Deformation mechanisms and rheology of Pre-cambrian rocksalt from the South Oman Salt Basin

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ABSTRACT: The Neoproterozoic to Early Cambrian Ara Salt from the South Oman Salt Basin has been investigated for creep behavior and microstructure. The salt behavior is of relevance for oil production out of intra-salt carbonate "stringers". Test and in-situ conditions are about T = 100 °C and p_c = 70 MPa. Strain rate stepping and temperature stepping tests have been performed, with creep ranges varying from $5 \times 10^{-8}$ to $10^{-5}$ s$^{-1}$. All tests were run to a total strain of 25 - 30%. Both secondary and primary creep parameters have been determined. Secondary creep is basically reached only at rates of $10^{-7}$ s$^{-1}$ or lower (15 MPa differential stress or lower), still orders of magnitude faster than expected in situ rates, except perhaps borehole closure related creep during drilling. Microstructural observations revealed that the main deformation mechanism during the tests was dislocation creep associated with grain boundary migration recrystallization. This is evidenced by subgrain size reduction, the development of slip lines and the formation of new strain-free grains growing at the expense of subgrain-rich grains. Comparing literature data, the Ara Salt appears to be quite common (average), both under the microscope as in creep behavior.

1 INTRODUCTION

1.1 Experimental deformation of halite

The vast amount of experimental creep data on halite shows that there is a significant factor of 100 - 1000 variation in creep rate for different types of natural halite deformed under same stresses and temperatures, due to differences in chemistry, water content, secondary phases and deformation history. By comparing dry versus wet (Ter Heege et al., 2005), pure versus doped (Heard and Ryerson, 1986), second phase free versus dirty (Hunsche et al., 2003) and annealed versus deformed samples (Ter Heege et al., 2005) we gained a first order approximation of the influence of these parameters on creep rate of halite and also on the physics behind the individual processes. However, when more variables are simultaneously present, it is still poorly known, how these processes interact and what their exact influence on rheology is. It is still impossible to predict creep rate parameters with any accuracy from chemical analysis and microscope studies. Lab creep testing will hence still be required.

The draw back of creep tests is that true simulation of relevant in-situ creep rates ($10^{-5}$ to $10^{-13}$ s$^{-1}$ for mining and oil-gas extraction related creep deformation) would require very long lasting experiments, since the creep rate must be maintained for several percents of strain to reach a steady state strain rate. This problem is normally solved by extrapolation from experiments done at higher than natural strain rate, stress or temperature. However, such an extrapolation is only valid, if the deformation mechanisms remain the same in the experiment and in nature.

Microstructure analyses provide insights into the correct, mechanism-based deformation processes. The constitutive models (including recrystallization processes) have to be used to verify the validity of extrapolation of laboratory data to natural conditions.

Although both, microstructure study and experiments on synthetic salts help to predict the rheology, the only way to derive this for a specific salt type is to carry out a set of triaxial tests, which establishes required parameters for describing rheology. This was performed for the Infra-Cambrian Ara Salt, which forms a number of large salt diapirs in the South Oman Salt Basin (SOSB) at a depth of 3 to 5 km. It encases highly over-pressured carbonate reservoirs (the so-called “stringers”) for oil and gas (Fig. 1).
Previous studies of the Ara Salt have shown that it once lost its seal capacity in the direct vicinity (~10 m of vertical distance) of the stringer reservoirs, which is evidenced by the presence of solid bitumen at grain boundaries and microcracks (Schoenherr et al., submitted). At the same time, this salt shows widespread evidence for intracrystalline plastic deformation by the presence of subgrains. Using subgrain size piezometry and the well established correlation between subgrain size and remaining in-situ stress (Carter et al., 1993), the calculated maximum past differential stress for the Ara Salt around the stringer reservoirs is less than 2 MPa.

The mechanical and transport properties of this salt are of relevance in predicting seal capacity, flow of salt around boreholes and in geomechanical modeling of the salt loading (compression) of the “stringer” reservoirs during production. Thus, the aim of our deformation experiments was to obtain as much as possible information on the rheological properties of Ara Salt under in-situ conditions and low strain rates, corresponding to deformations around a producing carbonate stringer reservoir over decades.

1.2 Deformation mechanisms in halite

Based on laboratory experiments, deformation of halite at high (or medium) confining pressures is dominated by different deformation mechanisms: dislocation cross-slip (CS), dislocation climb (CL) and pressure solution (PS). Several studies on natural halite indicate that at geological strain rates salt is in the transition regime where both dislocation creep and solution-precipitation creep mechanisms operate (e.g. Ter Heege et al., 2005). Assuming that dislocation creep processes and pressure solution act in parallel and in absence of microcracking, the steady-state flow of natural halite can be approximated by the following equation (Speirs and Carter, 1998):

$$\dot{\varepsilon} = \dot{\varepsilon}_{CS} + \dot{\varepsilon}_{CL} + \dot{\varepsilon}_{PS}$$

where $\dot{\varepsilon}$ is the total strain rate in s$^{-1}$, $\dot{\varepsilon}_{CS}$ and $\dot{\varepsilon}_{CL}$ are strain rates from dislocation creep processes, and $\dot{\varepsilon}_{PS}$ is the strain rate from pressure solution.

Dislocation creep behavior is characteristic in experiments using natural samples with grain sizes over 2 mm. The deformation mechanism is a combination of dislocation climb and dislocation cross-slip (Carter et al., 1993), in wet halite usually accompanied with dynamic recrystallization by fluid-assisted grain boundary migration. The combined creep mechanism is usually treated as a single combined creep mechanism for practical reasons. For halite, the strain rate ($\dot{\varepsilon}$) can be related to the flow stress ($\sigma$) using a power law creep equation:

$$\dot{\varepsilon} = A_1 \exp \left( \frac{-Q}{RT} \right) (\sigma - \sigma_i)^n$$

where the pre-exponential constant $A$ is expressed in MPa$^{-n} \cdot$ s$^{-1}$, $Q$ is the apparent activation energy for creep in J mol$^{-1}$, the gas constant $R$ is in J mol$^{-1} \cdot$ K$^{-1}$, the temperature $T$ is in K, the differential stress ($\sigma_1 - \sigma_3$) is in MPa and $n$ is the dimensionless stress exponent. A somewhat more scientific notation would be:

$$\dot{\varepsilon} = A_1 \exp \left( \frac{-Q}{RT} \right) \left( \frac{\sigma}{\sigma_y} \right)^n$$

where $A_1$ is in s$^{-1}$, $\sigma$ is the Von Mises stress in MPa ($= \sigma_1 - \sigma_3$ for triaxial loading) and $\sigma_y$ is a unifying constant of 1 MPa.

2 METHODS

2.1 Triaxial creep tests

The triaxial creep tests were carried out at the laboratories of the Bundesanstalt für Geowissenschaften und Rohstoffe (BGR), in Hannover, Germany. The creep tests were performed on cylindrical 50 x 100 mm samples and designed to best represent the in-situ conditions at the depth of ~ 5 km in the Zalzala-2H1 halite interval. For all experiments sample preparation was the same, which involves adding 50 or 20 ml brine (~ 35 drops) in between the jacket and the sample to restore the small amounts of water, which is always present in natural salt rocks (Roedder, 1984; Urai et al., 1986) but may have been lost during storage. The sample was put into...
the creep machine together with a thin Teflon foil between the ends of the sample and driving the pistons to minimize friction and barreling. The starting (confining) pressures were $p_c = 3.5 - 4 \text{ MPa}$ with a starting stress of $\sigma_1 = 4.5 \text{ MPa}$ and $6 \text{ MPa}$. After this, the sample was heated up to the required temperature ($80, 100, 120 \degree \text{C}$) with < 30 °C/h. The pressure was then increased hydrostatically with ~ 1 MPa/min until $p_c = 70 \text{ MPa}$ and $\sigma_1 = 72 \text{ MPa}$. During the experiments the $p_c$ was kept at ~ 70 MPa. The samples were dismantled and machined down (de-barreling) at 10% and 20% or at 15% depending on the test sequence.

2.2 Microstructure analysis

From the original halite cores we cut a 1 cm thick slab parallel to the long axis of the core, using a diamond saw cooled by water to avoid microcracking of the sample. This slab is used as an undeformed reference material. From the remaining material cylindrical samples were machined down for the creep tests. After the creep tests, 1 cm thick slabs were cut from the deformed cores parallel to the deformation axis. Following the conventional method at the institute of Geologie–Endogene Dynamik, RWTH Aachen, from both slabs, the experimentally undeformed material and the experimentally deformed material double polished and etched thick sections (~ 1.5 mm) were prepared.

Note that, for the sake of simplicity, we introduced short laboratory sample names to refer to the samples and experiments, respectively (e.g. ZAZ 1). For similar reasons, we use the term of “undeformed” for denoting the naturally deformed samples and the term “deformed” for experimentally deformed samples.

3 RESULTS & DISCUSSION

3.1 Triaxial creep tests

In this paragraph we describe some of the six deformation test, which is followed by a characterization of the corresponding microstructures. Six creep tests were performed and corrected for measured piston friction (1.5 MPa). All but one are strain rate stepping tests performed at 100 °C.

The stress-strain curve of experiment ZAZ 4 shows three steps, which correspond to a temperature decrease from 120 °C (0 - 10%), to 100 °C (10% - 20%) to 80 °C at 20% - 30% strain (Fig. 2c). Each temperature step of this test is characterized by an increase of ~ 2 MPa in differential stress due to the decreasing temperature. Steady state flow occurred in all temperature steps. This test was designed to estimate the activation energy $Q$ for the secondary creep law.

The ZAZ 9 test was performed at a constant strain rate of $10^{-7} \text{ s}^{-1}$ to a total strain of about 27% (Fig. 2e). The resulting microstructures of both processes, i.e. subgrains and lobate grain boundaries were observed in this sample (Figs. 5a and 5c).

Figure 2. Stress-strain curves for Ara Salt creep experiments; test parameters are indicated by insets. a) Creep test ZAZ 1 shows primary creep until a strain of ~ 17%, followed by steady state creep. b) Each strain rate step of test ZAZ 2 shows continuous strain hardening. The gap at 15% strain is due to interruption of the test during machining down the sample. c) Stress-strain curve for the ZAZ 4 experiment at a strain rate of $10^{-7} \text{ s}^{-1}$ and three temperature steps. d) Creep test ZAZ 5 has three strain rate steps. True steady state conditions were only reached between 8 - 10% strain in the 5 x $10^{-8} \text{ s}^{-1}$ strain rate step, while the other steps still show signs of strain hardening. e) The creep curve of sample ZAZ 9 shows steady state from a strain of ~ 8%. Small oscillations may be a result of competing processes of strain hardening and strain softening. f) Creep curve of test ZAZ 10 shows three strain rate steps from $10^{-7}$ to $10^{-5} \text{ s}^{-1}$. The $10^{-6} \text{ s}^{-1}$ step appears to approach steady state in the last few percents of strain.

The differential stress and steady state strain rate data were plotted in order to determine the relative position of the Ara Salt rheology to other reported halites (Fig. 3). The reason for the lack of the steady-state conditions in some experiments (ZAZ 2 and partially ZAZ 10) results from the relatively high strain rate. Only the rates at or below $10^{-7} \text{ s}^{-1}$ displayed steady state within 10-15% of strain. Figure 3 shows that Ara Salt rheology is comparable with the most common halite (e.g. Asse, Gorleben) for which an extensive dataset is available (Table 1). As a result of the limited range in strain rates at which steady state creep occurred, it is impossible to pinpoint the exact power $n$. With some assumptions on the primary creep mechanism, we can limit the range to about 4 - 6. We assume that in-situ, the Ara
Salt has the most commonly used stress exponent $n$ for halite, which is $\sim 5$.

In the literature, many flow laws were published for different types of halite (Table 1). These flow laws differ in $A$, $Q$ and $n$ values. If we assume the $n=5$ value discussed above, we are able to deduce the minimum activation energy $Q$ from the stepped temperature experiment ZAZ 4 by plotting $1/T$ versus ln differential stress. Then, $Q$ is $32400$ J mole$^{-1}$ and the pre-exponential $A$ is $1.82 \times 10^{-9}$ (MPa-n)s$^{-1}$ (Table 1 and Eq. 2 and 2a).

Table 1. Literature creep data for various halite types and data from this study.

<table>
<thead>
<tr>
<th>Source</th>
<th>Halite type</th>
<th>A (MPa-n s$^{-1}$)</th>
<th>Q (Jmol$^{-1}$)</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wawersik and Zeuch (1986)</td>
<td>Salado</td>
<td>8.82E-06</td>
<td>50160</td>
<td>4.9</td>
</tr>
<tr>
<td></td>
<td>Asse</td>
<td>7.26E-06</td>
<td>53920</td>
<td>5.0</td>
</tr>
<tr>
<td></td>
<td>West</td>
<td>3.15E-05</td>
<td>54840</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td>Hackbr.</td>
<td>8.93E-05</td>
<td>63290</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>Bryan mound</td>
<td>7.78E-03</td>
<td>74530</td>
<td>5.2</td>
</tr>
<tr>
<td></td>
<td>Bayou Choctaw</td>
<td>3.94E-06</td>
<td>49450</td>
<td>4.1</td>
</tr>
<tr>
<td>Spiers et al. (1990)</td>
<td>Synthetic</td>
<td>4.70E-04</td>
<td>24530</td>
<td>3.0</td>
</tr>
<tr>
<td>Carter et al. (1993)</td>
<td>Avery Island</td>
<td>1.60E-04</td>
<td>68100</td>
<td>5.3</td>
</tr>
<tr>
<td></td>
<td>Avery Island</td>
<td>8.10E-05</td>
<td>51600</td>
<td>3.4</td>
</tr>
<tr>
<td>Ter Heege et al. (2005)</td>
<td>Synthetic wet salt</td>
<td>2.75E-02</td>
<td>80000</td>
<td>5.6</td>
</tr>
<tr>
<td>This study</td>
<td>Ara Salt</td>
<td>1.82E-09</td>
<td>32400</td>
<td>5.0</td>
</tr>
</tbody>
</table>

3.2 Microstructural evolution

3.2.1 Undeformed samples

The starting (undeformed) material consists of almost pure colorless halite with 2 - 3% impurities of fine-grained idiomorphic anhydrite crystals and minor fine-grained carbonate grains. Grain size analysis revealed a grain size distribution between 0.5 - 7 mm with a mean equivalent circular diameter (ECD) of 2 mm.

![Microstructural evolution](image)

In all samples, some anhedral grains contain cube-shaped fluid inclusions in its centre, which are aligned in more or less parallel bands alternating with fluid inclusion-poor bands (Fig. 4a). Analyses of homogenization temperature of similar fluid inclusions microstructures suggest that one pair of a fluid inclusion-rich and a fluid inclusion-poor band reflect daily temperature variations of the brine, from which the crystal precipitated (Benison and Goldstein, 1999). Thus, such grains are considered to be of primary (syn-depositional) origin (Roedder, 1984). Grain boundaries generally contain abundant isolated and amoeboid fluid inclusions under transmitted light (Fig. 4b), which probably derive from the liberation of the primary fluid inclusions due to grain boundary migration recrystallization (GBMR) (Schléder and Urai, 2005). When the migrating grain boundary reaches the fluid inclusion, its content is collected and redistributed at the grain boundary.
Under reflected light, GBMR is also indicated by strain-free crystals, which show lobate grain boundaries related to substructure-rich grains (Fig. 4c). Fluid inclusions trails, which are aligned parallel to the lobate grain boundaries and terminations of small isolated fluid inclusions at the grain boundary, indicate that GBMR was fluid-assisted. Other less substructured grains show elongated subgrains with an orientation perpendicular to grain boundaries (Fig. 4d). This points to the operation of dislocation creep processes due to GBMR.

The experimentally undeformed samples are (incompletely) dynamically recrystallized as shown by anhedral primary grains (Fig. 4a). Grain boundary migration processes are indicated by substructure-free grains, which show a lobate-shaped geometry with respect to the substructured ones. Under reflected light, most grains show 150 - 300 µm-sized subgrains (Fig. 4c), which indicate a maximum past differential stress of ~1 MPa (subgrain size piezometry using data of Carter et al. (1993) and Franssen (1993); for results see table 2).

**Table 2.** Maximum in-situ differential paleo-stress of undeformed Zalzala-2H1 salt cores calculated from subgrain size.

<table>
<thead>
<tr>
<th>Sample</th>
<th>No. of subgrains</th>
<th>Subgrain diameter (µm)</th>
<th>Differential stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZAZ 3</td>
<td>106</td>
<td>222</td>
<td>0.96</td>
</tr>
<tr>
<td>ZAZ 9</td>
<td>76</td>
<td>208</td>
<td>1.01</td>
</tr>
</tbody>
</table>

### 3.2.3 Deformed samples

The final microstructure (after 25 - 30% strain) of the deformed Ara Salt shows that grain size slightly decreased (average grain size is ~1.5 mm). Most grains exhibit internal substructure such as ~30 µm-sized subgrains (Fig. 5a) and linear to wavy slip lines (Fig. 5b), the latter probably indicating cross slip of screw dislocations (Carter et al., 1993). The substructured grains are, in places consumed by new strain-free grains (Figs. 5a and 5c-d) by grain boundary migration, which is shown by highly serrated grain boundaries (Fig. 5c). Those grain boundaries show less fluid inclusions than grain boundaries in the undeformed samples.

The most significant differences between microstructures of the starting and deformed material are:

- subgrain size reduction from 150 - 200 µm to 10 - 50 µm
- no crystals with (primary) fluid inclusion bands were observed in the deformed samples
- presence of slip lines in the deformed samples
- replacement of grains with 150 - 200 µm-sized subgrains by almost strain-free grains in deformed samples

We interpret these differences due to dynamic recrystallization of the halite during the experiments, which produces some obvious changes in the microstructure. This is shown for instance by the absence of primary fluid inclusion bands, which have been consumed by GBMR. It has been shown that dislocation creep processes were active during the experiments, which are responsible for the continuous strain hardening at the higher strain rates. This is indicated by reduced subgrain size and the presence of slip lines. During deformation with the very low strain rates GBMR most likely operated in the later stage of the experiments (probably due to strain softening) as suggested by new strain-free grains grown at the expense of grains exhibiting 10 - 50 µm-sized subgrains. GBMR most likely removed the primary fluid inclusions, which were then liberated during fluid-assisted grain boundary migration.

### 3.3 Primary versus secondary creep

The test results were fitted with a combined hardening and softening law, the hardening (primary creep) law being derived from Fokker (1995), which can be translated to a Lemaître type of creep equation. The primary creep mechanism is not important for halite response on reservoir rocks (given the slow loading during decades) but might be of importance for bore hole closure during drilling. Unrav-
eling the mechanism gives some clues on the applicable mechanisms.

Equation 3 shows the applied hardening and softening law. The total (vertical) strain is denoted by \( \varepsilon \), the strain softening by secondary creep (calculated via Equation 2a) is denoted as \( \varepsilon_s \) and a correction strain is denoted by \( \varepsilon_{corr} \).

\[
\dot{\varepsilon} = A_p \exp \left( \frac{-Q_p}{RT} \right) \left( \frac{\sigma}{\sigma_0 (\varepsilon - \varepsilon_s - \varepsilon_{corr})^\beta} \right)^m
\]  

(3)

The correction strain is some strain hardening or strain softening effect due to the coring, machining or annealing, when no strain rate is applied on the sample. It is likely that during the build-out, build-in procedure for machining down the sample, some “strain softening” or annealing occurs, evidenced by repeated strain hardening effects at unchanged rates. Since the strain rate stepping was only performed at constant temperatures, we presumed an activation energy equal to the secondary creep mechanism \( Q_p = Q_s \), a hypothesis which was not tested.

The best fitting set of parameters for all creep tests appears to be: \( m = 10; \beta = 0.18; \sigma_0 = 22 \text{ MPa}; A_p = 0.00354 \text{ s}^{-1} \). Note that \( \sigma_0 \) is interdependent with \( A_p \) and \( m \). The primary creep law hence only requires 3 parameters to fit for constant temperature tests.

Figure 6 shows the resulting stress-strain simulation for the test on sample ZAZ 5. The best fit could be obtained by assuming a negative (hardening) strain correction of 4% at the beginning of the test and positive strain (softening) of 4% and 1.5% during the (dis)mounting and machining of the samples.

3.4 Test procedures

The applied test temperature of 100 °C is simply too low to obtain steady state creep at strain rates that differ an order of magnitude and are within the strain rate potential of the applied test apparatus. If similar tests should be repeated, it should be considered to run constant stress test for the lower strain rates or to modify the machines to be able to handle slower strain rates \( (10^{-8} \text{ s}^{-1}) \).

When halite samples are strained to 20-30%, they require machining down to prevent excessive barreling and sleeve failure. Advantage of using the same sample for 2 - 3 tests is that one is sure that there is no sample-to-sample variation. The high achieved strains do not enhance the reaching of steady state though, since healing effects are operating in the period that the sample is reworked, part of the period being at or near the test temperature. For the derivation of steady state parameters it is hence important to obtain steady state within 10 - 15% of strain, hence applying low strain rates or high(er) test temperatures.

4 CONCLUSIONS

The Ara Salt is comparable with the most common halite rocks for which an extensive dataset is available (e.g. Hunsche et al., 2003).

The basic results and conclusions are summarized:

1. The steady state mechanical data for the Ara Salt can be described by a power law equation with \( n = 5, Q = 32400 \text{ J mole}^{-1} \) and \( A = 1.82 \times 10^{-9} \text{ s}^{-1} \). Separate primary creep parameters have been derived.

2. Microstructure analysis has shown that dislocation creep processes led to the reduction in sub-grain size and the formation of (wavy) slip lines. In addition, fluid-assisted grain boundary migration recrystallization triggered growth of new strain-free grains, which replaced grains with a high dislocation density.

3. Test procedures for “deep salt” should be focused on obtaining steady state creep within 10-15% of strain with at least one order of magnitude in strain rates. Tests in the twilight zone of primary and secondary creep are more difficult to analyze.
REFERENCES


