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

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ABSTRACT

In the South Oman salt basin (SOSB), diapirs of infra-Cambrian Ara Salt enclose isolated, commonly overpressured carbonate reservoirs. Hydrocarbon-impregnated black rock salt shows that it has repeatedly lost and then regained its sealing capacity. The black staining is caused by intragranular microcracks and grain boundaries filled with solid bitumen formed by the alteration of oil. The same samples show evidence for crystal plastic deformation and dynamic recrystallization. Subgrain-size piezometry indicates a maximum differential paleostress of less than 2 MPa. Under such low shear stress, laboratory-calibrated dilatancy criteria indicate that oil can only enter the rock salt at near-zero effective stresses, where fluid pressures are very close to lithostatic. In our model, the oil pressure in the carbonate reservoirs increases until it is equal to the fluid pressure in the low but interconnected porosity of the Ara Salt plus the capillary entry pressure. When this condition is met, oil is expelled into the rock salt, which dilates and increases its permeability by many orders of magnitude. Sealing capacity is lost, and fluid flow will continue until the fluid pressure drops below the minimal principal stress, at which point rock salt will reseal to maintain the fluid pressure at lithostatic values.
INTRODUCTION

An essential element of a petroleum system in sedimentary basins is the presence of a seal. In the South Oman salt basin (SOSB), carbonate reservoirs (stringers) are fully enclosed in diapirs of the infra-Cambrian Ara Salt. This rock salt forms the top, side, and bottom seal for major hydrocarbon accumulations. In core samples directly above and below some stringers, the rock salt is stained black by solid hydrocarbons (Mattes and Conway Morris, 1990), which suggests that oil once flowed from the reservoirs into the rock salt.

Rock salt is known as the best seal for hydrocarbon accumulations based on three key properties. First, the near-isotropic stress state provides resistance to hydrofracturing. Thus, under most conditions, rock salt will hydrofracture under a higher fluid pressure than shale does (Hildenbrand and Urai, 2003) because the minimum principal stress ($\sigma_3$) is higher. Second, in-situ permeability and porosity of rock salt are already very low at a burial depth of merely 70 m (229 ft) (Casas and Lowenstein, 1989). Third, plastic deformation of rock salt in nature is ductile and, therefore, nondilatant (e.g., Ingram and Urai, 1999; Popp et al., 2001). This is reflected by Downey’s (1984) widely accepted ranking of seals: salt → anhydrite → kerogen-rich shale → clay shale → silty shales → carbonate mudstone → chert. However, under suitable conditions, all rocks can lose their sealing capacity. For engineering (short-term) applications, the criteria for leakage through rock salt have been defined experimentally (Urai, 1983; Fokker et al., 1995; Popp et al., 2001; Lux, 2005). However, the geological conditions for seal loss of rock salt are not well understood.

Although the black rock salt samples from the SOSB have been noticed for at least 15 yr, a detailed analysis of this staining has not been done; as far as we are aware, this is the first field study of a leaky rock salt top seal.

The scope of this article is to decipher the microstructural evolution of the hydrocarbon-impregnated rock salt to quantify the mechanisms by which rock salt in the deep subsurface is able to leak and resal and to generalize these results to rock salt seals in petroleum systems.

MECHANICAL AND TRANSPORT PROPERTIES OF ROCK SALT

Experimental determination of the mechanical properties of rock salt and extrapolation of these data to conditions of slow geological deformation have provided a solid basis for understanding rock salt rheology (e.g., Urai et al., 1986; Wawersik and Zeuch, 1986; Spiers et al., 1990; Carter et al., 1993; Peach et al., 2001). On the basis of these experiments, it is possible to differentiate the deformation mechanisms in rock salt based on their characteristic microstructure (Urai et al., 1987; Peach and Spiers, 1996; Schléder and Urai, 2005; Ter Heege et al., 2005).
Compared with other rock types, rock salt contains only trace amounts of H$_2$O, but this greatly enhances fluid-assisted grain-boundary migration and pressure solution (Urai, 1983; Urai et al., 1986; Spiers et al., 1988; Peach et al., 2001; Schenk and Urai, 2004; Ter Heege et al., 2005). Schenk and Urai (2005) have shown how, during grain-boundary migration, fluid inclusions interact with the moving grain boundary, and that this process strongly affects rheological properties.

In-situ permeability of undisturbed rock salt is about $10^{-21}$ m$^2$ ($10^{-20}$ ft$^2$) (Bredehoeft, 1988; Peach and Spiers, 1996; Popp et al., 2001). This low permeability allows rock salt to seal large hydrocarbon columns and fluid pressure cells over geologic time. Two processes are known to increase permeability. The first is microcracking and associated dilation (Peach and Spiers, 1996; Popp et al., 2001), and the second is the formation of topologically connected brine-filled pores and triple-junction tubes in halite grain aggregates at a pressure and temperature corresponding to depths greater than 3 km (1.8 mi) (Lewis and Holness, 1996).

**Microcracking of Rock Salt**

In experimental studies on the evolution of permeability in rock salt (Urai, 1983; Peach and Spiers, 1996; Lux, 2005), confining pressure clearly affects the fluid-transport properties of rock salt. Fluid-assisted dynamic recrystallization is inhibited at confining pressures less than or equal to 3 MPa at 150°C because of microcracking and grain-boundary disruption (Peach et al., 2001) and an associated increase in permeability of up to six orders of magnitude.

The dilatancy boundary in rock salt can be defined in the differential stress versus confining pressure space. This boundary separates a compaction domain from a dilatancy domain, where permeability begins (Peach et al., 2001; Popp et al., 2001). Dilation of rock salt is well documented in the walls of underground openings in salt caverns, but it is rapidly suppressed a few meters from the gallery walls. Fokker et al. (1995) considered the long-term evolution of permeability in the roof of abandoned solution-mining caverns and showed that when fluid pressures in the cavern approach lithostatic levels caused by creep convergence, the roof of the rock salt cavern undergoes diffuse dilation, allowing the slow escape of the fluids. Creep convergence occurs if the rock salt has an insufficient confinement (i.e., bonding strength and, thus, nonuniform internal stresses) such as the walls of underground cavities, resulting in dilation by microcracking or fracturing, possibly affecting the integrity of a salt mine.

In triaxial deformation experiments of Lux (2005), rock salt became permeable by the formation of grain-boundary cracks at low rates of fluid-pressure increase. If the fluid pressure ($P_f$) is increased rapidly to a sufficiently high excess pressure ($P_f > \sigma_3$), rock salt leaks by hydrofracturing.

Peach and Spiers (1996) proposed that this process could also occur under low effective stress during natural deformation of rock salt at great depth and high pore-fluid pressures.

**Pore-Fluid Connectivity in Rock Salt**

A different process, which produces permeability of halite by reequilibration, was proposed by Lewis and Holness (1996), who measured the equilibrium water-halite dihedral angles at grain-boundary triple junctions in experiments. If the dihedral angle is higher than 60°, small amounts of pore fluids in the halite are distributed in micrometer-size isolated fluid inclusions, and the permeability is zero. At temperatures and pressures corresponding to a depth of approximately 3 km (1.8 mi) in a normal geothermal gradient, the dihedral angle decreases to 60°, leading to a redistribution of the pore fluid into a thermodynamically stable three-dimensional (3-D) network of connected, fluid-filled channels at grain-boundary triple junctions (Figure 1).

At sufficiently high porosity, Lewis and Holness (1996) inferred a marked increase in permeability to a value of about $10^{-16}$ m$^2$ ($10^{-15}$ ft$^2$) (compared with $10^{-21}$ m$^2$ [$10^{-20}$ ft$^2$] after Bredehoeft, 1988; Peach and Spiers, 1996; Popp et al., 2001). They proposed that rock salt at depths greater than 3 km (1.8 mi) has an interconnected porosity, permitting the passage of brine. If halite contains several percent of brine in connected triple-junction tubes, and this brine is at pressures less than lithostatic, halite should compact and expel some of the brine, thereby decreasing its permeability. This will lead to an interconnected, brine-filled porosity along the triple-junction tubes with a near-lithostatic fluid pressure. Using a modified Carman-Kozeny equation of Guéguen and Dienes (1989), halite aggregates under the conditions mentioned above, with an average grain size of 5 mm (0.2 in.) and triple-junction tubes with a radius of 0.05 μm, can have a porosity of 0.01% and a permeability of about $3 \times 10^{-20}$ m$^2$ ($3 \times 10^{-19}$ ft$^2$).
**GEOLOGICAL SETTING**

**Tectonostratigraphic Evolution of the South Oman Salt Basin**

In the deep subsurface of interior Oman, three sedimentary basins developed in the Neoproterozoic to Early Cambrian on crystalline basement with radiometric ages between 870 and 740 Ma (Hughes Clarke, 1988). The basement is overlain by the Huqf Supergroup, which comprises volcanic and siliciclastic rocks (the early Vendian Abu Mahara Group), carbonates with intercalations of sandstone (the late Vendian Nafun Group), and a carbonate to evaporite sequence (the infra-Cambrian Ara Group) (Gorin et al., 1982; Amthor et al., 2003) (Figure 2).

The Ara Group consists of marine platform sediments, representing at least six third-order cycles of carbonate-evaporite sedimentation, of up to 4 km (2.5 mi) thickness (Mattes and Conway Morris, 1990). Each cycle is characterized by sedimentation of up to 1000 m (3280 ft) Ara Salt at very shallow-water depths, followed by the deposition of 20–250-m (66–820-ft)-thick isolated carbonate platforms (the so-called stringers) during transgressive periods (Mattes and Conway Morris, 1990; Amthor et al., 2005; Schröder et al., 2005).

These cycles are called A0 to A6 from bottom to top, whereby cycle A2 comprises carbonate stringer A2C and the associated evaporite interval A2E (Al-Siyabi, 2005). Intercalated sulfates can be a few meters to 20 m (66 ft) thick and form floor and/or roof anhydrite. The Huqf Supergroup (Figure 2) is overlain by thick continental clastics of the Haima Supergroup (Cambrian to Ordovician). The earliest halokinesis of the Ara Salt was most likely triggered by differential loading during the continental deposition of the Haima
siliciclastics onto the mobile Ara Salt, leading to passive diapirism during the Cambrian–Ordovician (Heward, 1990; Loosveld et al., 1996). Most of the carbonate stringers contain fluids at very high overpressures as shown by repeat formation tester (RFT) data (Mattes and Conway Morris, 1990; Al-Siyabi, 2005). Data on in situ stress in the carbonate stringers were obtained by leak-off pressure (LOP) delineating a minimum principal stress ($\sigma_3$) envelope with a gradient of 22 kPa/m (Figure 3).

The carbonate stringers have undergone intense diagenetic modifications with locally extensive cementation by halite and solid bitumen (e.g., stringer A1C of well Minassa-1H1 [Petroleum Development Oman LLC]; Al-Siyabi, 2005; Schoenherr et al., 2007). The stringers have been interpreted to be a self-charging system with first oil charge from mature type I/II source rocks (Peters et al., 2003) during the Early Cambrian to Ordovician at maximum burial temperatures after deposition of the Haima Supergroup (Visser, 1991; Terken et al., 2001). In addition, geochemical studies suggest external (pre-Ara deposition) oil and gas charge (Al-Siyabi, 2005). The present-day temperature profile of the SOSB corresponds to a geothermal gradient of $18–20^\circ$C/km, with temperatures between 90 and 110$^\circ$C in the depth range of 3500 to 4500 m (11,482 to 14,763 ft) sampled for this study.

**Sampled Core Intervals**

Samples were taken from cores in the greater Birba and the greater Harweel area, which are approximately 100 km (62 mi) apart (Figure 2). Six samples from

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**Figure 2.** Schematic map of the salt basins of interior Oman (stippled outlines). Rock salt samples studied are from the greater Birba and the greater Harweel area in the SOSB (modified from Qobi et al., 2001). The cross section AB illustrates Ara Salt diapirs and enclosed Ara carbonate stringer reservoirs. The infra-Cambrian Ara Group is overlain by thick continental clastics of the Haima Supergroup, which formed pronounced pods leading to diapirism (modified from Peters et al., 2003). Note that the legend in the cross section also defines the patterns in the chronostratigraphy.
four wells have been investigated for this study from both carbonate stringer and Ara Salt intervals (Table 1; Figure 4).

From the Birba North-1H1 well (Petroleum Development Oman LLC), three samples (B9, B10, and B11) were studied from a 10-m (33-ft)-thick heavily hydrocarbon-stained Ara Salt interval between stringer A5C and stringer A4C (Figure 4a), which is at near-hydrostatic fluid pressures at present (Figure 3). Wells Reef-1H1, Minassa-1H1, and Sarmad-2H1 (Petroleum Development Oman LLC) are located in the greater Harweel area. Sample B57 (Reef-1H1) is from a damage zone, a 6-m (20-ft)-thick interval showing halite-plugged pores and fractures in stringer A2C (Figure 4b), which is probably overpressured. Another sample comes from a rock salt vein (B16) in stringer A1C of well Minassa-1H1 (Figure 4c), which is overpressured (Figure 3). Sample B2 derives from a clear Ara Salt interval of well Sarmad-2H1.

**METHODOLOGY**

Circular rock salt plugs with a diameter of approximately 3 cm (1.2 in.) were taken perpendicularly to the core axis and the long axis of the grain-shape preferred orientation, respectively (Figure 4a). Slabs with a thickness of 1.5 cm (0.6 in.) were cut from the rock salt plugs using a diamond saw with small amounts of water to avoid microcracking (Schlèder and Urai, 2005).

Samples were gamma-irradiated for 3 months at 100°C with a dose rate between 4 and 6 kGy/h.
(kilograms per hour) to a total dose of about 4 MGy (megaga... at the research reactor of the research center in Jülich, Germany, to highlight otherwise invisible microstructures. The color intensity observed in most rock salt samples is heterogeneous, reflecting an irregular distribution of solid-solution impurities and crystal defects within halite grains (van Opbroek and den Hartog, 1985; Schleder and Urai, 2005).

From unirradiated and irradiated samples, thin sections were prepared using the method described by Urai et al. (1987) and Schleder and Urai (2005). The slabs were polished dry on grinding paper, etched with distilled water for 2 s, quickly dried with a tissue, then dried in a stream of hot air. This etching technique removes scratches and provides a microrelief on the surface of the slabs, improving the stability of mounting. The slabs were mounted on glass plates at room temperature using epoxy (Körapox 439) and cut with small amounts of water into thick sections of about 4 mm (0.16 in.). After drying, the thick sections were ground down to a thickness of about 1 mm (0.04 in.) with grinding paper (dry) and finally etched in a slightly undersaturated NaCl solution (~6 M) for 10 s, with the etchant flushed from the thin-section surface by a powerful jet of n-hexane followed by drying in a stream of hot air.

For study of grain-boundary morphology in 3-D, we used the procedure of Urai et al. (1987). Slabs of $10 \times 10 \times 5$ mm ($0.4 \times 0.4 \times 0.2$ in.) were cut out of a hydrocarbon-rich area within the sample, which was then carefully broken along the slab surfaces and coated with gold to differentiate carbon-rich phases during energy-dispersive x-ray (EDX) measurement in a Zeiss DSM-962 scanning electron microscope.

RESULTS

Grain Fabrics and Grain-Boundary Geometry

Grain Fabrics

Grain fabrics in most rock salt samples are characterized by grains of 2–8 mm (0.08–0.31 in.) in diameter with about 0.1-mm (0.004-in.)-size polygonal subgrains and lobate grain boundaries intersecting at T-shaped triple junctions (Figure 5). A strong shape-preferred orientation is present in samples B9 and B10 with strong aspect ratios of up to 3. The black rock salt sample B9 shows grains with cloudy dark cores surrounded by clearer rims toward brownish grain boundaries (Figure 5a). In contrast, sample B10 shows clear grains but also dark grain boundaries (Figure 5b).

Rock salt samples B11 and B16 are coarse grained, with grain diameters of 5–30 mm (0.2–1.2 in.) and gently curved grain boundaries intersecting at 120° triple junctions (Figure 5c, d). Like sample B11, sample B16 is clear overall, with some grains containing cubic, 80–300-μm (3.2–12-in.)-size solid bitumen inclusions (Figure 5d).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Well</th>
<th>Depth (m)</th>
<th>Lithostratigraphy</th>
<th>Solid Bitumen*</th>
<th>Formation Pressures**</th>
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<td>-</td>
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<td>3662.20</td>
<td>Top A5C</td>
<td></td>
<td>Unknown</td>
</tr>
<tr>
<td>-</td>
<td>Birba North-1H1</td>
<td>3662.20</td>
<td>Top A5C</td>
<td></td>
<td>Unknown</td>
</tr>
<tr>
<td>B09</td>
<td>Birba North-1H1</td>
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<td>ARA Salt</td>
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<tr>
<td>B10</td>
<td>Birba North-1H1</td>
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<td>ARA Salt</td>
<td>ic, mc, and gb</td>
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</tr>
<tr>
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<td>Birba North-1H1</td>
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<td>ARA Salt</td>
<td>ic and gb</td>
<td>Unknown</td>
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<td>-</td>
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<td>3680.00</td>
<td>Top A4C</td>
<td></td>
<td>OP</td>
</tr>
<tr>
<td>-</td>
<td>Minassa-1H1</td>
<td>3375.00</td>
<td>Top A1C</td>
<td></td>
<td>OP</td>
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<tr>
<td>B16</td>
<td>Minassa-1H1</td>
<td>3412.35</td>
<td>A1C halite vein</td>
<td>ic</td>
<td>OP</td>
</tr>
<tr>
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<td>Minassa-1H1</td>
<td>3445.00</td>
<td>Base A1C</td>
<td></td>
<td>OP</td>
</tr>
<tr>
<td>-</td>
<td>Reef-1H1</td>
<td>3540.00</td>
<td>Top A2C</td>
<td></td>
<td>OP</td>
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<tr>
<td>B57</td>
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<td>A2C halite vein</td>
<td>oil at mc</td>
<td>OP</td>
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</tr>
<tr>
<td>-</td>
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<td></td>
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<tr>
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<td>Sarmad-2H1</td>
<td>?</td>
<td>Base Ara Salt</td>
<td></td>
<td>Unknown</td>
</tr>
</tbody>
</table>

*Abbreviations describe the occurrence of solid bitumen in the samples studied; ic = intracrystalline; mc = microcracks; gb = grain boundary.
**NP = normally pressured; OP = overpressured.
Grain-Boundary–Related Structures

Grain boundaries in halite commonly contain arrays of fluid inclusions (Roedder, 1984; Urai et al., 1987). In the dark halite, grain boundaries contain a brownish, solid film up to 0.1 mm (0.004 in.) thick, with holes mimicking the fluid-inclusion pattern (Figure 6a). The brownish solid is a bright, reflective, carbon-rich phase, solid bitumen (Figure 6a, b). In the dark halite, all grain boundaries contain a continuous layer of solid bitumen, which in turn is locally fractured (Figure 6c). Dissolution of sample B9 yields a residue of 1.2 wt.% solid bitumen, with a mean random solid bitumen reflectance (BRr) of 0.35%, indicating a paleotemperature of about 95°C (Barker and Pawlewicz, 1994). An interesting microstructure is formed in sample B9 by arrays of solid bitumen particles parallel to the grain boundary with a long axis between 10 and 100 μm and a dropletlike shape with tapering terminations toward a fluid-inclusion–rich grain boundary (Figure 6d).

Intragranular Fluid Inclusions

Transmitted light microscopy of sample B2, which does not contain solid bitumen, shows abundant intracrystalline fluid inclusions in some subgrain-rich grains.
The fluid inclusions are concentrated in bands, which alternate with fluid-inclusion–poor to clear bands, respectively. These grains are anhedral in shape and are separated from clear halite grains by lobate grain boundaries containing networks of brine-filled tubes and isolated fluid bubbles.

Hydrocarbon-Impregnated Microcracks
In addition to solid bitumen in grain boundaries, most dark rock salt samples contain intragranular microcracks filled with solid bitumen (Figure 8a).

In places, partly healed microcracks are outlined by cube-shaped brown oil and brine inclusions, which are 10–50 µm in size (Figure 8d, sample B57). These inclusions are located between gas-filled tubes and in places contain a gas bubble (see detail of Figure 8b).

Subgrain Size Piezometry
In all samples, the halite crystals contain well-developed, 40–300-µm-size subgrains (Figure 9a). Using the experimentally calibrated relationship between differential stress ($\sigma_1 - \sigma_3$ in megapascals) and subgrain size ($D$ in micrometers) from the combined data set of Carter et al. (1993) and Franssen (1993),

$$D = 107(\sigma_1 - \sigma_3)^{-0.87}$$ (1)
we estimated $\sigma_1 - \sigma_3$ in four samples (Table 2) following the method of Schléder and Urai (2005). Boundaries of the polygonal subgrains were interpreted in micrographs taken in reflected light. Then, the equivalent circular diameter (ECD) of the digitized outlines was analyzed using the ImageJ software of Abramoff et al. (2004) (Figure 9b), and by taking $D = \text{ECD}$, we calculated $\sigma_1 - \sigma_3$. The results are shown in Table 2.

**Interpretation and Discussion**

Based on the observations presented, we discuss a model for the microscale processes by which oil entered the Ara Salt (stage 1), followed by resealing of the rock salt (stage 2). Subsequently, the oil was converted into solid bitumen, which deformed with the deforming salt (stage 3). Renewed dilation led to a second phase of oil flow into the rock salt (stage 4).

**Initial Situation**

Before oil impregnation, the Ara Salt acted as a perfect seal for the hydrocarbons. Deep burial of the stringers generated oil during the Ordovician (Visser, 1991; Terken et al., 2001). Initially, the oil pressure ($P_{\text{oil}}$) in the stringer reservoirs was lower or equal to the fluid pressure in the rock salt, which has an interconnected network of brine-filled triple-junction tubes with a pore fluid pressure $P_b$ (pressure of brine) close to the minimum principal stress ($\sigma_3$) (Lewis and

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**Figure 6.** Grain-boundary microstructures of Ara Salt. (a) Transmitted light micrograph of gamma-irradiated sample B11 shows gently curved grain boundaries, filled with solid bitumen. The detailed view of the triple junction in reflected light shows bright reflective solid bitumen. (b) Continuous layer of solid bitumen (confirmed by EDX analysis) in grain boundaries (white arrow). Scanning electron microscopy image of a broken piece of sample B11. (c) Steeply dipping grain boundaries (to three sides) covered by brownish solid bitumen. Note the dark fissures in solid bitumen (transmitted light micrograph of sample B9). (d) Micrograph of a steeply dipping fluid-inclusion–rich grain boundary in the upper part. In the lower part, droplet-shaped particles of solid bitumen are aligned parallel to the grain boundary (transmitted light; sample B9).
Holness, 1996). Under these conditions, rock salt was a perfect seal.

The partially recrystallized (anhedral) fluid-inclusion–rich crystals observed (sample B2, Figure 7) are interpreted as syndepositional (Benison and Goldstein, 1999; Schléder and Urai, 2005). These primary fluid inclusions were added to the pore fluid during grain-boundary migration (Drury and Urai, 1990). During this process, the intracrystalline fluid inclusions accumulated at the (migrating) grain boundaries and triple junction tubes into continuous fluid films and wormlike tubes, respectively (Figures 6d, 7) (Urai et al., 1987). In addition, the rock salt samples show abundant subgrains (Figures 5c, 9), indicating a maximum past differential
stress \((\sigma_1 - \sigma_3)\) of less than 2 MPa (Table 2), which agrees well with a worldwide data set of rock salt stresses (Carter et al., 1993; Franssen, 1993). This means that the overburden stress \((\sigma_o)\) is very close to \(\sigma_3\).

**Stage 1: Diffuse Dilation of the Ara Salt**

The microstructure of the hydrocarbon-impregnated Ara Salt shows clear evidence for dilation by diffuse grain-boundary microcracking and intragranular microcracking as opposed to hydrofracturing (Lux, 2005). The phase impregnating the rock salt must have been oil, which is inferred from investigations of Jacob (1989), who demonstrated that solid bitumen with a reflectance between 0.3 and 0.7% (grahamite), such as the extracted solid bitumen from sample B9 with a value of 0.35%, starts to flow at a temperature of above 160°C. In our model, the sampled rock salt interval of Birba North-1H1 did not undergo this temperature after solid bitumen was formed.

The experimentally derived dilatancy criterion by Popp et al. (2001) indicates that at a differential stress of less than 2 MPa, rock salt can dilate only at near-zero effective stresses (Figure 10), where fluid pressure in the rock salt is very close to \(\sigma_3\).

When the oil pressure is equal to the brine pressure in the rock salt, the capillary pressure still has to be overcome to allow entry of oil through the reservoir-seal interface into the pores of the halite crystals (Figure 11a, b).

The capillary pressure \((P_c)\) across an oil-brine meniscus in the pore throats of the halite crystals can be calculated using the equation of Washburn (1921):

\[
P_c = \frac{2\gamma \cos \theta}{r}
\]

where \(P_c\) is the capillary pressure (pressure difference across the oil-brine interface; in megapascals), \(\gamma\) is surface tension (0.025 N/m after Hocott, 1938), \(r\) is the

**Table 2. Maximum Past Differential Stress of Ara Salt Samples Calculated from Subgrain Size**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Number of Analyzed Subgrains</th>
<th>Mean Subgrain Diameter (µm)</th>
<th>Standard Deviation</th>
<th>Calculated Mean Differential Stress in MPa (95% Confidence Interval)</th>
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<tbody>
<tr>
<td>B09</td>
<td>229</td>
<td>100</td>
<td>77.58</td>
<td>1.94</td>
</tr>
<tr>
<td>B10</td>
<td>304</td>
<td>106</td>
<td>75.18</td>
<td>1.85</td>
</tr>
<tr>
<td>B11</td>
<td>202</td>
<td>141</td>
<td>116.45</td>
<td>1.44</td>
</tr>
<tr>
<td>B57</td>
<td>86</td>
<td>124</td>
<td>67.74</td>
<td>1.61</td>
</tr>
</tbody>
</table>

The experimentally derived dilatancy criterion by Popp et al. (2001) indicates that at a differential stress of less than 2 MPa, rock salt can dilate only at near-zero effective stresses (Figure 10), where fluid pressure in the rock salt is very close to \(\sigma_3\).
pore-throat radius, and ϑ is the wetting angle (0°, assuming the rock salt to be perfectly water wet). The capillary entry pressure condition, at which the oil displaces the brine in the halite triple junction tubes, is then described by

\[ P_b \approx \sigma_3 \quad \text{and} \quad P_{\text{oil}} \approx \sigma_3 + P_c \]  

(3)

The sealing capacity of rock salt is exceeded as soon as this condition is met. Assuming a pore-throat radius of 0.05 μm (Schenk and Urai, 2004; Schenk et al., 2006), the value of capillary pressure \( P_c \) becomes 0.1 MPa. At this pressure, oil will displace the brine in the triple junction tubes and in the wormlike grain-boundary inclusions, which in turn dilates the halite grain boundaries, leading to diffuse dilation of the halite grain fabric (Figure 11c).

Dilation of rock salt after the initial influx of oil results in a major increase in permeability by grain-boundary opening. At this point, Terzaghi’s (1923) law applies to the rock salt, and the effective stress, which is \( \sigma' = \sigma_3 - P_b \), becomes very close to zero. Based on the results of Lux (2005), we infer that the oil pressure did not rise to significantly higher values because this would have generated discrete hydrofractures instead of diffuse dilation.

Stage 2: Resealing of the Ara Salt

The microstructures show that the Ara Salt compacted and (dynamically) recrystallized after oil impregnation. The microscale deformation mechanisms (dislocation creep and fluid-assisted grain-boundary-migration recrystallization) continued after the oil pressure in the stringers dropped to values equal to \( \sigma_3 \) in the rock salt (initial situation). It has been shown experimentally that oil-filled halite grain boundaries are not mobile (Urai, 1983). Movement of oil with the grain boundary implies that these were filled with a two-phase fluid, oil and brine (Figure 12b). Under suitable conditions...
of fluid film structure and grain-boundary driving force (Urai et al., 1986; Schenk and Urai, 2005), the migrating grain boundary can leave behind oil inclusions in the growing crystal (Figure 12c). As grain-boundary migration stops because of the lack of the driving force, the oil inclusions in the grain boundary are rearranged into cigar-shaped inclusions or tubes together with the grain-boundary brine (Figure 12d). This explains how the hydrocarbons, which entered the rock salt along dilatant grain boundaries and intragranular microcracks,
are now found both in the grain-boundary network and in the recrystallized halite as ghost grain boundaries (Figure 6d).

Stage 3: Conversion of Oil into Solid Bitumen

After the oil was redistributed in the Ara Salt as discussed above, it was converted into solid bitumen. The processes leading to the conversion of oil into solid bitumen in the Ara Group are described in detail by Schoenherr et al. (2007). Solid bitumen reflectance (BR\textsubscript{r}) measurements on sample B9 indicate a paleotemperature of 95°C (Barker and Pawlewicz, 1994). The out-of-equilibrium shapes of solid bitumen inclusions (Figure 6d) may be caused by the lack of brine in some leftover oil inclusions in the growing grain (Schenk and Urai, 2005). Otherwise, the inclusions would be cubic as observed in sample B16 (Figure 5d). The oil inclusions were converted in situ into solid bitumen, a process that releases gas. However, the solid bitumen inclusions do not show any gas inclusions or gas-filled space (e.g., Figures 5d, 6d), so further research is required to determine the fate of the gas.

Fractured solid bitumen in halite grain boundaries (Figure 6c) indicates continued deformation of the Ara Salt after solid bitumen formed. This stage of salt deformation was less intense and ceased directly after conversion; otherwise, this solid bitumen would be more intensively deformed as shown in Figure 12d.

Stage 4: Renewed Dilation of Ara Salt

A second dilation stage of the Ara Salt is indicated in sample B57 (Figure 4b). Whereas the first oil was converted to solid bitumen, oil still exists as fluid inclusions in healed cracks of the halite crystals (Figure 8b). This indicates that these oil inclusions entered the Ara Salt after conversion of the first oil to solid bitumen in the carbonate matrix. Thus, we interpret that these rock salt samples underwent a second phase of dilation; otherwise, the oil inclusions would have been converted to solid bitumen as observed in sample B16 (Figure 5d). Assuming that oil was converted into solid bitumen more or less at the same time within the Ara Group, the Ara Salt must have dilated in multiple stages,

Figure 12. A schematic image sequence shows the possible evolution of the observed microstructures as oil is redistributed during dynamic recrystallization of halite. (a) Oil films flow along the grain boundaries of halite caused by microcracking. This process causes a diffuse dilation in the salt as shown in Figure 11c. Gray frame represents Figure 11b. (b) The oil- and brine-filled grain boundary starts to migrate because of grain-boundary migration recrystallization into the dislocation-rich grain (indicated by arrows). (c) Illustration shows the next stage of the evolution of oil in the grain boundary (gb) in detail. The oil is left behind a migrating brine-filled grain boundary, whereby it gets dragged and left behind because of the force of the migrating grain boundary. (d) Three-dimensional view at a grain boundary (shaded in gray), which dips to the top of the image (stippled line is the lower termination of grain boundary). White bubbles represent brine inclusions, which contracted in the grain boundary into spherical-shaped inclusions as grain boundary migration recrystallization stopped. The oil left behind the grain boundary was transformed into solid bitumen, which forms a ghost grain boundary parallel to the fluid-inclusion–rich grain boundary as shown in Figure 6d.

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when the fluid pressure increased again to trigger renewed dilation of the rock salt. At this stage, it is important to keep in mind that after the condition for oil entry (equation 3) is met and rock salt starts to leak, the seal will close up at the point when the oil pressure drops to just a few tenths of a megapascal below $\sigma_3$, so that the reservoir pressure remains near lithostatic. Only a small increase is required to initiate another phase of leakage and diffuse dilation.

Interestingly, the pressure data of stringer A4C of well Birba North-1H1, which is located directly below the black salt interval studied, indicate near-hydrostatic conditions (see Figures 3, 4). This can be explained by the dissipation of the overpressure caused by contact with the adjacent (permeable) Haima clastics after a period of hard overpressure in the past.

**Application for Seal Assessment of Rock Salt**

The black Ara Salt samples provide the first case study showing that rock salt is able to lose its seal capacity for oil at depth. Our results show the limits to the sealing capacity of rock salt. Leakage conditions can be readily incorporated into basin-modeling algorithms. These conditions are not restricted to the Ara Salt and apply to all other rock salt top seals worldwide.

For the SOSB, our study provides a basis to speculate whether oil flowed through the rock salt matrix into the overburden (Haima clastics) and the potential volumes involved. This depends on the amount of oil generated in a stringer and the proportion of this required to raise the reservoir pressure to lithostatic, after which all excess oil would leave the stringers and flow into the Haima clastics or into the overlying stringer. Further quantification of this process requires a basin-modeling study.

**CONCLUSIONS**

- The black rock salt cores from the SOSB clearly indicate that distinct parts of the Ara Salt lost sealing capacity for oil.
- Microstructures indicate a complex process of dilation and resealing for oil with associated dynamic recrystallization, conversion of oil into solid bitumen, and continued salt deformation.
- Based on subgrain size piezometry, the maximum past differential stress in the Ara Salt around the Ara carbonate reservoirs was less than 2 MPa.
- Oil initially leaked into the salt when oil pressure in the reservoir exceeded the near-lithostatic fluid pressures of the Ara Salt by a few tenths of a megapascal to overcome the capillary entry pressure, followed by diffuse dilation and marked increase in permeability, in agreement with laboratory-calibrated dilation criteria of rock salt.
- Oil could flow into the salt as long as the oil pressure remained larger than the minimum principal stress in the rock salt.
- The conditions inferred in this study represent the ultimate sealing potential of rock salt in the deep subsurface and can be applied to rock salt seals in any tectonic setting.

**REFERENCES CITED**


