

Mechanical Behavior and Microstructure Development in Consolidation of Nominally Dry Granular Salt

Ding, J.¹, Chester, F.M.¹, Chester, J.S.¹, Zhu, C.² and Arson, C.²

¹*Center for Tectonophysics, Department of Geology and Geophysics, Texas A&M University, College Station, Texas*

²*School of Civil and Environmental Engineering, Georgia Institute of Technology, Atlanta, Georgia*

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ABSTRACT: Uniaxial consolidation of granular salt is carried out to study the mechanical behavior and fabric development in a material that deforms by microscopic brittle and intracrystalline-plastic processes. Dry granular salt is sieved to produce well-sorted size fractions. The granular salt is consolidated in a heated cell at axial stresses up to 90 MPa and temperatures of 100 - 200 °C to document stress-consolidation relationships and microstructural development. Polished and chemically-etched petrographic sections of salt samples prior to and after deformation at 150 °C are studied using transmitted- and reflected-light optical microscopy. We show that temperature has profound effect on porosity reduction during consolidation. At tested conditions, the dominant deformation mechanism is crystal plasticity; brittle deformation is largely suppressed. Samples consolidated at higher maximum axial stress develop higher overall dislocation densities. The distribution of dislocations, however, is strongly heterogeneous from grain to grain because of the complex grain-scale loading geometries and the distribution of intragranular flaws such as fluid inclusions. Static recrystallization occurs in some highly strained areas, but overall is minor at 150 °C. The experiments help to improve our understanding of consolidation, and serve to guide the fabrication of synthetic rock salt as experimental material, as well as to inform and test constitutive models of deformation of granular salt for engineering needs.

1. INTRODUCTION

The rheological properties of rock salt have been an important research focus because it is considered a viable geomaterial for engineered repositories of waste and energy resources, such as radioactive waste, oil, and gas (Urai et al., 1986; Carter et al., 1993). Rock salt also is studied as an analog to other geomaterials in that various deformation mechanisms, including cracking, frictional sliding, pressure solution, crystal plasticity, and dynamic recrystallization are easily activated at laboratory conditions (Zhang et al., 2007).

Extensive experimental work has been done on rock salt to determine constitutive relationships and guide numerical modeling (e.g., Watanabe and Peach, 2002; Ter Heege, et al., 2005; Zhu and Arson, 2015). Both natural and synthetic rock salt have been used for experimental investigations; artificially prepared rock salt is of higher purity and can be fabricated in a way that best serves parametric studies (e.g., Carter and Hansen, 1983; Schenk and Urai, 2004; Bourcier et al., 2013). Consolidation of granular salt is a common method used to produce synthetic rock salt samples; however, salt is highly sensitive to moisture so it is

critical to have a good control of the humidity of the environment in which nominally-dry salt samples are produced, stored, and processed. Maintaining a consistently dry environment during all stages of handling salt samples can be challenging.

We developed an experimental procedure that allows preparation of nominally dry synthetic rock salt via one-dimensional consolidation and subsequent characterization of the microstructure of the rock salt, using a well-controlled low humidity chamber. In this paper, we document the consolidation conditions used to create synthetic rock salt samples with different porosities, and document the mechanical behavior and deformation mechanism during consolidation. In addition, we illustrate the reproducibility in sample production. Knowledge gained from this work will be used to fabricate suitable samples for future deformation experiments and guide numerical modeling of granular salt consolidation.

2. EXPERIMENTS

2.1. Starting material

Two types of granular salt are used in our consolidation experiments, sea salt and reagent-grade salt. The sea salt was purchased from SaltWorks®; it does not contain anti-caking or free-flowing additives or conditioners. The main benefit of using sea salt is that it comes in a variety of grain sizes, in contrast to the limited grain-size range of the reagent-grade salt. Before consolidation, the granular salt is sieved in a controlled low humidity environment (RH < 17%) to produce more uniform grain size distributions (Table 1). The starting water content is determined from the mass of the salt before and after heating in an oven to 500 °C for 2-3 hours. Heating to this temperature promotes thermal cracking which allows the release of fluid trapped in fluid inclusions. The difference in mass before and after heating is taken as a measure of the total water content.

Table 1. As-received condition of granular salt used herein.

Salt Type	Purity (NaCl wt%)	Grain Size (mm)	Water Content (wt%)
Reagent-grade salt	99	0.3-0.355	0.43
Sea salt	96	1-1.2	0.38
Sea salt	96	1.7-2	0.38

2.2. Apparatus

A consolidation system was developed to achieve consolidation at temperatures up to 230 °C and displacement rates up to 0.047 mm/s (Fig. 1). In this system, an alumina plate is placed between the stationary plate and bottom plate to minimize heat dissipation through the loading frame. A cylindrical die with a 19 mm (0.75 inch) diameter hole sits on the lower piston of the bottom plate, which contains a port for evacuation. To facilitate air flow during evacuation, a perforated stainless steel spacer is placed on top of lower piston. Both upper and lower pistons are equipped with o-rings seals to maintain a vacuum during consolidation. Two bevel rings are placed on the two ends of the consolidated sample to ensure that the ends of the consolidated sample are square. Resistance wire heating strips are wrapped around the cylindrical die and controlled to increase and maintain elevated temperature. A K-type thermocouple is inserted into a small-diameter blind hole in the die to measure temperature close to the salt sample. Loading and unloading are achieved either with a hydraulic cylinder placed between the stationary plate and alumina plate while the movable plate is fixed, or with an electric motor and gear-driven ball-screw that displace the movable plate at a constant speed. Axial force and displacement are measured via a load cell and LVDT, respectively. The double-ended moveable-piston

loading configuration helps to reduce stress gradients during consolidation.

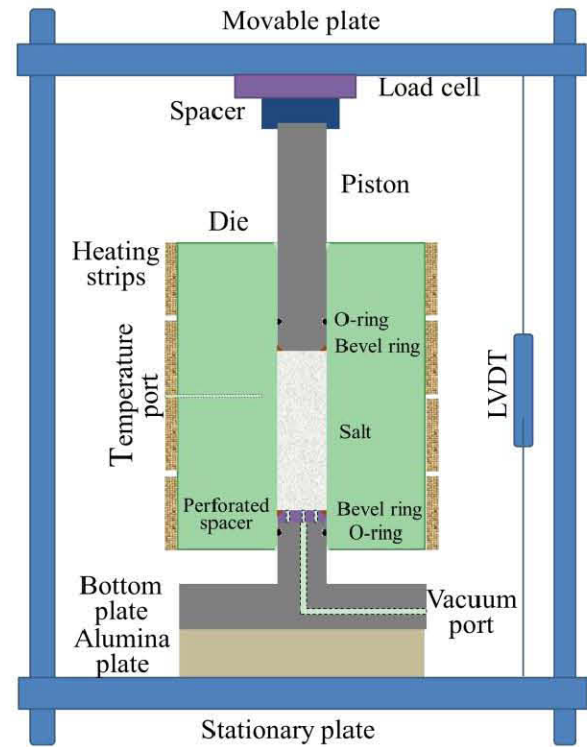


Fig. 1. Schematic diagram of the consolidation system.

A controlled low humidity environment is established by flowing dry compressed air through a glove box (Fig. 2). Relative humidity as low as 10% can easily be achieved and maintained. Granular salt and consolidated salt samples are handled at all stages of preparation for the experiments and microscopy in this low humidity environment to keep the samples nominally dry.

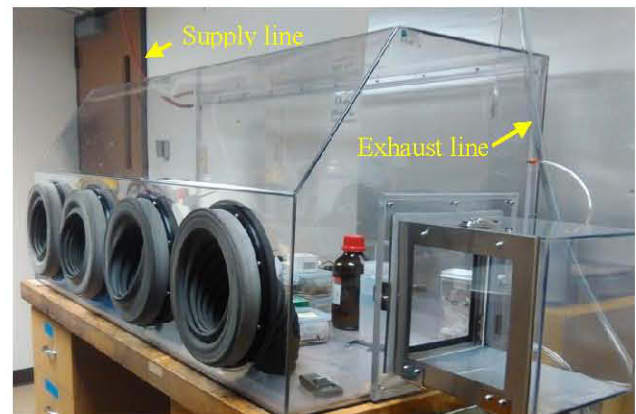


Fig. 2. Photograph of controlled low humidity environment achieved by flowing dry compressed air through a glove box. Flow rate is adjustable through a regulator (not in photo) to maintain relative humidity < 17%.

2.3. Procedures

Consolidation experiments are conducted for a range of conditions by adjusting grain size, temperature, load path, and maximum axial stress. First, the inner wall of consolidation die, end surfaces of the pistons, and bottom plate are uniformly sprayed with graphite film to reduce friction along the inner wall of the cylinder. Then heating strips are installed and temperature is increased to the target value. Granular salt is then poured into the die followed by inserting the upper piston. Once the stack is sealed, the system is evacuated. Two distinct load paths are employed: 1) A “creep-test” load path (LP1) is simulated using the hydraulic cylinder to raise axial load to the target value in less than 2 minutes, and then maintaining the load for 10 minutes; 2) A constant rate load path (LP2) is achieved using the electric motor/ball-screw system to drive the movable plate down at a constant displacement rate of 0.034 mm/s, corresponding to a strain rate of $\sim 5 \times 10^{-4} \text{ s}^{-1}$. Once the target stress is achieved, the motor is immediately reversed to begin unloading. For both load paths, the vacuum is monitored and maintained during consolidation. For LP2, granular salt is dried in-situ by maintaining an elevated temperature for 1-2 hours, during which the vacuum is re-established about every 15 minutes to remove additional water vapor released during heating. This step reduces water content in the salt by about half, yielding samples with $\sim 0.22 \text{ wt}\%$ water for sea salt, and $\sim 0.27 \text{ wt}\%$ water for reagent-grade salt. After unloading, the consolidation stack is cooled by two electric fans while maintaining the vacuum in the salt sample chamber. Upon reaching room temperature, the entire consolidation assembly is transferred to the controlled low humidity environment, where the pistons are removed and the salt sample is easily released due to the different thermal expansion coefficients of salt and steel (Peach and Spiers, 1996).

Consolidated salt sample dimensions and weight are measured to determine the final porosity. For microscopic study, the starting material and key consolidated samples are epoxy-saturated, cut, and polished to make petrographic sections, and then chemically etched to allow observation of grain-scale features, including grain boundaries and dislocations. The sectioning and etching procedures follow the techniques developed by Spiers et al. (1986) with minor modifications. During this entire process, the consolidated salt samples remain in the controlled low humidity environment.

3. RESULTS AND DISCUSSION

3.1. Mechanical behavior

The porosity evolution as a function of axial stress during consolidation is determined from measurements of sample dimensions and weight, axial displacement,

and axial force. Sea salt experiments for different grain size ranges deformed at different temperatures and the two load paths show that final porosity is related to peak stress and a strong dependence on temperature (Fig. 3). Starting porosities before consolidation are generally similar, ranging from 43.2% to 44.8%; the range likely reflects slight differences in initial packing. With an increase in peak stress, porosity decreases approximately linearly with log stress, except at porosities less than $\sim 5\%$. Temperature dependence of compaction is very apparent, as higher temperatures result in lower porosities at the same stress level. Grain size and load path have little influence on the final porosity.

The complete consolidation curves for sea salt deformed under LP2 at three different temperatures (Fig. 4) are consistent with the final porosity results shown in Fig. 3. The data show that at intermediate stress levels, void ratio is linearly related to log stress, except at very low and very high stress levels.

Reproducible mechanical behaviour is illustrated in Fig. 5 for two samples deformed at 4 MPa confining pressure, room temperature, and a strain rate of $1.4 \times 10^{-4} \text{ s}^{-1}$. Cyclic loading is applied in both elastic and plastic deformation regimes. Comparison of the two experiments shows excellent reproducibility in mechanical response. These two samples were consolidated at the same conditions, so we interpret that the reproducibility reflects similar porosity and microstructure produced during the consolidation stage.

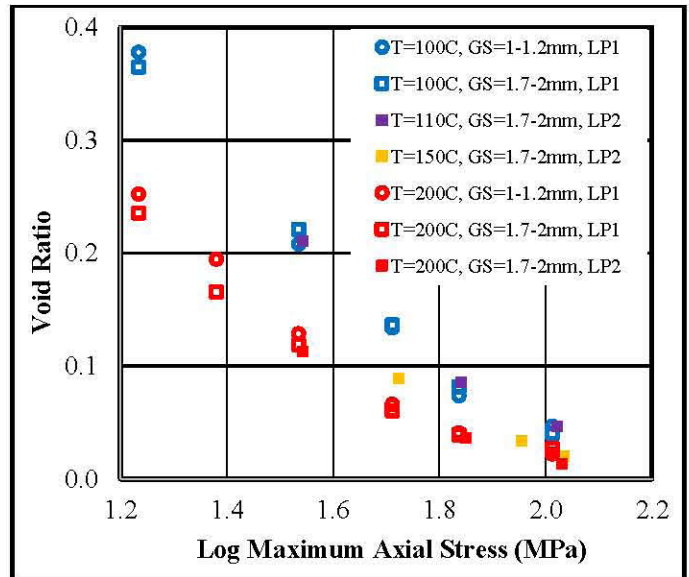


Fig. 3. Void ratio versus the logarithm of maximum axial stress for granular sea salt consolidated at various conditions. Void ratio, e , is related to porosity, ϕ , by $e = \phi / (1 - \phi)$.

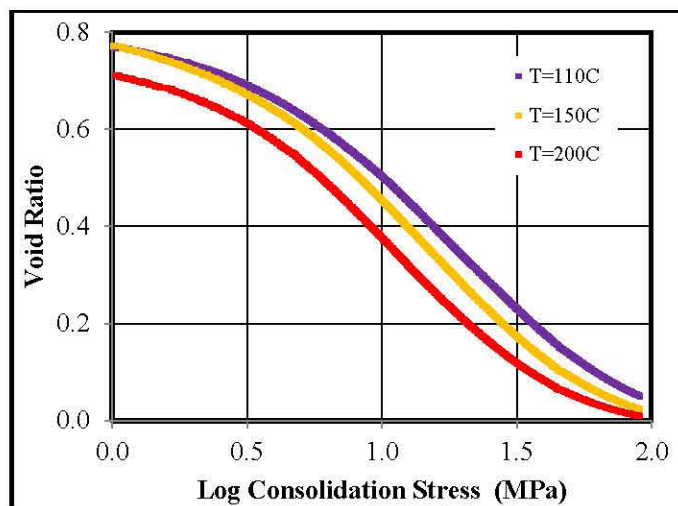


Fig. 4. Void ratio versus the logarithm of consolidation stress for granular sea salt (grain size 1.7-2 mm) consolidated at temperatures of 110 °C, 150 °C and 200 °C with LP2. Void ratio, e , is related to porosity, ϕ , by $e = \phi / (1 - \phi)$.

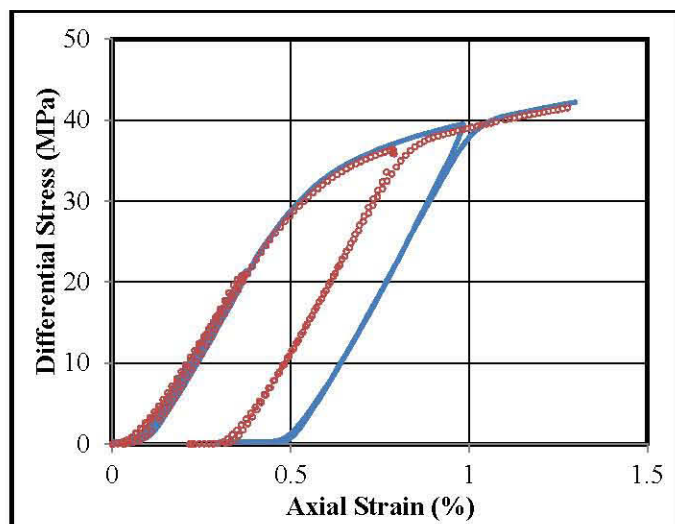


Fig. 5. Stress-strain curves of triaxial compression tests (confining pressure 4 MPa, strain rate $1.4 \times 10^{-4} \text{ s}^{-1}$, room temperature) on two synthetic rock salt samples consolidated with LP1 to a final porosity of $\sim 4\%$ at the same conditions (grain size 1.7-2 mm, maximum axial stress 69 MPa, temperature 200 °C). Cyclic loading is applied in both elastic and plastic deformation regimes.

3.2. Microstructures

Although the sea salt used in this study is of high purity and free of additives, fluid inclusions are abundant and have different sizes (Fig. 6, 7). It is common to observe fluid inclusions arranged in planar arrays, suggesting they are healed cleavage cracks. Most fluid inclusions are cubic in shape with a visible gas bubble, indicating the presence of brine (Van den Kerkhof and Hein, 2001). There are also tube-shaped fluid inclusions which are often larger in size. As fluid inclusions intersect with the polished surface of the petrographic section, under reflected light they appear as cubic or tube-shaped pits.

Synthetic rock salt samples consolidated using LP2 at 150 °C and maximum axial stresses of 53 and 90 MPa show very few intragranular cracks, indicating limited influence of brittle deformation during consolidation (Fig. 8, 9). Etched surface structures demonstrate inhomogeneous strain accumulation as evidenced by high dislocation density near grain contacts and relatively dislocation-free areas close to pores. Fluid inclusions manifest themselves in the form of dark pits and are often the sites of high concentration of dislocations, slip bands and subgrains. Static recrystallized grains, absent in the starting material, appear in some of the most highly strained areas of the consolidated samples. These recrystallized grains, characterised by straight boundaries and dislocation-feature-free interiors, grew by grain-boundary migration at expense of highly deformed grains.

At the lower maximum axial stress (53 MPa), the sample has a higher porosity and larger pores (Fig. 8). Grains adjacent to pores exhibit fewer or no apparent dislocations. In contrast, stressed grain contacts are characterized by dense dislocation structures. At higher stress (90 MPa), the sample is more compacted, and has a lower porosity and a greater dislocation density overall (Fig. 9). Both samples, however, display a heterogeneous distribution of dislocations because of the variable size and orientation of grain contacts, and variable internal grain structure (e.g., number and locations of fluid inclusions).

Although the reagent-grade salt is much smaller in grain size, the microstructures of the consolidated reagent-grade samples are similar to those noted in sea salt samples. The reagent-grade starting material is free of dislocations, but also contains abundant fluid inclusions (Fig. 10). Consolidation produces regions of high dislocation density, and recrystallized grains are also observed in some highly strained areas (Fig. 11).

Depending on applied stress, strain rate, temperature, time, and water content, consolidation of granular material often involves grain rotation, sliding, and crushing. For salt, crystal plasticity, dissolution-precipitation, and dynamic recrystallization are also important mechanisms (Hwang et al., 1993). Microstructural observations in this study suggest crystal plasticity dominates the deformation process, as evidenced by the high-density of dislocations and small number of recrystallized grains. There is some evidence of intragranular and grain-boundary fractures in samples consolidated at 150 °C, but overall brittle deformation is minor, even though the samples are nominally dry. As to the formation of the recrystallized grains observed in this study, we infer that they grow during the 2-3 hour period after the consolidation test, while the cell is cooled, because the recrystallized grains exhibit relatively strain-free interiors and cubic shapes.

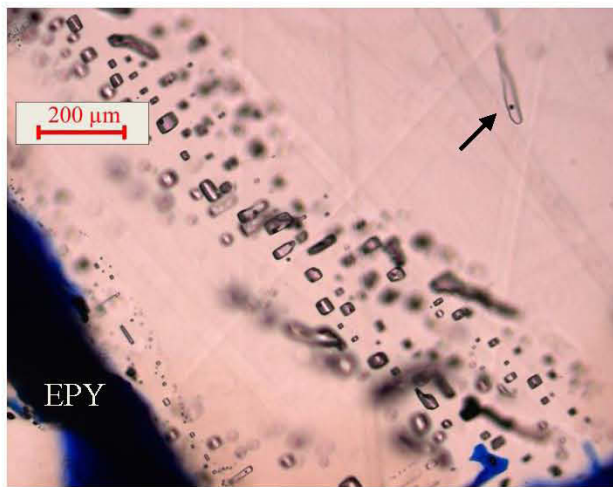


Fig. 6. Transmitted light micrograph of sea salt starting material. Translucent areas are salt grains, blue and dark blue, areas are epoxy; black band labeled EPY, also is epoxy. Planar arrangement of fluid inclusions is clearly visible. Arrow points to a tube-like fluid inclusion containing a small gas bubble (black dot).

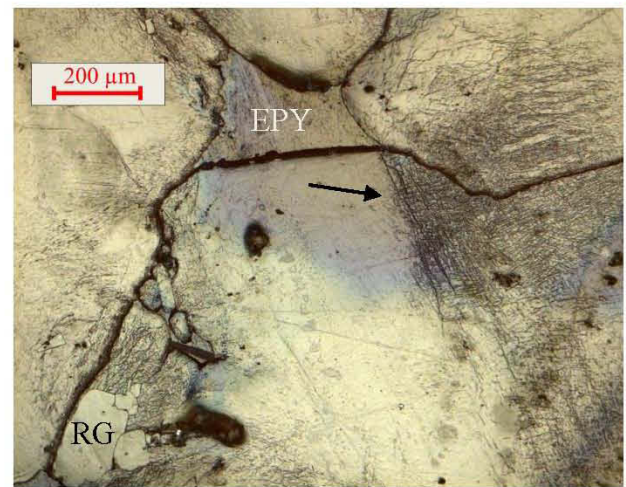


Fig. 8. Reflected light micrograph of sea salt sample consolidated at 150 °C and 53 MPa maximum axial stress. Surface is etched. Arrow points to slip bands in areas of high strain accumulation. Sections are cut parallel to the axial compression direction (vertical). RG - recrystallized grain, EPY - epoxy.

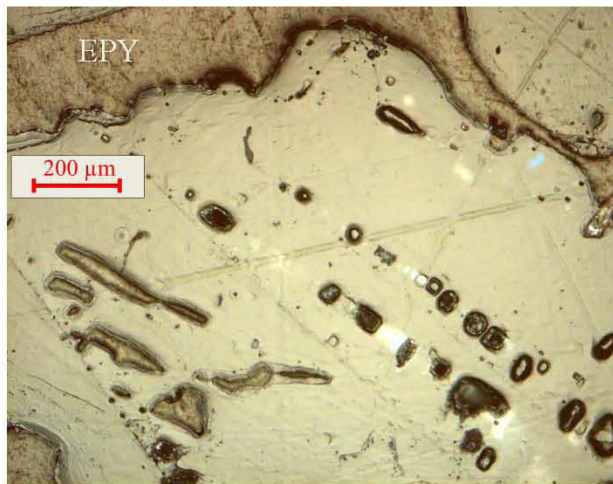


Fig. 7. Reflected light micrograph of sea salt starting material. Surface is etched. Salt grains are free of dislocation structure. Cubic and tube-like pits are the result of fluid inclusions intersecting polishing surface. EPY - epoxy.

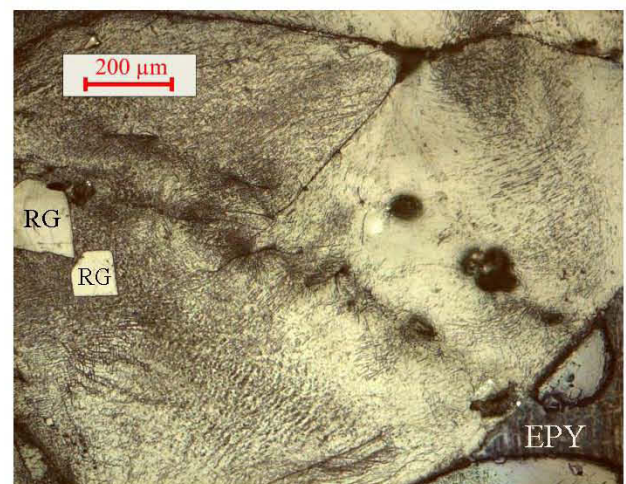


Fig. 9. Reflected light micrograph of sea salt sample consolidated at 150 °C and 90 MPa maximum axial stress. Surface is etched. Dislocation structure reveals strain accumulation. Pits are the result of fluid inclusions intersecting the polished surface. Two recrystallized grains (RG) have straight boundaries and are free of dislocations. Sections are cut parallel to the axial compression direction (vertical). EPY - epoxy.

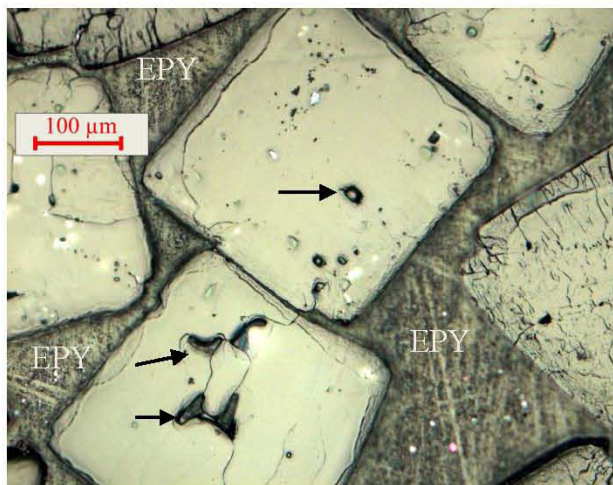


Fig. 10. Reflected light micrograph of reagent-grade salt starting material. Surface is etched. Salt grains are free of dislocations. Arrows point to pits resulting from fluid inclusions intersecting the polished surface. EPY - epoxy.

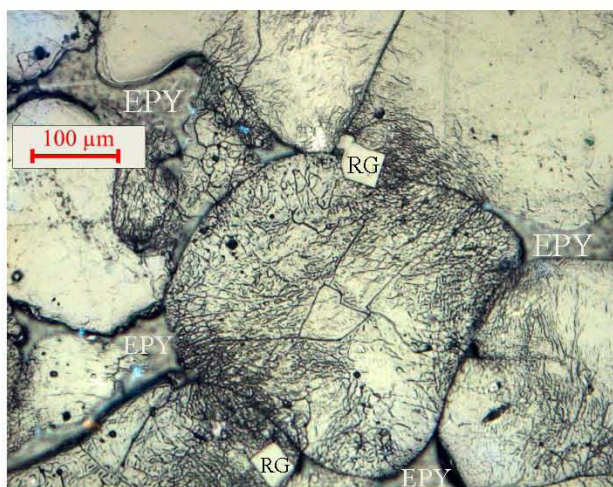


Fig. 11. Reflected light micrograph of reagent-grade salt sample consolidated at 150 °C and 53 MPa maximum axial stress. Surface is etched. Dislocation structure shows areas of high strain accumulation. Recrystallized grains (RG) develop in highly strained area. Sections are cut parallel to the axial compression direction (vertical). EPY - epoxy.

4. SUMMARY AND CONCLUSIONS

In this study, we conducted uniaxial consolidation experiments on sieved, nominally dry granular sea salt and reagent-grade salt at different temperatures and load paths. Mechanical behavior and microstructure observations demonstrate the following:

1. Consolidation in granular salt is strongly affected by temperature, whereas the grain size and load paths employed in this study show limited influence.
2. At the tested conditions, the dominant mechanism of consolidation is crystal plasticity.

3. In some highly strained areas of the consolidated salt samples, recrystallized grains are observed and interpreted to reflect static recrystallization.

Microscopic observation shows that our synthetic rock salt samples have high dislocation densities, and therefore are hardened relative to the starting state. Our future work will involve conducting experiments on these samples at low pressures in the brittle deformation regime to investigate the effect of cracking and healing on mechanical properties. Knowledge gained from this study will be used to fabricate samples most suitable for the deformation tests, as well as to guide appropriate models for designing rock salt fabrics for engineering needs.

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