Relating natural heterogeneities and rheological properties of rocksalt: New insights from microstructural observations and petrophysical parameters on Messinian halites from the Italian Peninsula

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A B S T R A C T

The importance and economic interest of rocksalt as well as its influence on tectonics and applicative purposes such as mining, hydrocarbons extraction, and nuclear waste storage are well known. Careful characterization of physical and chemical properties of rocksalt is fundamental as the rocksalt behavior may influence its potential use for applicative purposes. Mechanical and rheological properties of rocksalt have been extensively studied in the past. However, the role of natural heterogeneities within rocksalt and their effect on salt rheology have not been investigated quantitatively. Here we present a comprehensive salt facies study on Messinian rocksalt from several Italian sites (Volterra Basin, Tuscany, Caltanissetta Basin, Sicily and Crotone Basin, Calabria). Four salt facies end members have been identified and analyzed by optical analyses. The main facies-defining characteristics resulted to be the primary salt crystal abundance, crystal size, roundness and orientation, as well as the clay inclusion contents. Three out of four facies were placed on an evolutionary path from an “immature,” with respect to the deformation history, to a “mature,” rocksalt. So we observed, with increasing rocksalt maturity, a progressive disappearing of primary crystal remnants, increasing crystals elongation and iso-orientation and decreasing in crystal size. This trend has been confirmed by differential stress calculations from subgrain size. Through seismic waves velocity measurements and uniaxial compressive runs, specific salt facies were tested. Results of the investigations demonstrate that the facies parameters have a distinct influence on the rocksalt petrophysical parameters like P- and S-waves velocity, dynamic and static Young Modulus, elastic limit, and strain at peak. Finally, this study allowed to suggest the subdivision of Volterra’s salt sequence in three different units that have been subjected to variable deformation degree in response to the different salt characteristics.

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1. Introduction

The importance and economic interest in rocksalt deposits and salt bodies are well known and extensively studied (see Hudec and Jackson, 2007 and references therein). The physical and rheological properties of rocksalt have a profound influence on the tectonics and they are considered to be crucial for understanding the mechanical response of geo-sites for applicative purposes such as mining, petroleum, and nuclear waste storage (Hunsche and Hampel, 1999). However, the majority of the literature uses the descriptive term “salt” for all rocks composed mostly of halite, despite the paucity of pure halite sequences in nature (Hudec and Jackson, 2007, and references therein).

“Pure” rocksalt itself cannot be considered as an ideal isotropic rock made up of salt crystals with the same characteristics (Pouya, 1991). In fact, rocksalt is mainly constituted by two types of halite crystals: primary and secondary. Primary “cloudy” crystals are developed by precipitation from water and/or overgrowth at the bottom of water body (Lowenstein and Hardie, 1985). They appear white/gray to the naked eye and opaque when looking with a microscope in transmitted light. This is due to the countless cubic-shaped and randomly distributed primary fluid inclusions contained in primary crystals (Speranza et al., 2013). Growth bands and shapes like chevrons, cubes, and hoppers are typical primary salt features. Secondary “clear” crystals derive from primary crystals that are dissolved and deposited or recrystallized (Lowenstein and Hardie, 1985, and references therein). Secondary crystals appear transparent under the microscope due to the progressive dissolution of the “cloudy” crystals in favor of secondary salt with lower fluid inclusions content. However, not all the fluids escape the
mineral. Secondary fluid inclusions, gas- or water-filled, are always present at crystal grain boundaries and in healed fractures as the result of fluid transfer during deformation/recrystallization processes (Peach and Spiers, 1996; Pennock et al., 2005, 2006; Schléder and Urai, 2005; Ter Heege et al., 2005a, b; Urai and Spiers, 2007).

It is well known in fact that halite is prone to deformation, even early after deposition and under weak tectonic stresses (Carter and Hansen, 1983; Carter et al., 1993; Franssen, 1994; Hunsche and Hampel, 1999; Senseny et al., 1992; Ter Heege et al., 2005a). During deformation/recrystallization processes, the shape and dimension of rocksalt crystals can be easily changed and other structural anisotropy features can be induced by deformation in a salt body (Dubey and Gairola, 2000; Dubey and Gairola, 2008; Lebensohn et al., 2003; Lloyd et al., 1997; Piazolo et al., 2006). Repeated changes of stress conditions will also affect the mechanical properties of rocksalt (Fuenkajorn and Phueakphum, 2010). Therefore, not only the salt facies characteristics but also the deformation history may exert important influence on the rheological behavior of rocksalt and therefore has to be taken into account.

Previous studies on halite mechanical properties and rheological behavior have been mostly based on synthetic rocksalt or commercial salt (e.g. Franssen, 1994; Peach and Spiers, 1996; Pennock et al., 2006; Spiers et al., 1990; Ter Heege et al., 2005a, b), which are usually devoided of heterogeneities and impurities. To our knowledge, the role of natural heterogeneities and their effect on salt rheology have never been quantitatively investigated. Moreover, non-evaporitic rocks as clay that can be found dispersed in saltrocks may inﬂuence the overall rheology of a “salt” body.

The objective of this investigation is to fill this substantial gap of knowledge, by studying the microstructural and mechanical characteristics of a natural rock salt sequence characterized by a marked salt facies variability and a signiﬁcant deformation history, such the Messinian Saline di Volterra Formation, Volterra Basin, Tuscany, Italy (Fig. 1). Our goals are to recognize and analyze quantitatively the main microstructural features characterizing Volterra’s natural rocksalt by means of thin section analysis using both transmitted/reflected light microscope and digital image analysis. Once identified the salt facies end members, we characterized the main petrophysical and mechanical parameters in laboratory by carrying out ultrasonic waves velocity measurements and uniaxial compression tests. In addition, further Messinian rocksalt samples from Petralia and Racalmuto salt mines, Caltanissetta Basin, Sicily, and from Zinga 1 and Vitravo 1 salt diapirs, Crotone Basin, Calabria, have been selected and studied by thin section analysis as terms of comparison to the salt facies end members identified from Volterra samples. Furthermore, the Saline di Volterra Formation gives us the possibility to observe and analyze the transition between the facies end members, so we can suggest an evolutionary path that relates rocksalt characteristics, degree of deformation, and its evolution and petrophysical and mechanical parameters. Our study on natural rocksalt revealed the significant variability of salt facies that can be found in nature and that different facies show their own mechanical response, suggesting that when one deals with synthetic salt, natural conditions need to be taken in account.

2. Geological settings

2.1. The Volterra Basin

The Volterra Basin is located in the western part of Tuscany (Fig. 1). It is an NW-SE elongated extensional basin opened in response to the eastward migration of the Apennine thrust-belt. The basin is ﬁlled with a sedimentary succession spanning from late Tortonian to Pliocene (Testa and Lugli, 2000), which includes an upper Messinian evaporitic succession consisting of alternating gypsum, conglomerates, sandstone, claystone, and halite. The salt deposit of the Saline di Volterra Formation is divided into several levels, with intercalations of clay, silt, laminitic anhydrite, sandstones, and microconglomerates, for a total thickness ranging from few meters to nearly 200 m. Halite represents roughly 40% of the total volume of the series, and it is characterized by moderate bending and minor diapiric movements. Gypsum, gypsenite, and microcrystalline gypsum are present in the series along with salt. The Volterra area was extensively drilled for the exploitation of salt levels by the Solvay Company. Study samples come from cores of S1113 borehole, which cuts through the rock salt for 43 m between 122 and 165 m depth. We collected 16 cores (around 30 cm long with a diameter of 11 cm) for a total thickness of 4.2 m (Fig. 2). According to Testa and Lugli (Testa and Lugli, 2000) and references therein, the Saline di Volterra Formation had undergone a maximum burial depth of about 800 m during Lower Pliocene, when the heat ﬂow in the basin area was of 85 mW/m². Considering also the conductivity of shale, that represents the majority of the sediments column overlying the salt deposit in Lower Pliocene (Testa and Lugli, 2000), a maximum burial temperature for salt of about 50–60 °C can be estimated. Since the late Early Pliocene, the emplacement of several magmatic bodies at upper crustal depth took place around the Volterra Basin area, producing a heat ﬂow increase up to 110 mW/m² (Testa and Lugli, 2000). However, the magmatic bodies’ emplacement led to basinal uplift and progressive decrease of the salt burial depth, ruling out a remarkable temperature increase compared to the maximum burial temperature.

2.2. The Caltanissetta and Crotone Basin

The Crotone Basin (Lugli et al., 2007; Roda, 1964; Zecchin et al., 2004) is a forearc basin in central Calabria, between the Sila Massif and the Ionian Sea. Its Neogene evolution is related to the Calabrian Arc and to the rollback of the Calabrian subduction zone since Serravallian time (Faccenna et al., 2004; Faccenna et al., 2007; Mattei et al., 2007; Speranza et al., 2003 and references therein). The Serravallian-to-Pleistocene sedimentary succession of the basin consists of three sedimentary cycles made up of conglomerates, sandstones, and claystone deposits, as well as a Messinian evaporitic interval that includes gypsum beds, gypsum breccias, claystones, sandstones, conglomerates, and halite. In the Crotone Basin, the halite deposit has
variable thickness (up to 200 m) and crops out in the northern part of
the basin, particularly in the Vitravo River area. There, small diapiric
structures cut through the uppermost Messinian and Lower Pliocene
claystone deposits. Samples for this study were collected from the
outcrop of the Zinga 1 and Vitravo 1 diapirs, close to the Zinga village
(Fig. 1).

The Caltanissetta Basin (Decima and Wezel, 1971; Keogh and Butler,
1999; Roveri et al., 2008) is an NE–SW elongated depression located in
south-western Sicily, consisting of a complex array of synclines that
host a Tortonian to Plio-Quaternary sedimentary succession. The folds
form a series of sub-basins that continued to deform as part of the
evolving Maghrebian orogenic wedge. The sedimentary fill of the
whole basin includes Tortonian subaerial clastic rocks, shallow marine
sands, patch reefs, hiatuses and deposits related to the Messinian Salin-
ity Crisis (MSC), such as the Lower Evaporites and the Upper Evaporites
(including halite), which are separated by a basin-scale erosional
surface (the Messinian Erosional Surface, MES). Plio-Quaternary chalk
and conglomerates complete the sedimentary filling of the basin. Halite
deposits occur as several independent bodies aligned NE–SW in a
20-km wide belt. These bodies, with an average thickness of 500 m,
are affected by intense shortening. A typical rock salt succession in the
Caltanissetta Basin shows a basal layer of breccia, followed by halite,

Fig. 2. Schematic drawing of the collected S1113 borehole cores (Volterra Basin), showing salt, clay blocks, and thin strata (black lines) and some other salt features and structures easily recognizable to the naked eye. Depths of the base and top of each core are reported. Green and blue circles specify the source and names of samples used for the petrophysical experiments. Names and sources for the thin sections are also provided. Finally, pictures of analyzed cores and salt facies end members location (colored bars) are displayed.
anhydrite, and K–Mg salts with claystone intercalations. In this study, halite samples are from the active salt mines of Petralia Sottana and Racalmuto (Fig. 1).

3. Macroscopic description of salt faces

At Volterra, from the base of the salt series (165 m of depth) to 161 m, the salt is light gray (Fig. 2, A–B). Primary “cloudy” crystals, from less than 1 mm to 1 cm in length, are very abundant. Some typical primary features, like chevron crystals and growth bands, are easily recognizable (Speranza et al., 2013). Some very thin (around 2 mm) clay strata, intercalated with salt, are visible. Between 158 and 155 m (Fig. 2, C), primary crystal abundance decreases in favor of large (up to 4 cm), secondary “clear” crystals. Particularly, a thin (around 6/8 cm) salt layer made up only of secondary, rather big and blocky crystals can be observed at a depth of around 158.6 m. Thin clay strata are also present together with small (1 mm) inclusions (Fig. 2, D). The grain size appears to be smaller with clearly dark, with very few primary crystal remnants and some little clay inclusions, around 141 m depth, the salt becomes very slightly lighter colored halite and the predominance of secondary crystals, elongated on a slightly inclined horizontal plane, formed (Fig. 2, E). Here the halite is dark gray, made up mainly of secondary crystals, elongated on a slightly inclined horizontal plane, with only few remnants of “cloudy” salt. Above 130 m of depth (Fig. 2, F–G), the clay fraction abundance decreases. Between 130 and 126 m depth, the salt fraction becomes prevalent again, with slightly lighter colored halite and the predominance of secondary crystals. Primary features are nevertheless still preserved and evident. Moreover, here, the rock salt is clearly banded, with alternating darker and lighter bands, on a dm scale, inclined by 25–35°. Namely, lighter bands show greater abundance in primary crystals compared to darker ones. At the top of the salt series (122 m depth), only a small amount of salt crystals remains as fractures-filling cement between very large and fractured clay blocks.

From these macroscopic observations, we could identify four main salt facies along the Saline di Volterra series (Fig. 2). We assigned to each facies the following roman numbers and colors:

1) Type I (green): salt with great abundance in primary crystal remnants, located at the base of the Saline di Volterra Formation (165–161 m of depth);
2) Type II (black): salt made up mainly of secondary crystals with abundant clay inclusions and some primary crystal remnants, that constitute the upper part of the salt sequence (132–126 m of depth);
3) Type III (red): salt made up of secondary crystals, with an appreciably smaller grain size, found in the middle of the salt series at about 141 m of depth;
4) Type IV (blue): salt made up of rather large and blocky crystals of secondary origin found at 158 m of depth.

Salt samples from Petralia and Racalmuto mines (Speranza et al., 2013) and from Z1 and V1 diapirs selected for comparison with Volterra series, although coming from different locations with different geological settings, appear to fit quite well in the classification scheme defined above. In particular, salt samples from Racalmuto mine and V1 diapir are very rich in primary crystal remnants and appear almost undeformed (type I); Petralia mine sample is rather comparable to the type IV salt being constituted by only secondary, slightly deformed salt crystals, although much smaller than those pertaining to Volterra series; finally, Z1 diapir salt is very highly deformed and recrystallized and so can be easily associated to the type III salt.

4. Analytical and experimental methods

Salt facies were investigated by i) microstructural analysis with Imagej software, ii) laboratory ultrasonic waves velocity measurements, and iii) uniaxial compressive tests.

4.1. Microstructural analysis

Microstructural analyses of the selected samples were performed in order to recognize and define quantitatively the most important features characterizing the rocksalt facies. Ten thin sections representative of the four main salt facies defined above were prepared (cf. Table 1). Each section has a length of 4.8 cm. Thin section name shows the salt type (I, II, III, or IV) and, for the Volterra Basin samples only, the sample depth within the Saline di Volterra Formation (cf. Fig. 2).

Preliminary observations on the thin sections were performed using a plane polarized transmitted light microscope (Nikon optiphot-pol) equipped with a digital camera. Then the sections were scanned using an Epson V750 Pro unit equipped with a transparency adapter. The scanning resolution (interpolated) was 1800 dpi yielding images of around 3500 by 2000 pixels. Each image was enhanced with a photo-editing software in order to increase the contrast and highlight the grain boundaries.

Afterwards, each scanned image was imported and mapped in the Imagej software (http://rsbweb.nih.gov/ij/index.html). For each section, crystals were manually outlined to produce binary images. This operation was repeated for primary crystals remnants and clay inclusions, when present. Binary images were then analyzed with the Imagej software, and several numerical parameters for each particle were extracted, including the crystal area, orientation, and size (expressed as major and minor axes of the ellipse of equivalent area; e.g., Higgins, 2006). Particle elongation is quantified as the roundness parameter (minor axis/major axis), which corresponds to the inverse of aspect ratio. Decreasing roundness from unity (equant shape) indicates increasing particle elongation. To express the distribution of particle shapes in each sample, we used the average roundness value and a parameter (shape uniformity, χ), representing the deviation from this value. Shape uniformity χ is calculated as the coefficient of determination (R²) of the linear regression of major vs. minor axes data. High values of χ (up to χ = 1) indicate a uniform distribution of the crystal shapes. Three-dimensional crystal habits were then obtained by applying a 2D–3D stereological conversion using the CSDslice software implemented by Morgan and Jerram (2006). This software compares the distribution of 2D size measurements to a database of shape curves, determining through regression calculation a best-fit 3D crystal habit in terms of short (S), intermediate (I), and long (L) axis.

As a final step, all thin sections plus six additional sections (see Table S1 in the Supplementary Material) were etched under running water for few seconds and prepared for further observations. Further analyses were then performed using a plane polarized reflected light microscope (Zeiss Stemi 2000C) equipped with a Nikon Coolpix digital camera. Etching process (Urai et al., 1987) is important as it allows to see subgrains, visible in reflected light only. The subgrain size analysis, through reflected light microscope observation and Imagej software, was carried out in order to obtain differential stress values.

4.2. Laboratory ultrasonic waves velocity measurements

Nine samples were cut in the shape of cubes (C1–C9) of 5 cm length from the collected S1113 cores (Saline di Volterra Formation). Five samples are representative of the type I salt (C3–C7), and they differ by the presence of horizontal or inclined thin clay layers; three are from the type II salt (C2, C8, and C9), whereas one sample is from the type III salt (C1) (cf. Fig. 2). Facies IV samples were not prepared because the amount of type IV salt was insufficient for ultrasonic waves
measurements. P- and S-waves velocity measurements were carried out along each cubic axis \((X, Y, Z)\) at the HP-HT Laboratory of Istituto Nazionale di Geofisica e Vulcanologia, Rome. A high-voltage (900 V) pulse generator was used to generate elastic waves while P and S piezoelectric transducers with resonance frequency at 1 MHz were used in order to keep a correct ratio between wavelength and sample size. From the velocity measurements, the dynamic Young modulus \((E_d)\) and the Poisson ratio \((\nu)\) were calculated (Ciccotti et al., 2004; Vajdova, 1999; and references therein).

### 4.3. Uniaxial compressive experiments

Uniaxial experiments were carried out at both ambient temperature and 200 °C on prismatic samples cut from the cubes used for the ultrasonic waves velocity measurements.

Uniaxial runs at constant strain rate of \(5.5 \times 10^{-5} \text{s}^{-1}\), \(T = 200{\text{C}}\) and atmospheric pressure were carried out on five samples measuring \(5 \times 3 \times 3 \text{ cm}^3\): C3 and C7 (type I), C2 (type II), and C1 (type III) (cf. Fig. 2). As for ultrasonic waves velocity measurements, deformation experiments were only carried out on type I, II, and III salts and not on samples from type IV. Tests were carried out at the HP-HT Laboratory at Istituto Nazionale di Geofisica e Vulcanologia, Rome, using a servo-controlled uniaxial testing machine which allows to test the sample under a variety of loading conditions up to 250 kN. The hydraulic system consists of a cylinder with a servo valve control and a free-standing pump/reservoir. The system is completed by a linear variable displacement transducer (LVDT) extensometer, developed to measure axial deformations with a resolution of \(\pm 0.1 \mu\text{m}\), during the loading.

Stress/strain data were registered during the deformation process until failure occurrence. From these data, static Young modulus \((E_s)\), deformations with a resolution of \(\pm 1 \mu\text{m}\), sonic waves velocity measurements.

### 5. Results

#### 5.1. Textural analysis

##### 5.1.1. Qualitative observations

Preliminary reflected and transmitted light microscope observations of the thin sections have been performed in order to recognize the main features of the facies end members.

Type I samples, V161-I-v, V161-I-h, and Ra1-I, are characterized by a great abundance of primary crystals, showing as a result dark gray colors in transmitted light and white/light gray colors in reflected light. The crystals vary in sizes from less than 1 mm to around 1 cm in length and have typical primary salt shapes like chevrons and hoppers (Lowenstein and Hardie, 1985; Speranza et al., 2013) with easily recognizable growth bands (b and d in Fig. 3; d-7 in Fig. 4). A sub-vertical preferred orientation of the chevron crystals axes is visible. The majority of the primary crystals are “remnants,” being more or less eroded and worn. The erosion of a primary salt crystal leads to the formation of a secondary “clear” salt that appears white in transmitted light and dark gray in reflected light. Indeed, type I salt crystals are almost always constituted by a “core” of primary salt surrounded by a rim of secondary salt (g in Fig. 3). However, primary remnants can be found not only in the core but also at the edge of a given crystal (b and d in Fig. 3) or in a off-center position. Areal ratio between primary and secondary salt within a single crystal is variable and sometimes more than one remnant can be found inside the same crystal. Two kinds of transitions between primary and secondary salt could be identified within a given crystal: the first transition sharp and clear (a-1, b-3, and b-4 in Fig. 3; b - 1 in Fig. 4) and the second transition more gradual (a-2 and g in Fig. 3) and the second transition more gradual (a-2 and g in Fig. 4) and the second transition more gradual (a-2 and g in Fig. 4). Both transitions can be found on the same primary crystal remnant. Type I crystals are not elongated and do not have a clear iso-orientation, and pure secondary salt crystals are very rare. In some cases (d and h in Fig. 3), crystals shape, dimension, and orientation are closely related to those of the contained primary crystal remnant. However, in other crystals (c and e in Fig. 3), such a relationship is

### Table 1

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<tr>
<th>Slice</th>
<th>Facies</th>
<th>Site</th>
<th>Number of grains</th>
<th>Average crystal area (mm²)</th>
<th>Orientation (°)</th>
<th>Average roundness</th>
<th>Shape uniformity (γ)</th>
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<td>Volterra</td>
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<td>1.968</td>
<td>92</td>
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#### Slice Facies Site
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- V130-II II Volterra
- V126-II II Volterra
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</table>
absent. Crystal boundaries appear as thin dark lines (transmitted light, Fig. 3) or light lines (reflected light, Fig. 4), with the thickness depending on the angle between crystal boundary and thin section plane, and appear always decorated by a complex network of secondary fluid inclusions (d-6 in Fig. 4). Healed fractures containing arrays of secondary fluid inclusions have also been observed.

Type III and IV salt samples (V141-III, Zi1-III, V158-IV, Pe3-IV) contain almost only secondary salt crystals with no or very few primary salt remnants. Moreover, in the type III salt, crystals show a very clear and pronounced elongation and the grain size is noticeably smaller compared to the other types of crystals (c in Fig. 3). Type II salt may show the characteristics observed in both type I and III–IV with an intermediate crystal elongation and primary crystal content. If larger clay inclusions are present (V132-II and V130-II), salt crystals in their proximity appear highly elongated and deformed. In a few cases, longer primary crystal remnants appear fractured (f in Fig. 2), without noticeable displacement at crystal boundaries.

Thin sections etched with water revealed the presence in all samples of subgrains (f and g in Fig. 4). It should be noted that subgrain-rich crystals are more abundant in type II, III, and IV salt compared to type I. Subgrain size appears larger in types I and IV compared to type II and III salts.

Solution–precipitation features and fluid-enhanced grain nucleation and grain boundary migration evidences have been observed (Fig. 3 and 4), particularly in type II, III, and IV salt samples. These samples show higher amounts of subgrain- and fluid inclusions poor crystals, features that can be interpreted as due to new, strain-free, recrystallized grains (Carter and Hansen, 1983; Schléder and Urai, 2005; Senseny et al., 1992) (b in Fig. 3; a, f, and g in Fig. 4).

5.1.2. Quantitative determinations

Results of the image analysis are reported in Table 1. In particular, the number of analyzed particles, average particles area, orientation of the major axis (0° means vertical), particles average roundness (minor/major axis), and shape uniformity χ, 3D particles shape and the estimated relative amounts of different mineral components (Salt%, Primary Salt%, Clay%), are reported.

The number of analyzed particles varies between 169 (Ra1-I) and 761 (V141-III) with an average area ranging between 0.5 (V141-III) and 4.9 mm² (V158-IV). Major axis angle averages approximately 90° (horizontal, V161-I-v, Ra1-I) with a maximum inclination of 35–40° (V141-III, Pe3-IV); average roundness is comprised between 0.27 (Zi1-III, very elongated crystals) and 0.63 (Ra1-I, much more rounded) and the shape uniformity χ results higher (around 0.8–0.9, indicating lower data dispersion) for V161-I-h, V161-I-v, and Ra1-I sections and lower (around 0.5–0.65) for Zi1-III and V141-III sections. Crystal habits vary between 1:1, 25:1, 7 (Ra1-I, almost cubic crystals) and 1:2, 9:7 (Zi1-III, elongated prismatic crystals). Finally, the estimation of the components distribution indicates that type I salt contains about 20% of primary crystals while this percentage decreases to ca. 14–7% for the type II, reaching almost 0% in the type III and IV salt. Also the clay component is variable, reaching 17% in the type II salt (V132-II sample).

From the 3D scatter plot (Fig. 5) relating average crystal area, average crystal roundness, and shape uniformity χ, it can be observed...
Fig. 4. a) Suggested solution–precipitation-related salt crystals. Two primary crystal remnants (dark gray areas) were pushed together (red lines – white arrows) while newly formed clear salt is deposited and crystals grow in a direction more or less perpendicular to the compression axis. b) Detail from a), showing the effect of a suggested solution–precipitation mechanism in dissolving (area within dashed line) progressively a primary salt remnant. 1: sharp transition between primary and secondary salt; 2: crystal boundary; 3: primary crystal remnant; 4: isolated fluid inclusions, perhaps left after the primary salt remnant erosion. c) Crystal boundaries (5) appear bright in reflected light due to the presence of secondary fluid inclusions. d) Detail from c) showing the complex network (6) of secondary fluid inclusions almost always present at crystal boundaries, suggesting the effectiveness of fluid migration and entrapment in salt. 7: primary salt remnant (within the dashed line) made up of countless primary fluid inclusions organized in growth bands. e) Fluid-assisted grain boundary migration evidence (within the red frame). f) Detail from e) showing a crystal boundary (9) migrated toward the upper-left corner from a former position outlined by a straight secondary fluid inclusions array (yellow dashed line). The migrating crystal boundary has worn out part of a large, subgrain and primary fluid inclusions-rich (8) crystal, leaving backward new, subgrain and fluid inclusions poor salt. Notice that the core of the crystal (within white dashed line) is still primary fluid inclusions rich. g) In more deformed salt, together with subgrain-rich crystals (12), strain-free, subgrain poor crystals (13) become more abundant. a), b), and g): V12G-II section. c), d), e), and f): Ra1-I section. b) to g) are reflected light photomicrographs.
that these three parameters are directly proportional. Moreover, it can be seen that type I samples (V161-I-v, V161-I-h, and Ra1-I) and the V158-IV (type IV) sample show higher roundness and average crystal area, as well as a lower shape data dispersion (higher $\chi$). In type II (V130-II, V126-II, and V132-II) and III (V141-III, Zi1-III) samples, a decrease in average crystal area coupled with a lower roundness (i.e. higher crystal elongation) and higher shape variability (lower $\chi$) occurs. In this case, the Pe3-IV (type IV) sample fall close to type II samples, showing rather different characteristics with respect to the V158-IV section.

Rose diagrams for the crystal orientation (Fig. 5) show that, regardless of the elongation, crystals’ major axis lies on a horizontal or inclined direction, with the tilt angle mostly comprised in the $0°–30°$ range. Nevertheless, type I samples show a more pronounced tilt angle variability (for instance Ra1-I section) compared to type II and especially III samples (V141-III and Zi1-III), whose crystals show the highest degree of iso-orientation.

Furthermore, selecting only type I samples, rich in primary crystals, and plotting in the same rose diagram, the major axis orientation for both all crystals and only for the primary ones (Fig. 6), a rather good correlation between the two datasets can be observed. Finally, the average crystals area vs primary crystal content plot (Fig. 6) suggests that these two parameters are directly proportional, with the exception of the V158-IV section.

In addition to the textural analyses presented above, we performed subgrain size analysis and differential stress calculation on all sections except for the V161-I-v sections, where subgrains are nearly absent. It is known (Carter et al., 1993; Schléder and Urai, 2005; Senseny et al., 1992) that the steady-state subgrain size is inversely proportional to...
the stress difference experienced by the crystals following the relation:

\[ D = k \sigma^{-m} \]

where \( D \) is the average subgrain size (μm), \( \sigma \) is the differential stress (MPa), while \( k \) and \( m \) are material constants. We used this relation to calculate the stress difference for our samples, setting \( k = 214 \) and \( m = 1.15 \), as best suggested values in \((\text{Carter et al., 1993})\). Thus, the relation (1) becomes

\[ \sigma = 105 D^{-0.87} \]

The average subgrain diameter (D) varies between 411 μm (V158-IV-h) and 97 μm (Ra2-IV), so the calculated (Eq. (2)) differential stress values resulted between 0.57 MPa (V158-IV-h) and 1.99 MPa (Ra2-IV) (Table 2).

In Fig. 7, differential stress data as a function of sample type are plotted. Lower stress values, ranging between 0.57 Mpa (V158-IV-h) and 0.88 MPa (V126-II) are associated with all type I samples, three type IV samples and two type II samples. Higher stress values, ranging between 1.16 MPa (Zi-Ill and Pe3-IV) and 1.43 MPa (V132-II), are associated all type III samples, the remaining type II samples, and the Pe3-IV sample (type IV). The Ra2-IV sample falls at the highest stress value of 1.99 MPa. A clear separation between the “lower stress” (V158-IV-h, V158-IV, V122-IV) and “higher stress” (Pe3-IV, Ra2-IV) type IV samples (named IV-L and IV-H, respectively) can be observed, so we decided to divide the type IV facies in these two groups.

By considering the average stress values for each facies group and relative standard deviation (Fig. 7, black line), a progressive stress difference increase from type IV-L to type I and II toward type III and finally type IV-H samples can be observed. Furthermore, type II and IV-H samples show higher stress variability compared to type IV-L, III, and I, the last displaying the lowest standard deviation.

### 5.2. Laboratory ultrasonic waves velocity measurements

Table 3 shows the results of laboratory ultrasonic waves velocity measurements on the different salt types. \( V_p \) (P-waves velocity, average from X-Y-Z axes), \( V_s \) (S-waves velocity, average as for \( V_p \)), and the standard deviation (relative to the three axes values) for these two parameters are reported, along with calculated dynamic Young modulus \((E_d)\) and the Poisson’s ratio \((\nu)\) \((\text{Ciccotti et al., 2004})\).

\( V_p \) and \( V_s \) values for X, Y, and Z axes of each sample are shown in Fig. 8. It can be observed that \( V_p \) and \( V_s \) values for X and Y axes are very similar and homogeneous in all the investigated salt facies (especially for P-waves), whereas \( V_p \) and \( V_s \) values for Z axis are more variable \((\nu \text{ show higher standard deviation})\) and are generally lower than the \( V_p \) and \( V_s \) values for X and Y axes, both on average and within the same sample. Average \( V_p \) values vary between 4196 m/s (C7, type I) and 4521 m/s (C2, type II), with a systematic lower value of about 5% for type I. Average \( V_s \) values are comprised between 2425 m/s (C4, type I) and 3072 m/s (C2, type II) with a lower value of about 9% for type I. On average, the standard deviation for \( V_p \) is about two times higher for type I samples (C5 through C7) than for type II and III samples (C1–C2 and C8–C9). The standard deviation for \( V_s \) shows similar trends and it is approximately three times higher for type I samples compared to values for the other samples types.

In terms of dynamic Young moduli, values mirror, as expected, the elastic waves velocity (Table 3) behavior, with values higher of about 10% for type II and III samples with respect to type I samples.

### 5.3. Uniaxial compressive experiments

Table 4 shows the results of uniaxial compressive experiments. At room temperature, peak stress values range from 28.3 MPa (C3, type I) and 31.7 MPa (C1b, type III). Static Young modulus \((E_s)\) varies between 2.8 GPa (C1b, type III) and 10.2 GPa (C3, type I) (average value 5.3 GPa). Strain at peak spans between 2.6% (C3, type I) and 10% for type II and III samples with respect to type I samples.

![Fig. 7. Calculated differential stress from subgrain size plot (colored dots) and average values for each facies group along with relative standard deviation (line and bars). Values are grouped according to the salt facies. Further explanations in text.](image-url)
reached, suggesting a transition from brittle to plastic deformation. \( E_s \) is comprised between 0.35 GPa (C8, type II) and 0.48 GPa (C6, type III). \( E_s \) values at 200 °C are approximately 1/10 than the values at 20 °C. We calculated that \( E_s \) decreases of about 0.015–0.035 GPa/K, a decreasing rate in agreement with that reported in Senseny et al. [1992]. Moreover, at 200 °C, elastic limit approximate values are lower than at 20 °C, varying from 6 Mpa (C8, type II, and C1, type III samples) and 9 Mpa (C6, type III).

Fig. 9 shows deformation curves (Fig. 9a) and related peak stress and static Young modulus values (Fig. 9b) from compressive runs at the temperature of 20 °C. Deformation curves show the prevalence of plastic behavior with deformation typical of the ductile regime with different rates of strain softening once reached the peak stress. In particular, samples C3 and C7 (type I) begin to deform gradually at lower strain rates in agreement with that reported in Senseny et al. [1992]. Moreover, at 200 °C, elastic limit approximate values are lower than at 20 °C, varying from 6 Mpa (C8, type II, and C1, type III samples) and 9 Mpa (C6, type III).

Stress/strain curves from compression runs at 200 °C (Fig. 10) show again a plastic deformation behavior, but this time, a strain hardening behavior is observed. An initial pseudo-elastic deformation, up to about 3–4% axial strain and values of up to 6–8 MPa, is followed by a long strain hardening phase with the whole strain mirrored from a stress increase of only about 6 MPa typical of a ductile deformation. The samples show similar behavior, with higher stress values during deformation pertaining to type I sample (C6) and slightly lower values related to type III (C1) and type II (C8) samples, respectively.

### 6. Discussion

#### 6.1. Microstructural facies definition and characterization

Description, origins, evolution, and relationships between primary and secondary salt features have been extensively investigated in the past (Lowenstein and Hardie, 1985, and references therein; Schlèder and Urai, 2005; Schlèder and Urai, 2007). Primary salt crystals (chevron, cubes, hoppers) with primary fluid inclusions-rich growth bands can be partially eroded even in a syndepositional stage (Lowenstein and Hardie, 1985) by undersaturated brine arrival in a shallow perennial or ephemeral lake setting. Results of such a process are primary crystals sharp truncations and deposition of clear, void-filling, halite cement, around partially eroded crystals. These processes can operate repeatedly also during the early diagenetic stage (Lowenstein and Hardie, 1985). Moreover void-rich, porous salt can also be subjected to deformation/recrystallization processes, with progressive vanishing of primary crystal remnants, formation of new, clear halite crystals, deformation of crystal shape, fluid migration (Peach and Spiers, 1996; Pennock et al., 2006; Ter Heege et al., 2005a, b; Urai and Spiers, 2007). In such cases, deformation and accommodation-related processes like pressure solution, dislocation creep, fluid-enhanced grain boundary migration, microcracking, and so on, are active depending on the surrounding P-T and stress conditions (Chester, 1988) and finally tend to produce a more or less deformed, void lacking, totally recrystallized salt (Schlèder and Urai, 2005). Depending on the extent of the processes that operate on “pristine” salt after deposition, actual rocksalt can appear in various conditions, from a near-pristine to a completely

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**Table 4**

Uniaxial compressive tests data summary for both ambient temperature (above) and 200 °C temperature (below) experiments.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Facies</th>
<th>Temp. (°C)</th>
<th>Strain rate (s⁻¹)</th>
<th>Peak stress (MPa)</th>
<th>Static E (GPa)</th>
<th>Strain at peak (%)</th>
<th>Elastic limit (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C3</td>
<td>I</td>
<td>20</td>
<td>5.5×10⁻⁵</td>
<td>28.3</td>
<td>10.2</td>
<td>2.6</td>
<td>6.5</td>
</tr>
<tr>
<td>C7</td>
<td>I</td>
<td>20</td>
<td>5.5×10⁻⁵</td>
<td>30.9</td>
<td>4.1</td>
<td>2.8</td>
<td>10</td>
</tr>
<tr>
<td>C2b</td>
<td>II</td>
<td>20</td>
<td>5.5×10⁻⁵</td>
<td>30.5</td>
<td>6.5</td>
<td>3.6</td>
<td>11.5</td>
</tr>
<tr>
<td>C1</td>
<td>III</td>
<td>20</td>
<td>5.5×10⁻⁵</td>
<td>30.8</td>
<td>3.1</td>
<td>3.7</td>
<td>13</td>
</tr>
<tr>
<td>C1b</td>
<td>III</td>
<td>20</td>
<td>5.5×10⁻⁵</td>
<td>31.7</td>
<td>2.8</td>
<td>3.9</td>
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<tr>
<td>Average</td>
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<td></td>
<td></td>
<td>30.4</td>
<td>5.3</td>
<td>3.3</td>
<td>10.8</td>
</tr>
<tr>
<td>C6</td>
<td>I</td>
<td>200</td>
<td>1×10⁻⁵</td>
<td>–</td>
<td>0.48</td>
<td>–</td>
<td>9</td>
</tr>
<tr>
<td>C8</td>
<td>II</td>
<td>200</td>
<td>1×10⁻⁵</td>
<td>–</td>
<td>0.35</td>
<td>–</td>
<td>6</td>
</tr>
<tr>
<td>C1</td>
<td>III</td>
<td>200</td>
<td>1×10⁻⁵</td>
<td>–</td>
<td>0.36</td>
<td>–</td>
<td>6</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td>0.4</td>
<td>–</td>
<td>7</td>
<td></td>
</tr>
</tbody>
</table>

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deformed/recrystallized state. Moreover, considering impurities, as clay, and other deposition- or deformation-related structures that can be found along with and within salt, it is clear that the number of possible rocksalt facies is very high.

Along the Saline di Volterra Formation, both macro- and microstructural study led to the observation, definition, and characterization of four salt facies, which can be considered a good estimate of typical end members that can be found within a salt deposit.

The 43 m thick salt sequence starts at the bottom (Fig. 2) with the type I salt, rich in primary crystal remnants that have suffered a variable degree of erosion and dissolution and are in contact with clear salt that could have been generated both in a syndepositional–diagenetical or early deformation stage. Crystals made up only of secondary salt are nearly absent and just few contain subgrains. Thus, on the whole, the deformation degree is low, as shown also by the higher average crystal roundness, with high shape uniformity $\chi$, poor crystals iso-orientation and cubic-like 3D crystals habits (Table 1). Calculated differential stress values are the lowest in our dataset (Fig. 7). We believe that the type I salt characteristics are typical of a relatively “immature,” with respect to the degree of deformation, rocksalt. Among the samples collected from the other sites for comparison purposes, Ra1-I sample from the Racalmuto mine and Vi1-I sample from Crotone Basin showed similar values of the facies-related characteristics, fitting well into the type I salt.

As previously said, two kinds of primary crystal boundaries were observed, the first sharp, probably related to dissolution in a syndepositional stage (Schléder and Urai, 2005), and the second gradual (a–2 and g in Fig. 3) (cf. also b in Fig. 4). We suggest that the gradual blending of a primary salt remnant in secondary clear salt could be explained by solution–precipitation creep and fluid-assisted grain boundary migration mechanisms. In this case, fluid transfer (Pennock et al., 2006; Ter Heege et al., 2005a, b; Urai et al., 1987) would gradually remove primary fluid inclusions from primary salt and move the fluids toward grain boundaries and healed fractures, leaving backward newly formed, fluid inclusions poor, clear salt (a–b and e–f in Fig. 4). Type I salt, despite the deformation degree “immaturity” and the pristine-salt-like appearance, shows some evidences of climb-controlled deformation and fluid-assisted grain boundary migration, suggesting that these deformation mechanisms can be active in rocksalt even at low stress and deformation degree. The fluids abundance and the effectiveness of fluids migration (c–d in Fig. 3), that may ease the
deformation mechanisms activity, are confirmed by the widespread presence of secondary fluid inclusion networks at grain boundaries and in healed fractures.

Type II salt, occupying the upper part of the Volterra’s salt sequence, is characterized by a greater deformation and recrystallization degree, as highlighted by the higher crystal elongation (lower average roundness), lower shape uniformity \( \chi \), a clearer crystal iso-orientation, a much lower abundance in primary crystal remnants, and a lower average crystal area (Table 1). Much more crystals are subgrain-rich, and there are several strain-free, fluid inclusion-poor crystals that could have been possibly formed in response to recovery processes (grain nucleation and grain boundary migration) (g in Fig. 4). Also differential stress values are higher, with average value of 1.14 MPa (Fig. 7). We suggest that in this case, deformation mechanisms as solution–precipitation creep, climb creep, and fluid-assisted grain boundary migration were diffusely active in response to the higher stress suffered by type II salt (Senseny et al., 1992; Ter Heege et al., 2005a, b, and references therein), that we believe typical of an “intermediate maturity” rocksalt.

The type III salt, located in the middle of the Saline di Volterra Formation, shows the lowest average crystals area, higher crystal elongation (lower average roundness) with a clear crystal iso-orientation, higher shape variability (lower \( \chi \)), and almost no primary crystal remnants (Table 1). Subgrain-rich crystals are abundant, as well as recrystallized, subgrain-, and fluid inclusion-poor crystals. Differential stress values are the highest in our dataset (Fig. 7). Therefore, also in this case, solution–precipitation creep, climb creep, and fluid-assisted grain boundary migration seem to have been pervasively active in developing such a “mature,” with respect to deformation degree, rocksalt facies. Among samples selected for comparison, the Z1-I1 sample fits very well in the facies III type.

The type IV salt is quite different from the others, being represented, within the Volterra’s sequence, by a thin layer made up of large (highest average area), uniform crystals (shape uniformity \( \chi \)), slightly deformed (high average roundness) without a clear crystal iso-orientation and with almost no primary salt (Table 1). Indeed, the type IV salt layer, located at 158 m of depth (Fig. 2), lies between two horizons of type I like salt and has been subjected to similar differential stress values (Fig. 7).

Furthermore, both macroscopically and microscopically, type IV salt layer appears rather similar to the fracture-filling rocksalt facies sampled within clay blocks from the top of the Saline di Volterra Formation (122 m, cf. Fig. 2 and Figure S1 in the supplementary material). Thus, we suggest that the type IV salt layer was developed during a syndepositional or early diagenetical stage as the filling of a previously developed void by highly saturated brine circulating within the salt deposit. Later, this layer has been deformed to a degree similar to that of the preceding and surrounding type I salt. Among the samples chosen for comparison, the Pe3-IV salt is rather different, showing much lower grain size compared to the V158-IV section, higher differential stress and the presence of many subgrain-free, newly developed crystals. Thus, we suggest that in this case, widespread recovery mechanisms have obliterated the original facies characteristics in response to high stress/deformation degree. So the Pe3-IV sample could be considered as a “very mature” salt type. Higher stress/deformation degree and high “maturity” should be also considered for the Ra2-IV section, whose subgrain size resulted in the highest differential stress among our samples (Fig. 7). Thus, as previously illustrated, we decided to classify type IV salt into a lower and a higher deformation degree end (IV-L and IV-H, respectively), assigning the Volterra samples (V158-IV, V158-IV-h, V122-IV) to type IV-L and Petralia (Pe3-IV) and Racalmuto (Ra2-IV) samples to type IV-H.

6.2. Salt facies evolution and its effect on rocksalt rheological parameters

It is important to stress that the actual salt facies belonging to the Saline di Volterra Formation originated from a unique “pristine” salt, deposited in a shallow water basin (Testa and Lugli, 2000) as an aggregate of primary crystals as described in (Lowenstein and Hardie, 1985), alternating with moderately thick clay layers. The facies illustrated above are the result of subsequent dissolution/deposition and deformation/recrystallization processes operating during the depositional, diagenetic, and deformation stages. The investigation of the Volterra salt facies has allowed us to follow the evolutionary path from the original salt through the type I, II, and III facies, the last one representing the most “mature” end, with respect to deformation degree. Within this framework, the Volterra IV-L type salt, as previously mentioned, represents a peculiar case, while the type IV-H salt (missing in Volterra) can be considered as the final stage of a deformation-related history, with a complete salt facies recrystallization and obliteration of original facies characteristics.

From the salt facies characteristics plot of Fig. 5, we suggests that, starting from the type I/IV-L through the type II/III toward the type IV-H, with increasing deformation degree the average crystal area, average roundness and shape uniformity \( \chi \) decrease, as also suggested by 3D crystal habits. Moreover, deformation tends to establish a higher crystal iso-orientation, as can be observed in samples of the type II, III, and IV-H salt (Fig. 5).

We also suggest that when primary crystal remnants are still abundant (type I), like in “immature” salt, crystal textures are partially controlled by primary crystals characteristics. Particularly (cf. d–h in Fig. 3), crystal orientation seems to be related to primary crystal remnants orientation (rose diagrams in Fig. 6), mainly of chevron remnants, and crystal size is always at least slightly larger than primary remnants size. Later, with increasing clear salt formation in response to deformation and progressive erosion of primary remnants (type II, III, IV-H) (cf. Fig. 6), crystal textures become controlled mainly by the deformation mechanisms discussed above.

Differential stress values calculated from subgrain size measurements resulted lower for facies I and IV-L and progressively higher for facies II, III, and IV-H, confirming that the deformation history played a major role in the salt facies textural evolution.

Ultrasonic waves velocity measurements and uniaxial compressive tests allowed us to study how rocksalt petrophysical parameters vary with facies textural evolution.

Type I salt is characterized, on average, by lower seismic waves velocities (Fig. 8) and, at ambient temperature, by lower dynamic Young modulus, strain at peak, and slightly lower elastic limit, while peak stress is like that of type II and III (Fig. 9 and Table 3). However, at higher temperature (200 °C), type I salt shows higher stress values during deformation as well as higher static Young modulus and slightly higher elastic limit (Fig. 10 and Table 4). We suggest that this behavior may be related to the effect exerted on the bulk properties of type I salt by primary crystals, greatly abundant in these samples. Their size, shape, and orientation, as previously shown, affect rocksalt texture evolution during the beginning of the deformation history. The higher fluids abundance, mainly related to primary fluid inclusions with cubic shape dispersed in the crystal lattice, could be invoked to explain the observed higher attenuation of seismic waves velocity and related dynamic Young modulus. Also, lower strain at peak and elastic limit values at ambient temperature, indicating a higher “softness” of type I salt, may be due to the higher fluids content of primary crystals. At high temperature, the type I salt seems slightly “harder” than type II and III, and we suggest that this could be related to the different distribution of fluid inclusions in primary and secondary crystals. Secondary salt (the main component of type II/III salt), containing mobile secondary fluid inclusions concentrated at grain boundaries or in healed fractures, can become, with increasing temperature, slightly more “soft” and prone to deformation compared to primary salt, whose cubic primary fluid inclusions, less mobile and randomly dispersed, give some higher strength to primary crystals under tested conditions (Fig. 11). In addition, at higher temperature, all experiments showed strain hardening (Fig. 10). This behavior has not been
observed by Ter Heege et al. (2005a,b) on wet polycrystalline halite samples deformed under confined conditions and similar temperatures. We believe that under our experimental conditions, samples lost their natural content of fluids and dried out, thus enhancing the strain hardening observed and preventing weakening mechanisms controlling the steady-state flow observed in Ter Heege et al. (2005a,b).

Inclined thin clay layers, particularly evident in type I salt samples (e.g. C3 sample) used for seismic waves velocity measurements and uniaxial compression at ambient temperature (cf. Fig. 8), may have enhanced Vp velocities attenuation along Z axis (Fig. 8), as well as they have behaved as preferred slip planes during deformation, contributing to the observed behavior.

With increasing salt “maturity” (type II and III), the seismic waves velocity and the dynamic Young modulus increase (Fig. 8 and Table 3), with slightly higher values for facies II compared to facies III. Peak stress is alike type I salt, whereas strain at peak and elastic limit at ambient temperature are higher compared to type I salt (Fig. 9 and Table 2). At $T = 200 \, ^\circ C$, instead, static Young modulus and elastic limit are lower than type I salt (Fig. 10 and Table 4), as well as stress values during deformation, with C8 sample (type II) showing the lowest values. As previously discussed for type I salt, we suggest that in this case, the observed behavior may be due to the characteristics of the secondary salt crystals, that are the main component of type II/III salt. Less seismic waves velocity attenuation and a “harder” behavior at ambient temperature may be explained with a lower fluid abundance mainly due to the absence of

**Fig. 11.** Macroscopic pre- and post-run appearance of type I salt sample (C6) uniaxially compressed at 200 °C to a final axial strain of 20%. In the post-run picture, primary crystal remnants (P) seem to be less deformed than secondary ones (S), suggesting that secondary salt is softer and more prone to deformation at tested conditions. Schematic block diagrams showing the appearance of cubic primary fluid inclusions (PI) within primary crystals and secondary fluid inclusions networks and arrays, fluid- or gas-filled (SI or Sg), found in healed fractures (HL) or at crystal boundaries, are also provided.
primary fluid inclusions. Grain size, crystal roundness, and iso-orientation may also affect the type II/III salt response to deformation. The smaller crystals characteristic of the more “mature” rock salt are likely to be stronger during uniaxial deformation at ambient temperature. At higher temperature, on the contrary, an opposite behavior is observed. Although, as previously mentioned, all samples show a strain hardening behavior, type II/III salt, rich in secondary salt crystals and seem to become softer and more easily deformed compared to type I crystals (Fig. 10). In this case, mobile secondary fluid inclusions network at grain boundaries may enhance deformation mechanisms as solution–precipitation creep and fluid-assisted grain boundary migration.

We have observed that, on the whole, primary and secondary salts show rather complex deformation behavior. Thus, a detailed analysis of primary vs secondary crystals rheology is desirable in order to get a better understanding of the overall rock salt rheology.

Fig. 12 shows combinations of the various parameters obtained from both thin sections analyses and petrophysical experiments at ambient temperature, useful to link salt facies characteristics evolution with rock salt behavior. Notice that the whole parameters dataset is available only for three or four samples, representative of the type I, II, and III salt.

We found that both Vp and E are inversely proportional to average roundness (Fig. 12, a) with a clear separation between the type I and II–III salt. Thus, higher crystal elongation brings about higher seismic waves velocity attenuation. Es, instead, is directly proportional to the average crystal area, primary crystals abundance, and shape uniformity $\chi$.

![Fig. 12](image-url)
(Fig. 12b, c). Also, $E_v$ decreases as the angle of major axis increases, that is, $E_v$ is higher when average major axis orientation is more or less perpendicular with respect to the compression direction (type I sample), while decreases progressively as the angle between major axis and compression direction decreases (type II–III samples) (Fig. 12, d, e). Furthermore, both elastic limit and strain at peak (Fig. 12, d, e) appear to be inversely proportional to the average crystal area and the primary crystal abundance. So, textural characteristics and fluid inclusions type and abundance seem to influence rocksalt petrophysical parameters in a systematic way.

We are aware that care must be taken in reading our data because of the limited sample dataset. Considering this, we believe that the correlations envisaged are sufficiently constrained for a general interpretation. Of course, further studies are desirable in order to strengthen our observation and interpretation of the data.

Finally, in Fig. 13, we summarize our results about salt facies textural characteristics and related petrophysical parameters evolution in response to deformation degree. As it can be observed in the squared cartoons, an “original” salt, made up of fluid inclusions-rich primary crystals, is subjected to an initial dissolution/redeposition stage followed by an early deformation/recrystallization stage, acquiring the characteristics of the type I salt. Then, through an evolutionary path in response to increasing stress/deformation, type I may turn first into type II and later type III, with a progressive disappearing of primary crystal remnants, increasing crystal elongation, and iso-orientation and decreasing in crystal size. Later, as the final stage, very high deformation implying widespread recovery processes, new grain nucleation, and grain boundary migration may lead to more uniform and less elongated crystals with no primary salt remnants, as showed by the Petralia-like type IV-H salt.

At any stage of the evolutionary path, in response to dissolution/redeposition processes exerted by undersaturated brines, a salt facies can acquire characteristics similar to the Volterra-type IV-L salt, with rather equigranular, large, and undeformed crystals and no primary salt remnants (cf. with the first square that show how should appear an “original,” just deposited, salt). Colored dots refer to the colors and roman numbers assigned to each facies. Black arrows indicate observed facies transitions (Volterra Basin) while gray arrows indicate supposed/possible transitions. Framed text refers to some facies characteristics and petrophysical parameters (from experiments at ambient temperature) and how they change from a facies to another; + (plus) sign means an increase of a given parameter with respect to the preceding facies while (minus) sign means a decrease in the same way. Reported parameters are: Rec (recrystallization), Def (deformation degree), Round (average roundness), SHU (shape uniformity), Pry (primary remnants abundance), $V_p$ ($P$-waves velocity), $E_d$ (dynamic Young Modulus), $E_l$ (elastic limit), $S_p$ (strain at peak), and Dim (average crystals area). Colored arrows on the right show that moving downward in the facies transitions both deformation degree and recrystallization increase while moving upward dissolution/redeposition are active. Notice the difference between the Volterra-like facies IV-L, made up by void-filling salt with a lower “maturity” with respect to deformation degree, and the Petralia-like facies IV-H, that here is the most “evolved” facies, originating from recovery mechanisms in response to very high deformation degree.
remnants within the dissolution area, regardless of the source salt characteristics (Figure S1). Of course, facies IV-L may be subjected to subsequent deformation, acquiring the characteristics of a more “evolved” salt facies.

Along with the schematic facies evolution, facies-related and petrophysical parameters evolution (cf. Fig. 12) are reported in framed text. Type I salt is assumed to be on the less “evolved” extremity of the evolutionary path, so parameters are defined in terms of high/low while for the other salt types, the variation (−, −, or +) for each parameter with respect of the previous facies is provided. So it can be seen that with increasing facies “maturity,” recrystallization degree, P-S wave velocity, Es, elastic limit, and strain at peak increase, while average roundness, shape uniformity χ, primary crystals abundance, Eσ, and average crystal size decrease. This is true until the occurrence of widespread recovery processes with new grain formation (type IV-H) that eventually leads to previous textural characteristics obliteration.

6.3. Salt facies in the Saline di Volterra Formation

In Fig. 14, we show a schematic draw of the type I, II, III, and IV-L salt arrangement within the Saline di Volterra Formation, together with the main textural and petrophysical salt type characteristics. A division into three “deformative units” (A, B, and C) based on the whole parameters dataset is also proposed. It is clear that, omitting the type IV salt due to its peculiarity, the salt type arrangement does not follow a stepwise order from the less deformed type I toward the highly deformed type III. In fact, as it can be seen, the type I salt is at the base, followed by the very thin type IV-L salt layer that, as previously said, shows a similar deformation degree. The rather thin, highly deformed type III salt follows in the middle of the sequence after a clay interval, while the type II, much thicker, takes up all the upper half of the salt formation, that ends with type IV-L salt, filling fractures between clay blocks at the salt sequence top. Suggested deformative units are along this trend. So the “lower deformation” unit (A), comprising the type I and IV-L salt, is followed in turn by the “higher deformation” unit (B), comprising the type III and part of the type II salt (V139-II and V132-II sections). The higher deformation degree is clearly highlighted by the differential stress, rapidly shifting to values as much as two times higher than those of the “lower” unit, as well as by the smaller grain size, lower primary salt content, and a clear crystal iso-orientation. The B unit shows also lower static Young modulus as well as higher strain at peak and elastic limit values compared to the A unit. The following “intermediate deformation” unit (C), comprising the type II V130-II and V126-II samples as well as the type IV-L fracture-filling salt, is characterized by differential stress falling again very rapidly to values as low as half than previous, increasing grain size and primary salt abundance as well as again lower strain at peak and elastic limit values plus higher static Young modulus. However, average roundness, shape uniformity χ, and average crystal orientation remain alike those of the preceding unit. We suggest that this deformation arrangement can be explained considering the Saline di Volterra Formation as divided in three (or more) salt intervals that were moved and deformed as individual bodies. So, the “higher deformation” B unit may correspond to a salt interval characterized by higher mobility, as if it was intruded like a “sill” between the clay intervals of 149–141 m and the salt above 132 m. This hypothesis is supported by evidences of strong plastic deformation of clay blocks and strata present along salt in the 140–132 m interval, with a near-complete destruction of former clay layer continuity, and later transport of inert clay blocks within the moving salt (cf. Fig. 2 and Figure S2 in the Supplementary Material). We suggest that the B unit higher mobility could be explained invoking the high-temperature fluid circulation that affected the Volterra Basin since late Early Pliocene. Extensional fault systems characterizing the basin could have channelled warm fluids around the B unit, triggering its movement (Testa and Lugli, 2000). Within this hypothesis, the “intermediate deformation” C unit could have been
coupled to the underlying unit, maintaining somehow a rather high mobility, as shown again by clay blocks deformation and also by the presence of saturated brine circulation, highlighted by the salt-filled, vertically oriented fractures observable within the clay blocks at the top of the salt sequence. Here, clay fracturing may be induced by the underlying salt movement with intrusion of the saturated brine in the newly formed fractures. The “lower deformation” A unit, below the clay layer at 149–141 m, seems to have had rather less mobility and seems that it has been uncoupled with B and C, although, as showed by the microstructural observations, and it has been subjected to some degree of deformation.

7. Conclusions

Our comprehensive salt facies study including salt cores from the Saline di Volterra Formation (Volterra Basin, northern Italy) and salt samples from Petralia and Racalmuto salt mines (Caltanissetta Basin, Sicily) and Zinga 1–Vitravo 1 salt diapirs (Crotone Basin, southern Italy) allowed to identify, analyze, and test in laboratory four salt facies end members believed to be a good estimate of the wide range or rocksalt facies that can be found in nature. Moreover, we were able to place the facies on an evolutionary path in response to increasing deformation degree.

Initially, we identified four salt facies that have been named as type I, II, III, and IV, analyzing the Saline di Volterra Formation. Facies I shows a great abundance in primary salt remnants and rather rounded crystals so it has been considered as the less deformed and recrystallized end member. Proceeding toward increasing deformation degree, primary salt remnants are gradually dissolved, new, “clear,” secondary salt is formed and a progressive increase in crystals elongation and iso-orientation as well as a crystal size decrease can be observed. So, type II and type III have been identified, presenting increasing deformation degree, with almost no primary salt remnants and smaller, very elongated crystals with a clear iso-orientation in type III. Differential stress calculation from subgrain size is along this trend, with lower values related to the type I and higher values for the type II and III. The main deformation-related mechanisms as observed by microstructural analysis are solution–precipitation creep, climb creep, and fluid-assisted grain boundary migration. The type IV cannot be placed on this evolutionary path and is likely to derive from the type I due to an early dissolution/redeposition process, later acquiring a similar (lower) deformation degree, so it has been named IV-L. Salt samples from Caltanissetta and Crotone Basin fitted well into type I and III, confirming the effectiveness of the proposed classification scheme. Moreover Petralia and Racalmuto samples allowed identifying a high deformation type IV (IV-H), whose crystals are the result of widespread recovery mechanisms.

From ultrasonic waves velocity measurements and uniaxial compressive test on the type I, II, and III salt from Volterra Basin, we obtained petrophysical parameters that we linked to facies characteristics. We found that crystal average roundness, shape uniformity $c$, average crystal size, primary salt abundance, and average crystal orientation may influence the rocksalt petrophysical parameters such as $V_p$ and $V_s$ (P- and S-waves velocity), $E_p$ and $E_q$ (static and dynamic Young modulus), elastic limit and strain at peak in a systematic way. Moreover, the primary salt rich type I salt showed a “softer” behavior at ambient temperature compared to type II/III while resulted slightly “harder” at 200 °C. This could be related to the higher fluids abundance and to the cubic shape of the primary fluid inclusion characteristic of type I salt, lacking in the type II/III salt that host, instead, mobile secondary fluid inclusions at crystal boundaries and in healed fractures.

Finally, the arrangement of type I, II, and III salt within the Saline di Volterra Formation, with the less deformed type I located at the base of the salt series followed by the highly deformed type III in the middle and the type II in the upper part, suggested, together with related textural and petrophysical parameters, a division into three deformative units that behaved differently and independently as a result of the different salt characteristics.

To conclude, we believe that rocksalt type, i.e. textural characteristics, primary vs secondary crystals abundance and fluid inclusions type and abundance, have to be taken into account when assessing any study or application regarding rocksalt.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.tecto.2015.10.018.

References


