Lacunae and Inclusions in the Halite from Valence and Bresse Saliferous Basins

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ABSTRACT

To deepen knowledge of the behavior of rock salt used in storage cavities, investigations were carried out on halite crystals. The study is essentially concerned with discontinuities in the rock, that is, with intra- and intercrystalline lacunae and inclusions, their distribution, size, and contents. Study of the contents includes identification of the constituent mono-, bi- or triphase with chlorides, sulphates, sulfides, clays etc. Next a study was undertaken of samples which were tested under different experimental stresses with respect to the existence of these lacunae and inclusions.

INTRODUCTION

For the last ten years or so Gaz de France has concerned itself with providing natural gas storages in cavities formed by leaching in salt strata. The geological site where the first cavities (Tersanne storage) were set up is the salt-bearing basin of Valence. There two cavities were dug in 1968–1969 and set under gas pressure in 1970. Three further cavities became operational in 1976–1977 and the process of extending such storage facilities will continue over the next few years. The goal is to have 14 cavities by 1984 with a total capacity of about 540 million $m^3(n)$ of natural gas.

For some years Gaz de France has been interested in the salt-bearing basin northeast of Lyons (the Bresse basin). Several cavities are already undergoing leaching there. The first one is due to be set under gas pressure in 1979. The present objective is to create about 30 cavities over the next twenty years.

The Valence salt-bearing basin. The Valence subsidence deep runs north-northeast/south-southwest for about 50 kilometers and is 15 kilometers wide. The salt-bearing strata belong to the Oligocene Epoch (Sannoisian). The top of the salt strata lies 1,400–1,500 meters deep in the area where the cavities are located. Although the substratum has never been reached, the thickness of the lower salt-bearing series can be evaluated at about 500 meters. A thick detrital series with a dominant clayey facies separates the lower salt-bearing series from a second evaporate stratum of lower power in which Gaz de France has elected to locate its cavities.

In the leached zones, three main components are encountered in the salt mass, the halite itself, anhydrite (8 to 10%) and clay (4 to 6%). The sample cores often reveal alternating horizontal beds of insolubles and salt. The halite crystals are usually of medium size, and the presence of inclusions often gives them a grayish tinge.

The Bresse basin. Like the one at Valence, the Bresse salt-bearing basin belongs to the Oligocene Epoch. The numerous wells in this region have revealed a very large evaporatic series, reaching a thickness of 1500 meters in some wells. This series consists of two large salt-bearing masses separated by an intermediate sterile stratum of marl and anhydrite. The cavities now being leached are located in the lower salt-bearing series (the salt roof being located about 1,350 meters down). Gaz de France does not rule out the possibility of subsequently creating cavities in the upper salt-bearing series (where the salt roof is at a depth of between 650 and 700 meters.) The Bresse salt very frequently takes the form of large interlocking translucid crystals separated by clay and anhydrite seams.

Sample measurements and tests. Extensive coring programs have been carried out at the Tersanne and Bresse drilling sites. The core samples have been subjected to various measurements in order to determine the proportion of insolubles, as well as mechanical tests in order to ascertain their elastic properties (Young's modulus, Poisson's coefficient) and plastic properties (cohesion and internal friction angle). These mechanical tests are of paramount importance in determining how the cavities will behave in operation. In addition to these measurements designed to describe the macroscopic behavior of salt, it was felt that it would be useful to take a closer look at the structure of the crystalline aggregates forming the salt-bearing mass and to see whether, on the finer scale of these observations, modifications could not be brought about in the organization of these aggregates when they were subjected to mechanical stresses. All these observations were conducted in the laboratories of the Université de Paris VI and form the subject of this paper.

Description of the Lacunae and Inclusions in the Halite Crystals

The samples investigated are pieces of core obtained from wells made on the Tersanne (Valence basin) and Etrez (Bresse basin) sites. The examinations were carried out at different scales.

1. With a binocular on polycrystalline core pieces (obtained by fragmenting or sawing), on core sections in the centimetric thickness range and small parallelepiped shapes (obtained by sawing), or on small mono- or polycrystalline fragments obtained by breakage or cleavage. Examinations are made either directly across the cleavage surfaces or using Canada balsam covered with a slide in the case of breakage or sawn surfaces.
2. With an optical microscope on small previously-selected fragments (either dry or mounted in Canada balsam), or on sufficiently transparent parallelepiped shapes.
3. With a scanning electron microscope (SEM) on the freshly-cleaved or broken surfaces of small fragments examined beforehand with a binocular or an optical microscope.

INTRACRYSTALLINE LACUNAE

Liquid inclusions. While some are distributed in random fashion through the crystals, most of them form different kinds of groupings, e.g., in milky zones and stripes, inclusions lying in crystallographic planes, or inclusions dotted over surfaces that are more or less warped, of random orientation in relation to the crystallographic directions. Zones entirely devoid of inclusions also exist (on the scale of microscopic observations).

Inclusions of milky zones and stripes. To the naked eye or under the low magnification of the binocular, these zones appear as whitish masses included in the halite. Actually this milky appearance is due to the grouping of liquid inclusions whose cavities are usually cubic in shape. The size of the smallest, and most numerous, of them is in the region of one micron and they are also about one micron apart. All these inclusions occupy cloud-shaped spaces or appear as families of parallel stripes on the faces of the cube (Fig. 1).

These milky zones have been described in connection with experimental crystal growing, where they stem from conditions of crystallogenetic imbalance (oversaturation, presence of foreign elements, etc.) during the crystal's growth (Deicha, 1955; Sella and Deicha, 1962; Rasumny, 1972, 1974, 1976). The disposition of the milky stripes is reminiscent of growth wraiths. These crystallization lacunae and their contents date back to the crystals' growth and testify to at least the ultimate recrystallization of the halite. It has already been stated that the size of the most numerous inclusions is in the region of one micron and that they are spaced from one to a few microns apart. Generally speaking, the inclusions come in sizes of between 1 and 10 to 20 microns, their numbers diminishing with increasing size. A few larger inclusions belong to these zones as well.

It is possible to give estimated orders of magnitude for the total volume occupied by the cavities in a lactaceous part of a crystal. Assume a volume of 1 mm^3 of halite to be occupied by cubic cavities with sides measuring 2 microns, arranged in a simple cubic lattice at a distance of 10 microns from one another. 1 mm^3 of crystal will contain 10^6 cavities with a unit volume of 8 μm^3. The cavities will therefore occupy 8×10^6 μm^3 or 0.008 mm^3 in 1 mm^3 of crystal. In actual fact, the distribution of the cavities in such a zone is

Figure 1. Photomicrograph of a halite fragment obtained by cleavage showing numerous tiny fluid inclusions in cavities of mostly cubic habitus (negative crystals) arranged in bands parallel to the halite crystallographic directions. (Rock salt from Etrez, width of the photograph = 720 μm).

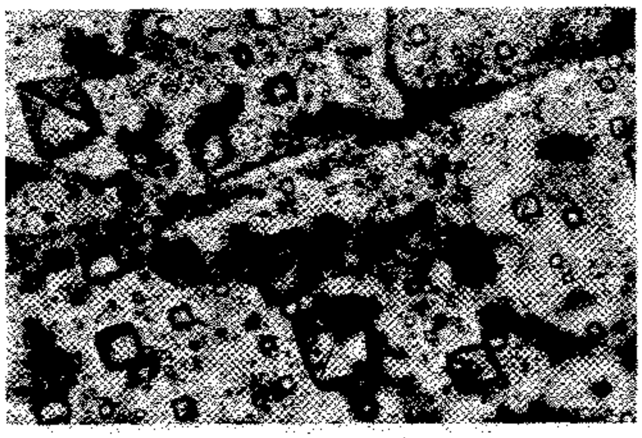


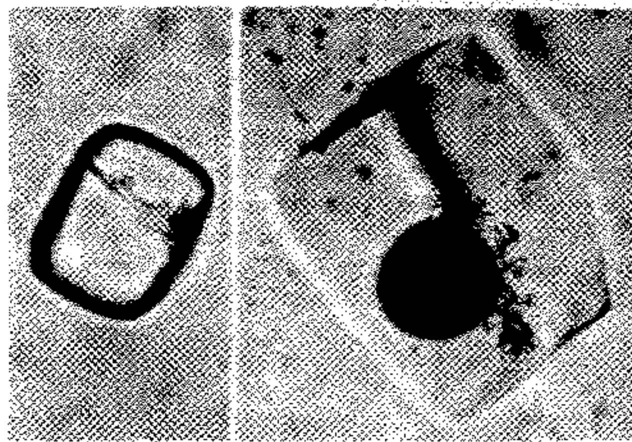
Figure 2. Photomicrograph of a halite fragment obtained by cleavage showing brine-filled cubic negative crystals sometimes containing one or two anhydrite needles. The cavities are more widely spaced and are generally larger than in figure 1. (Rock salt from Etrez, width of the photograph = 430 μm).

not quite so uniform, many of them are smaller and more tightly packed. Others are larger and more widely spaced. Cavities with sides measuring 1 μm, spaced 2 μm apart, would give a volume of 125×10^6 μm^3, or 0.125 mm^3 per mm^3, a cavity with sides measuring 100 μm would occupy 0.001 m^3, and so on.

It is possible to arrive at zones which do not have this whitish appearance, where the cavities are more widely spaced and are uniformly distributed, Fig. 2. The cavities contain either brine alone or brine and one or more solids whose total volume varies with the cavity's volume, from one cavity to another. The included minerals may be partly included in the host mineral and are identical to certain minerals which are completely included in the halite. These facts must lead one to recognize the preexistence of these solids, which have been trapped mechanically by a lacuna during the crystalline growth or which have themselves triggered the formation of a lacuna, as has been demonstrated on various occasions (Deicha, 1976). A crystallization lacuna formed in this way encompasses the foreign element more or less perfectly (Fig. 7); thus one ranges gradually from a solid which appears to be completely included in the host mineral to a solid occupying a small part of the cavity.

The solid phase trapped in the lacunae is in most cases anhydrite (Fig. 8a) (determined optically and confirmed by elementary analyses with a scanning electron microscope). The anhydrite is often present in the form of more or less elongated needles (Figs. 2, 3a) or of feltings of such crystals. Also present are colored minerals whose nature the authors did not determine, and potassium minerals (probably chlorides) (according to the SEM).

Inclusions distributed in the crystallographic planes (Figs. 4 and 5). The inclusions lie in planes parallel to the three faces of the cube or along diagonal planes. The



A B

Figure 3. a) and b)—Photomicrograph of a halite fragment obtained by cleavage showing anhydrite crystals trapped in brine-filled cubic negative crystals, (Rock salt from Tersanne).

a) One of the anhydrite crystals is rod-shaped and is partly embedded in the host mineral (halite). (Width of the photograph = 35 μm).

b) Cluster of short needle-shaped anhydrite crystals. The large bubble is due to a leakage of the cavity. (Width of the photograph = 210 μm).

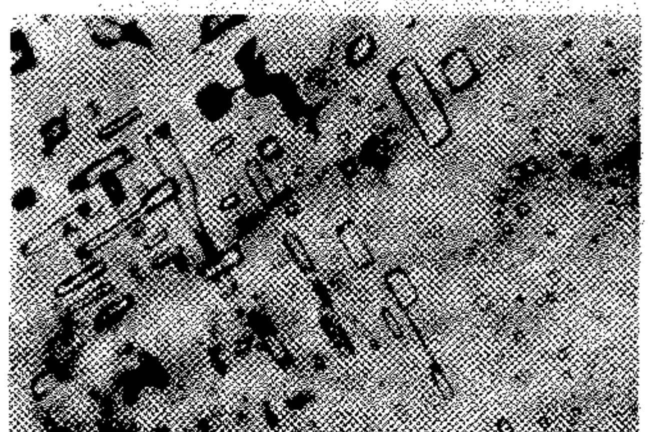


Figure 4. Photomicrograph of a halite fragment obtained by cleavage showing fluid inclusions scattered along cleavage planes. The cavities have crystallographic shapes (negative crystals) and are generally flattened perpendicularly to their planes and elongated. (Rock salt from Tersanne, width of the photograph = 465 μm).

cavities are generally flattened. Most of them have a rectangular parallelepiped shape, the faces being either squares and/or rectangles (the aspect ratio can be higher than 10). The others may be of any shape whatsoever, elongated or ramifying, and are bounded by curved surface portions and flat portions invariably parallel to the crystallographic directions.

One frequently observes, in a given plane, a lateral passage and an evolution from large cavities of elongated or ramifying shape to increasingly smaller and more numerous cavities of crystallographic shape (rectangular parallelepipeds). The dimensions of the cavities vary. Most are between 1 μm and 10–20 μm in their small size (in the case of the faces parallel to the inclusions plane), the smallest being the most numerous (spaced by about 1 μm—Fig. 5); a few have largest dimensions of several hundred μm.

These cavities are filled with brine and those of certain families may enclose a very small shrinkage bubble.

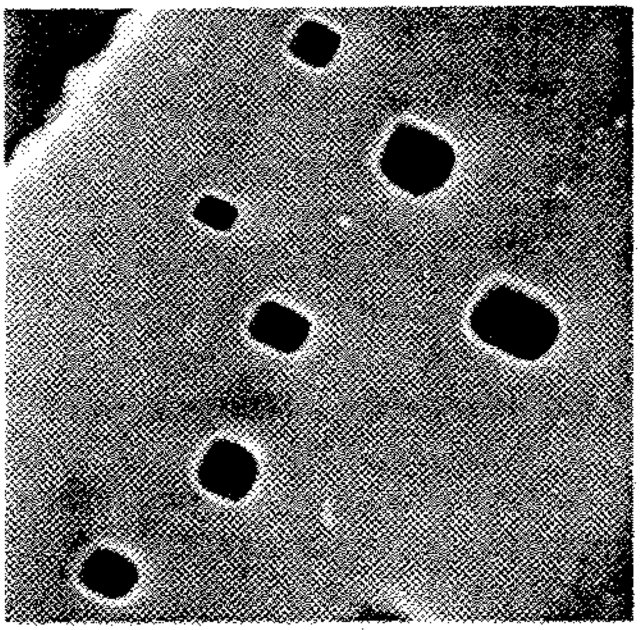


Figure 5. Scanning electron microscope fractography showing cavities arranged along a cleavage plane; the smallest are less than 1 μm in size, their interspace being in the same range. (Rock salt from Tersanne, length of the photograph = 10 μm).

Figure 6. Photomicrograph of a halite fragment obtained by cleavage showing fluid inclusions which had occurred along cracks and are arranged in veils. The cavities may have channel shapes. (Rock salt from Etrez, width of the photograph = 390 μm).

Inclusions along more or less warped surfaces of any orientation (Fig. 6). These inclusions lie along old fractures. The cavities are more or less flattened, from random to rectangular or cubic parallelepiped shapes. They evolve laterally, from large cavities of any shape to increasingly smaller and numerous cavities of crystallographic shape, thus indicating a progressive closing of the surfaces which bounded the original cracks (i.e. wedge-shaped cracks) between which the penetration of a liquid lamina led, through corrosion and depositing processes, to recementations which ultimately isolated liquid inclusions that continued to evolve (Rasumny, 1976).

Because of the irregular orientation of the fracture surfaces relative to the crystallographic directions (although, in readily cleavable crystals like halite, a fracture surface of apparently random orientation often decomposes into portions which follow the crystallographic directions locally), the cavities may assume more complex forms than those discussed earlier, such as networks of more or less anastomosed channels, some parts of which follow the crystallographic directions.

The dimensions and spacing of these inclusions are of the same order of magnitude as the ones discussed before. Certain veils corresponding to very fine cracks have only very small and very closely spaced inclusions. The cavities are filled with brine.

Also encountered are veils associated with solid inclusions. This shows the disrupting effect of the presence of alien elements on the construction of the crystal lattice. The veils are more or less flexuous and form veritable networks in certain halite samples.

Figure 7. Scanning electron microscope fractography showing polycristalline inclusion within a cavity closely moulded on it. The rod-shaped crystals are anhydrite, the others contain potassium. (Rock salt from Tersanne, length of the photograph = 91 μm).

Like the inclusion planes described before, these flexuous veils consequently bear witness to the penetration of fluids in cracks and cleavages that opened up after the crystal lattice had been constituted. But the stresses which opened up these intracrystalline spaces may have affected the crystal either in its internal zone while its growth was not completed, in which case the trapped liquid is seed solution, or subsequent to crystallization, in which case the content of the inclusions may be different from the generating media. The authors made no attempt to distinguish between these genuinely secondary latter inclusions and the pseudo-secondary former inclusions (Ermakov, 1972).

Large isolated inclusions. The cavities may be negative crystals or of any shape (with surface portions following the crystallographic directions more or less), with a largest dimension of as much as several hundred μm; they are filled either with brine or with brine and solids like those described above.

All these zones constitute discontinuities in the crystals and must certainly affect their mechanical behavior.

Solid inclusions. By "solid inclusion" is to be understood an inability to distinguish the associated crystallization lacuna, or a very small lacuna. Here one is dealing essentially with anhydrite in the form of rods (frequently clustered together), squat crystal aggregates and occasionally very thin laminae. The rods and laminae are perfectly limpid and colorless but may contain fluid inclusions themselves, and a few cleavages perpendicular to the elongation can have occurred. They are sometimes linked with the clay-anhydrite or the anhydrite cement that joins the halite crystals together in some cases. The aggregates are variously colored (yellowish, pink, etc.) and their abundance and distribution vary from one part to another of the crystal and from one crystal to another. They are disseminated through the body of the halite or grouped together in the form of veils, thereby underlining growth or recrystallization faces.

The authors have also observed blackish mineral aggregates which they believe to be sulphides and which are systematically associated with clay-anhydrite portions. Thus, in certain samples from the Bresse basin, these opaque minerals included in the halite lie uniformly along intercrystalline surfaces and are connected to the minerals of the clay-anhydrite matrix which separates the halite crystals (Fig. 8b).

Solid inclusions occasionally consist of clays associated with the anhydrite, carbonates, etc., but this listing is by no means exhaustive. For the reasons given, we believe that part of these inclusions existed before the halite recrystallisation.

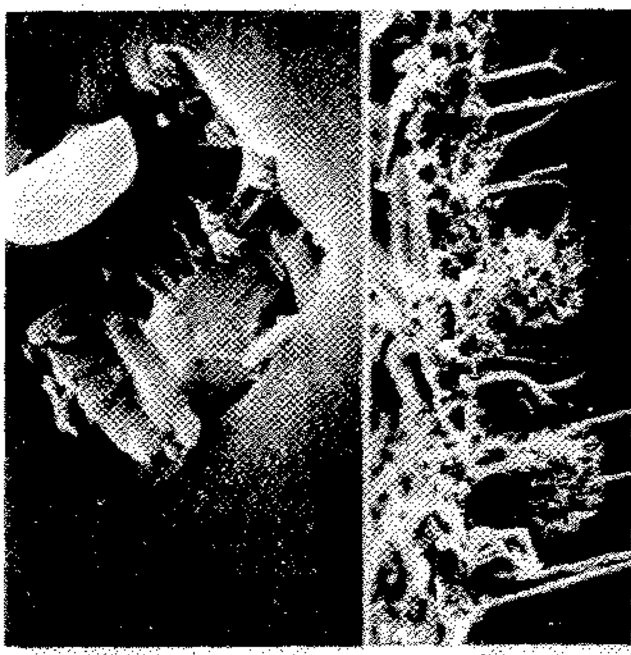


Figure 8. a) Scanning electron microscope fractography showing anhydrite occurring in a cavity in halite. (Rock salt from Etrez, width of the photograph = 9 μm).

b) Scanning electron microscope fractography showing crystalline inclusions in halite scattered along an intercrystalline surface and connected with the clay and anhydrite matrix which occurs along certain halite grain boundaries. (Rock salt from Etrez, width of the photograph = 176 μm).

INTERCRYSTALLINE LACUNAE

The samples of rock salt consist of two types of aggregates: joined halite crystals whose size varies from a few millimetres to a few centimetres, and halite crystals linked to one another by an anhydrite or clay-anhydrite matrix. The grain limits of the first type of aggregate will now be described.

An examination of the intercrystalline surfaces with the binocular (Fig. 10) shows that the crystals are not joined together over the whole of these surfaces. Here and there, the latter are bound in more or less anastomosed wedge-shaped cavities and networks of sinuous channels reminiscent of the inclusions of certain veils of intracrystalline inclusions. These spaces between the grains enclose brine and sometimes gas (possibly air due to a loss of hermeticity during the fragmentation operations). They can be occupied by crystallizations when the crystals are separated for observation. Examination with the scanning electron microscope shows, along the contact lines of numerous grains, empty spaces which are more or less associated to crystallizations and, on the intercrystalline surfaces, depressions which correspond sometimes to corrosion figures, sometimes to growth lacunae (the former being possibly derived from the latter, Fig. 9) and figures corresponding to the wedge-shaped cavities observed with the binocular.

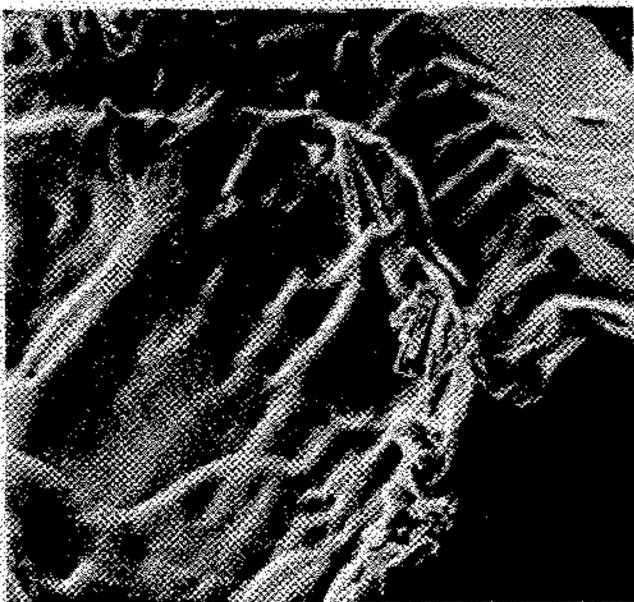


Figure 9. Scanning electron microscope fractography showing halite intercrystalline surface with pits and hillocks. (Rock salt from Tersanne, length of the photograph = 400 μm).

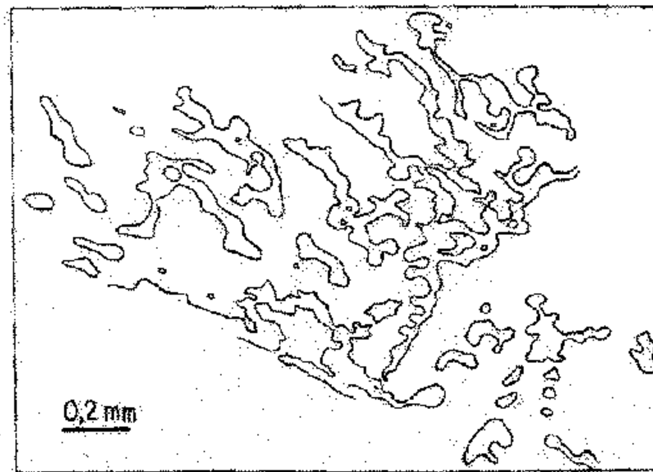


Figure 10. Sketch showing an intergranular microtopography pattern. (Rock salt from Etrez).

In addition, blooming crystallizations appear secondarily along the external lines of separation of the crystals. The SEM shows these recrystallizations as aggregates of small halite cubes and testify to the circulations of fluids and the remobilizings of matter that can take place under the effect of the external medium as a result of the intergranular spaces.

This possibility also certainly exists for the clay matrices which bind other halite crystals, since very fine halite fibres can appear there secondarily. We are therefore confronted with a second type of discontinuity constituted by intercrystalline lacunae.

Examination of Samples Subjected to Uniaxial and Triaxial Stresses

FLUID INCLUSIONS

The samples examined contain veils of fluid inclusions (which were particularly numerous in the crystals of a sample which had been subjected to simple compression). Some of these veils revealed a notable difference with respect to the veils of natural epigenetic inclusions. Above all, the natural mechanical cracks are as a rule filled entirely with brine (either connate or as secondary infiltration) whereas the artificial cracks are filled with air. Only very exceptionally are these breaks dry; capillarity carries the moisture to the ends of wedge-shaped cracks. This moisture authorizes corrosion and recrystallization phenomena which transfrom the break into cavities imprisoning brine and air, with some containing only air (Fig. 11).

Part of the moisture is of internal origin and may have two complementary sources. First, the inclusion brine is in the natural intracrystalline cavities which are opened by

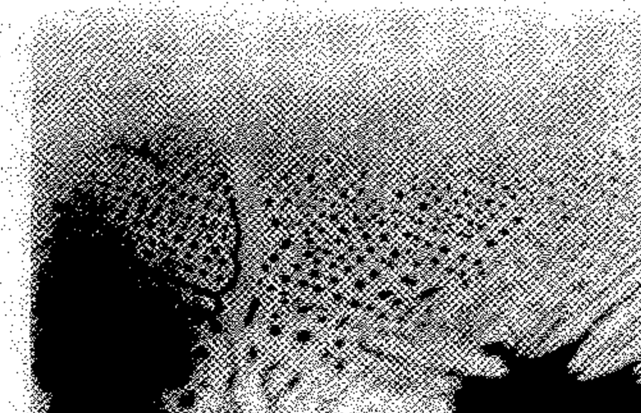


Figure 11. Photomicrograph of a halite fragment from a rock-salt sample tested uner uniaxial stress (compression), showing air-filled cavities arranged along a plane. (Rock salt from Tersanne, width of the photograph = 465 μm).

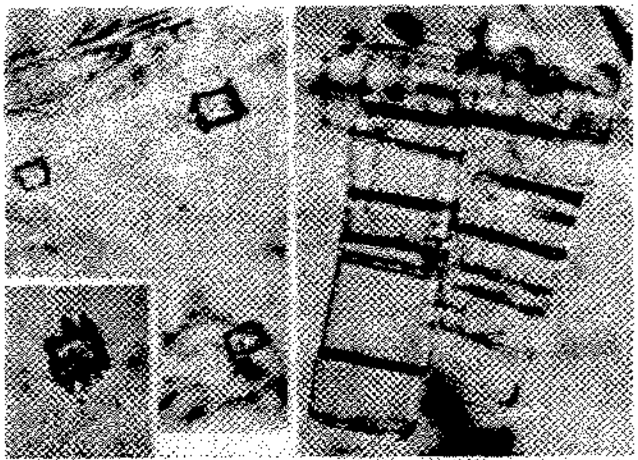


Figure 12. a), b), c) Photomicrographs of a halite fragment from a rock salt sample from Etrez tested under triaxial stresses, showing:
a) and b) : brine-filled cubic cavities which have been deformed and the walls of which have been altered. (widths of the photographs: a = 325 μm, b = 88 μm).
c) : elongated anhydrite crystals which have been broken, along cleavages perpendicular to the elongation, into parts a few μm to 40 μm long. Some of these parts have been offset along the cleavages. (Width of the photograph = 173 μm).

running cracks, and secondly there are the solutions which are interposed in the intergranular cavities and which shift as the joints loosen.

The isometric cavities uniformly distributed through the body of the original halite crystals sustain modifications to their walls during the triaxial tests (Fig. 12a,b). They undergo a slight distortion by comparison with what is customarily observed in "healthy" samples.

SOLID INCLUSIONS

The anhydrite rods react to stresses in particular. They suffer breaks at several places along cleavage lines perpendicular to their elongations (Fig. 12c). This type of examination is not entirely satisfactory since it is not possible to examine the interior of the sample to be tested. It is consequently not possible to assert that certain phenomena which appear to be new did not already exist. Furthermore, transformations could occur which lead to forms similar to those already familiar or which become masked by the recrystallization that can affect deformed crystals (Shlichta, 1968). It accordingly appeared necessary to conduct experiments under the microscope.

Initial Results of Microscope Experiments: Gradual Development of Inclusions Subjected to Mechanical or Thermal Stress

TESTS WITH CRUSHING STAGE

Principle. The halite sample (a fragment obtained by cleavage and having at least two parallel surfaces) is subjected to uniaxial pressure by compressing it between two glass plates maintained parallel to the slide of a binocular or microscope in a mount fixed to the latter. The plates are gradually moved towards each other by a screw exerting pressure on the upper plate (Nachet, *Surplatine à écrasement:* G. Deicha, *Notice 1671*). Observations are carried out during the compression process.

Results. The initial effect of the stressing is to cause the onset of cleavages and oblique breaks which stem partly from the latter. The cracks do not necessarily pass through planes or veils of liquid inclusions. This no doubt depends on the latter's orientation relative to the pressure exerted; however, a break can partly open up a veil of fluid inclusions.

The authors observed some minor changes in the morphology of certain flattened liquid inclusions along the veils. These changes may occur as the result of the opening, not necessarily detectable during the observations of a cleavage running through the inclusion. This remobilizing of material continues when the sample is no longer subjected to stress, and an examination made several days after the experiment showed other changes in the shapes of a few flattened or channel-shaped inclusions. Certain liquid inclusions of rectangular parallelepiped shape uniformly distributed through the body of the crystal are surrounded by a ring of bursts. The anhydrite rods included in the halite break in very numerous places, along the cleavage perpendicular to the direction of elongation.

Limitations of the method. Since the sample is not restrained in the directions perpendicular to the compression force, cleavages and breaks appear very readily. This could mask what happens in the inclusions zone, because the fragments issuing from it separate and the sample breaks up.

TESTS WITH HEATING STAGE

The sample placed in the heating stage was a halite splinter with an area of a few mm^2 and a thickness of between a fraction of a millimeter and 1 millimeter (Sabouraud, 1976). It is proposed to describe the gradual development of two types of fluid inclusions between 20°C and 370°C: a) inclusions belonging to a milky zone (isometric cavities: cubes), and b) flattened inclusions along a surface (a cleavage or old break, cavities of random to crystallographic shape).

Fluid inclusions belonging to a milky zone. The authors observed a group of large cubic cavities (the edges and corners are in fact always more or less rounded) with sides measuring several tens of μm, spaced apart by from one to three times the lengths of their sides. Some contained a very slim anhydrite needle. The temperature rise was fairly slow, being less than 1°C per minute on an average. A gradual change in the shape of the walls was noted quite soon (before 100°C, approximate temperatures), resulting for instance in a hollowing effect in the middle of a face.

At 200°C, the face is still well hollowed out, but above this seems to be less depressed. In actual fact, a further hollowing takes place on either side of the first one (near the edges) whose effect is to tend to flatten the face while increasing the volume of the cavity. A small hollow may persist along an edge. This increase in volume has already been described. Bradshaw et al (1968) attribute it to a greater expansion of the brine than of the surrounding salt. They noted an increase in volume proportional to the temperature, reaching 7.5% at 225°C. Jenks (1971) thinks that this increase helps the formation of a gas-vapor phase inside the cavity. Geguzin and Dzyuba (1973 a) measured it at atmospheric pressure and at 120 bars and carried out calculations by introducing a limit salt-brine pressure resulting from the difference in expansion and the increase in the salt's solubility. In addition (1973 b), they determined the rates at which cativities approach each other as a function of their distances and their respective positions (this movement towards each other can result in cavities merging). The hollowing of the faces and the movements of the anhydrite needles which betray currents in the brine indicate that corrosion phenomena assisting this increase in volume are at work.

Around 300°C the cavities have fairly flat walls and clearly defined edges. A gas bubble appears in each cavity above 300°C, at temperatures which vary with the experiments and the cavities (330°C, 350°C, etc.).

At about 350°C the angles lose their sharpness and the increase in volume is reflected in a clearly visible moving of the cavities towards one another. As this tendency continues, the shapes become rounder and rounder, the gas bubbles become larger, with some of them ultimately occupying almost the whole of their cavities, and the cavities come closer and closer together. At around 370°C observation becomes difficult; some cavities can no longer be distinguished, whereupon the heating was arrested.

Subsequent to cooling, the very small cavities which formed the lactaceous zone can no longer be recognized and there are a few large cavities (empty or filled with air) which did not exist previously.

Oblate fluid inclusions along a surface. There are two examples of the growing development of veils: α, Veil consisting of large cavities whose sections parallel to its surface are isometric and cavities these sections of which are elongated and slightly flexuous.

Quite soon it is possible to observe a gradual change in the shape of the cavities (already substantial at 70°C). Wall hollowing and filling phenomena lead to a lengthening of the "isometric" cavities and to a squatter, rounder shape of the elongated cavities (by a reduction in length and increase in width). Above 300°C the walls become more blurred, but some become clear once more at 315°C, the temperature at which the experiment was halted.

After cooling, a gas bubble appears inside each inclusion. An examination made on the day following the experiment showed a further morphological evolution with many faces forming the walls.

β, Veil consisting of rectangular parallelepiped cavities and cavities of random shape (with contractions) (Fig. 13). Here too the morphological evolution begins fairly quickly and is already perceptible at around 80°C.

When a cavity exhibits constrictions in some parts, the latter thin down more and more and the cavity ends up by splitting up into several separate cavities (Fig. 14 to 16) with each cavity continuing to develop towards an isometric shape. During the authors' experiment, the first division occurred at about 139°C, the second at about 230°C. This is a perfect illustration of a process of natural evolution of the fluid inclusions classically described in the literature on the basis of static observations. Generally speaking, almost all the cavities of initially more or less distorted shape evolve towards a regular shape, with the appearance of faces, and tend towards the fairly isometric negative crystal (beyond 310°C).

Around 230–240°C, the bubbles which existed in certain cavities (probably because of a loss of hermeticity during preparation of the sample) disappear. A gaseous phase appears at 315°C in one of the cavities resulting from the divisions (at 340°C in others) and proceeds to grow. At 365°C (when the experiment ended) this phase had not appeared in all the cavities.

As was the case before, the cavities move towards one another as their shapes evolve and the volume increases, some of them ultimately becoming anastomosed (at 339°C, then 364°C for those in the group in the upper left of Fig. 13). At this latter temperature, the shape of some cavities is affected by the presence of the gas bubble.

Finally, it should be noted that crystallizations occurred on the walls of one of the cavities (240°C, Fig. 16). This

Figure 13.

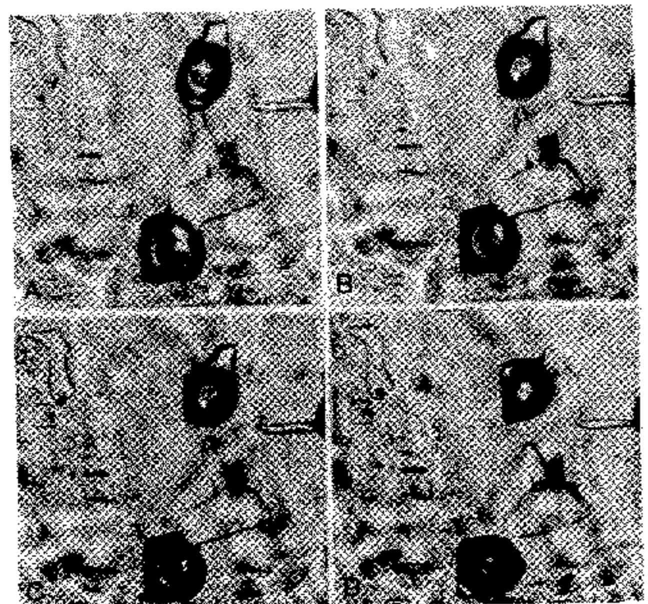


Figure 14.

Figure 15.

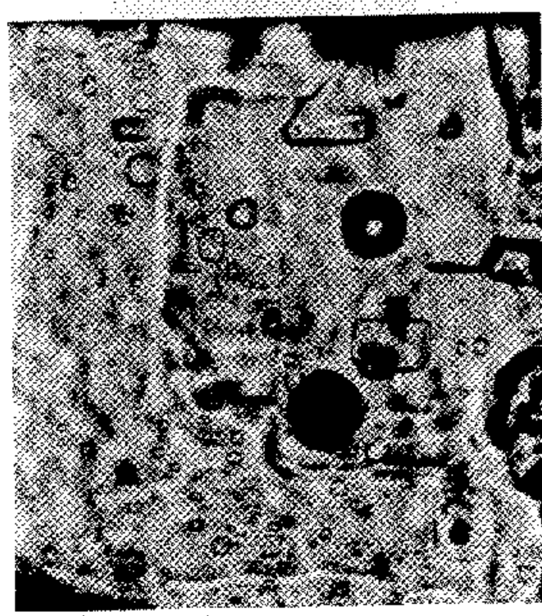


Figure 16.

Figures 13 to 16. Experiments on the heating stage; photomicrographs of a halite fragment obtained by cleavage, heated between 20°C and 339°C, showing the evolution of the cavity shapes. (Rock salt from Etrez).

13. Fluid inclusions arranged along a cleavage surface. t = 20°C. (Length of the photograph = 520 μm).

14. a), b), c), d): Progressive disconnection of an inclusion containing two bubbles (due to a leakage of the cavity). (Length of the photographs = 26 μm).

a) : t = 130°C

b) : t = 138°C

c) : t = 139,9°C

d) : t = 177°C.

15. The inclusions of figure 13 at 300°C. (Width of the photograph = 440 μm).

16. The inclusions of figure 13 at 339°C. (Width of the photograph = 440 μm).

phenomenon was observed by Rasumny (1972) during heating experiments on inclusions rich in salts other than NaCl ($MgCl_2$, etc.), in the course of which, in addition to gradual changes in shape, a veritable aqueous fusion occurred at temperatures above 500°C.

Conclusion. The temperatures attained during the authors' experiments are much higher than those applied to the material constituting the walls of the cavities in which Gaz de France stores natural gas. However, it is likely that the slight evolution observed in the inclusions at under 100°C would have continued had the sample been left at that temperature for some time, since the time factor can partly replace the speed factor (the authors having moved from 20°C to 370°C in the space of a few hours). In fact it was observed that if an accidental drop in temperature followed by a rise in temperature once more occurred, the inclusions continued to evolve even when the temperature did not revert to its initial value. And the same effect was produced when the temperature did not increase for a short while.

CONCLUSION

Aggregates of rock salt halite exhibit two types of discontinuity liable to affect their mechanical properties: 1) Networks of cavities bounding intercrystalline spaces in which fluid circulations and recrystallizations can take place under the effect of the environment and which reduce the contact areas between individual crystals; 2) Zones of variously distributed intracrystalline lacunae containing brine and/or minerals and capable of evolving under mechanical and/or thermal stress. Various authors have shown that the fluid inclusions shift in thermal gradients (Bradshaw, 1969; Anthony and Cline, 1974; Holdoway, 1974) and in acceleration fields (Anthony and Cline, 1970; Wilcox, 1972). Without imposing a gradient, a rise in temperature causes the shapes of the inclusions to evolve and their volumes to increase, leading ultimately to possible coalescences.

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