. 17–29.

Brodsky, N.S., 1994. Hydrostatic and shear consolidation tests with permeability measurements on Waste Isolation Pilot Plant crushed salt. In: SAND93-7058. Sandia National Laboratories, Albuquerque, New Mexico.

Brodsky, N.S., Hansen, F.D., Pfeifle, T.W., 1996. Properties of dynamically compacted WIPP salt. In: SAND96-0838C. Sandia National Laboratories, Albuquerque, New Mexico.

Broome, S.T., Bauer, S.J., Hansen, F.D., 2014. Reconsolidation of crushed salt to 250 °C under hydrostatic and shear stress conditions. 48th US Rock Mechanics/Geomechanics Symposium, Minneapolis, Minnesota.

Callahan, G.D., Loken, M.C., Hurtado, L.D., Hansen, F.D., 1996. Evaluation of constitutive models for crushed salt. In: SAND96-0791C. Sandia National Laboratories, Albuquerque, New Mexico.

Callahan, G.D., Mellegard, K.D., Hansen, F.D., 1998. Constitutive behavior of reconsolidating crushed salt. Int. J. Rock Mech. Min. Sci. 35 (4–5).

Carter, N.L., Heard, H.L., 1970. Temperature and rate dependent deformation of halite. Am. J. Sci. 269, 193–249.

Carter, N.L., Hansen, F.D., Senseny, P.E., 1982. Stress magnitudes in natural rock salt. J. Geophys. Res. 87 (B11), 9289–9300.

Case, J.B., Kelsall, P.C., Withiam, J.L., 1987. Laboratory investigation of crushed salt consolidation. In: 28th US Symposium on Rock Mechanics, Tucson, Arizona.

Desbois, G., Závada, P., Schléder, Z., Urai, J., 2010. Deformation and recrystallization mechanisms in actively extruding salt fountain: microstructural evidence for a switch in deformation mechanisms with increased availability of meteoric water and decreased grain size (Qum Kuh, central Iran). J. Struct. Geo. 32, 580–594.

Finley, S.J., Hanson, D.J., Parsons, R., 1992. Small-scale brine inflow experiments – data report through 6/6/91. In: SAND91-1856. Sandia National Laboratories, Albuquerque, New Mexico.

Fuenkajorn, K., Daemen, J.J.K., 1996. Sealing of boreholes and underground excavations in rock. Chapman & Hall, London.

Hansen, F.D., 2014. Micromechanics of isochoric salt deformation. In: ARMA 14-7012. 48th US Rock Mechanics/Geomechanics Symposium, Minneapolis, Minnesota.

Hansen, F.D., Bauer, S.J., Broome, S.T., Callahan, G.D., 2012. Coupled thermal-hydrological-mechanical processes in salt: hot granular salt consolidation, constitutive model and micromechanics. In: FCRD-UFD-2012-000422, SAND2012-9893P. Sandia National Laboratories, Albuquerque, New Mexico.

Hansen, F.D., Popp, T., Wieczorek, K., Stührenberg, D., 2015. Salt reconsolidation applied to repository seals. In: Roberts, L., Mellegard, K., Hansen, F. (Eds.), The Mechanical Behaviour of Salt VIII, Rapid City, South Dakota. Taylor & Francis Group, London, pp. 179–189.

Holcomb, D.J., Shields, M., 1987. Hydrostatic creep consolidation of crushed salt with added water. In: SAND87-1990. Sandia National Laboratories, Albuquerque, New Mexico.

Hunsche, U., Hampel, A., 1999. Rock salt – The mechanical properties of the host rock material for a radioactive waste repository. Eng. Geol. 52 (3), 271–291.

Kröhn, K.P., Zhang, C.L., Czaikowski, O., Stührenberg, D., Heemann, U., 2015. The compaction behaviour of salt backfill as a THM-process. In: Roberts, L., Mellegard, K., Hansen, F. (Eds.), The Mechanical Behaviour of Salt VIII, Rapid City, South Dakota. Taylor & Francis Group, London, pp. 49–59.

Munson, D.E., Dawson, P.R., 1984. Salt constitutive modeling using mechanism maps. In: Hardy, H., Langer, M. (Eds.), Proc. 1st Conf. Mech. Beh. of Salt, Clausthal-Zellerfeld, Germany. Trans Tech Pub, pp. 717–737.

National Research Council, 1957. The disposal of radioactive waste on land. In: The National Academies Press, Washington, DC.

NEA Salt Club, 2015. https://www.oecd-nea.org/rwm/saltclub/.

Paneru, L.P., 2016. Measurement of thermal properties and porosity of consolidated salt. http://digitalrepository.unm.edu/ce_etds/127.

Peach, C.J., Spiers, C.J., Trimby, P.W., 2001. Effect of confining pressure on dilatation, recrystallization, and flow of rock salt at 150 °C. J. Geophys. Res. 106 (B7), 13,315–13,328.

Pennock, G.M., Zhang, X., Peach, C.J., Spiers, C.J., 2007. Microstructural study of reconsolidated salt. In: Wallner, M., Lux, K.H., Minkley, W., Hardy, H.R.J. (Eds.), The Mechanical Behavior of Salt – Understanding of THMC Processes in Salt: Proceedings of the 6th Conference (SaltMech6), Hannover, Germany. Taylor & Francis Group, London, pp. 149–158.

Roedder, E., Bassett, R.L., 1981. Problems in determination of the water content of rock-salt samples and its significance in nulcear-waste storage siting. Geology 9, 525–530.

Schléder, Z., Urai, J.L., 2005. Microstructural evolution of deformation- modified primary halite from Hengelo, the Netherlands. Int. J. Earth Sci. 94 (5–6), 941–956.

Schuler, K.W., 1978. Lateral-deformation gage for rock-mechanics testing. Exp. Mech. 18 (12), 477–480.

Schutjens, P.M.T.M., Spiers, C.J., 1999. Intergranular pressure solution in NaCl: grain-to-grain contact experiments under the optical microscope. Oil Gas Sci. Tech. 54 (6), 729–750.

Senseny, P.E., Hansen, F.D., Russell, J.E., Carter, N.L., Handin, J.W., 1992. Mechanical behavior of rock salt: phenomenology and micromechanisms. Int. J. Rock Mech. Min. Sci. & Geomech. 29 (4), 363–378.

Skrotzki, W., Haasen, P., 1984. Role of cross slip in the steady-state creep of salt. In:

Hardy, H.R.J., Langer, M. (Eds.), Proceedings of the 2nd Conference of the Mechanical Behavior of Salt, Hannover, Germany. Tran Tech, Aedermannsdorf, Switzerland, pp. 66–81.
Spiers, C.J., Brzesowsky, R.H., 1993. Densification behavior of wet granular salt: theory versus experiment. In: Seventh Symposium on Salt. Vol. I. pp. 83–92.
Spiers, C.J., Carter, N., 1998. Microphysics of rocksalt flow in nature. In: Aubertin, M., Hardy Jr.H.R. (Eds.), The Mechanical Behaviour of Salt IV. Trans. Tech., Clausthal-Zellerfeld, Germany, pp. 115–128.
Spiers, C.J., Schutjens, P.M.T.M., Brzesowsky, R.H., Peach, C.H., 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, London, pp. 215–227 Spec. Publ. 54.
Stührenberg, D., 2007. Long-term laboratory investigation on backfill. In: Wallner, M., Lux, K.H., Minkley, W., Hardy, H.R.J. (Eds.), The Mechanical Behavior of Salt – Understanding of THMC Processes in Salt: Proceedings of the 6th Conference (SaltMech6), Hannover, Germany. Taylor & Francis Group, London, pp. 223–229.
Stührenberg, D., Schulze, O., 2012. Porosity and permeability of crushed and damaged rock salt during compaction. In: Bérest, P., Ghoreychi, M., Hadj-Hassen, F., Tijani, M. (Eds.), Mechanical Behavior of Salt VII. Taylor & Francis Group, London, pp. 275–282.
Ter Heege, J.H., De Bresser, J.H.P., Spiers, C.J., 2005. Dynamic recrystallization of wet synthetic polycrystalline halite: dependence of grain size distribution on flow stress, temperature and strain. Tectonophysics 396 (1–2), 35–57.
Urai, J.L., Spiers, C.J., 2007. The effect of grain boundary water on deformation mechanisms and rheology of rocksalt during long-term deformation. In: Wallner, M., Lux, K.H., Minkley, W., Hardy, H.R.J. (Eds.), The Mechanical Behavior of Salt – Understanding of THMC Processes in Salt: Proceedings of the 6th Conference (SaltMech6), Hannover, Germany. Taylor & Francis Group, London, pp. 149–158.
Urai, J.L., Means, W.D., Lister, G.S., 1986a. Dynamic recrystallization of minerals. In: Hobbs, B.E., Heard, H.C. (Eds.), Mineral and Rock Deformation: Laboratory Studies–The Paterson Volume. vol. 36. pp. 161–199 AGU Geophysical Monograph.
Urai, J.L., Spiers, C., Zwart, H., Lister, G., 1986b. Weakening of rock salt by water during long-term creep. Nature 324, 554–557.
Urai, J.L., Schléder, Z., Spiers, C.J., Kukla, P.A., 2008. Flow and transport properties of salt rocks. In: Littke, R., Bayer, U., Gajewski, D., Nelskamp, S. (Eds.), Dynamics of Complex Intracontinental Basins: The Central European Basin System. Springer-Verlag, Berlin, Heidelberg, pp. 277–290.