

Deformation mechanisms operating in naturally deformed halite rocks as deduced from microstructural investigations

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Abstract

In this paper we describe the microstructures of naturally deformed rock-salt samples from the Asse salt anticline, FRG, as revealed by a chemical polishing-etching procedure, and by gamma-irradiation. Evidence is presented for the operation of dislocation creep processes, accompanied by extensive strain-induced grain boundary migration. Grain boundaries can be shown to have contained thin brine films during recrystallization, suggesting that solution-precipitation processes could also have been important deformation mechanisms. Recrystallization and solution transfer processes have not been reported in most experimental work to date, thus casting doubt on the validity of extrapolating these data to predict the long-term creep behaviour of salt during natural flow.

Introduction

During halokinesis evaporite rocks are subjected to large deformations. Studies of salt flow and salt dome development (Jackson & Talbot 1986) rely heavily on an understanding of the mechanical properties of rock-salt at the low strain rates of these deformations.

One way of obtaining this information is extrapolation (towards low strain rates) of experimentally calibrated constitutive laws describing the thermomechanical behaviour of salt rocks (Wawersik & Zeuch 1986). The standard 'materials science' method of testing the validity of such extrapolations is to determine the deformation mechanisms active during deformation of salt in nature and to compare these with those operating during experimental deformation (Frost & Ashby 1982; Nicolas & Poirier 1976).

A large amount of experimental data exists on the deformation of rock salt (see e.g. Carter & Hansen 1983 for a recent review). In contrast, sur-

prisingly few studies are available of deformation mechanisms and microstructures in naturally deformed rock salt (Muehlberger & Clabaugh 1968; Talbot 1981; Carter & Hansen 1983; Jackson 1985; Gresner 1980; Brokmeier 1983). Although most workers have concluded that deformation of rock-salt in nature involves dislocation processes (e.g. Schwerdtner 1968) and recrystallization (involving grain boundary migration), it is by no means clear whether this recrystallization is dynamic or static (Carter & Hansen 1983). Nor is it clear how extensive grain boundary migration can take place at the relatively low temperatures (around 100° C) experienced by most salts during deformation under natural conditions, particularly since recent experimental work (Guillopé & Poirier 1979) suggests that (dynamic) grain boundary migration in rock-salt is impossible below 500° C.

The aim of the present paper is twofold. Firstly, a number of new and relatively easy sample preparation techniques for microstructural work on rock salt are described. Secondly, a detailed micro-

structural study is presented for a set of salt samples from the Asse II mine (W-Germany), in an attempt to elucidate the microscale mechanisms during natural deformation.

Sample preparation techniques

The material used in this study was obtained from the Na2SP unit at the 800 m level in the Asse mine (Essaid & Klarr 1982). Cores, 11 cm in diameter, were drilled dry at this location, sealed in air tight bags after extraction, and handled further in a room with relative humidity below 20%. (This humidity control can be achieved fairly simply by dry compressed air flow into a small, sealed room at a rate of ~50 liter/minute (G. Halmos, pers. comm. 1980)). Preliminary sectioning into rectangular blocks of about 3 cm edge was done dry with a small band saw. Further (dry) cutting was then performed with a low velocity metallurgical saw (Buehler Isomet), equipped with a jeweller's saw blade. This method allowed rapid cutting and produced almost no visible damage to the sample. Grinding was carried out on emery paper, with a final polish using 0.3 micron alumina powder on a lens tissue. Samples intended for inherent brine content studies were ground and polished dry. Others were prepared using oil (Shell S-4919). Thin sections were prepared using a high viscosity cyanoacrylate cement (loctite). The following additional preparation techniques were used:

- a) chemical polishing + etching of mechanically polished surfaces, in a slightly undersaturated (~5.5 molar) NaCl solution, containing about 0.8 wt% $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. After gently agitating for about 10 seconds, the etchant was removed from the surface using a powerful jet of n-hexane, and the section dried in a stream of hot air. (This method eliminated surface scratches and revealed the grain and subgrain boundaries);
- b) a dislocation etching procedure on (001) cleavage planes, following the procedures described by Skrotzki (1980);
- c) epitaxial overgrowth of fine NaCl crystallites on etched surfaces was produced by coating the sample with a thin layer of the etchant described

under a., and allowing it to dry slowly. This technique produced results similar to those of Friedman et al. (1984), providing information on the crystallographic orientation of grains;

- d) samples were cut into $3 \times 3 \times 15$ mm blocks, which were carefully broken. Fractures commonly formed along high angle grain boundaries, thus exposing the grain surfaces which were studied by scanning electron microscopy;
- e) selected samples were irradiated with gamma-rays using the Gamma-Irradiation Facility at the Energieonderzoek Centrum Nederland, Petten, The Netherlands. Irradiation temperatures were around 120°C, dose rates varied between 20 and 80 MRad/h and total doses were about 5×10^9 Rad (Urai et al. 1985). This technique decorated otherwise invisible microstructures, providing important additional microstructural information.

Microstructural observations on polished and etched samples

Grain and subgrain structure

The Speisesalz (Na2SP) samples are almost pure halite (~99%), with polyhalite ($\text{K}_2\text{MgCa}_2(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$) as the main impurity phase. The material has a grain size of 3–10 mm and a moderately developed grain shape fabric (Fig. 1). Reflected light examination of etched surfaces revealed two types of etch patterns (Fig. 2a): (i) a deeply etched set of continuous lines defining a cellular pattern, frequently decorated by small bubbles and polyhalite grains, and (ii) a less clearly etched set of undecorated lines defining a cellular pattern with a smaller cell size (average diameter 0.2 mm).

The epitaxial technique (Fig. 2b) showed the deeply etched lines to correspond to high angle grain boundaries and the less deeply etched lines to correspond to subgrain boundaries, with misorientation rarely more than a few degrees. Results of the dislocation etching experiments are in agreement with this interpretation. The patterns produced by this technique (Fig. 3a) are interpreted to represent a population of free dislocations inside a

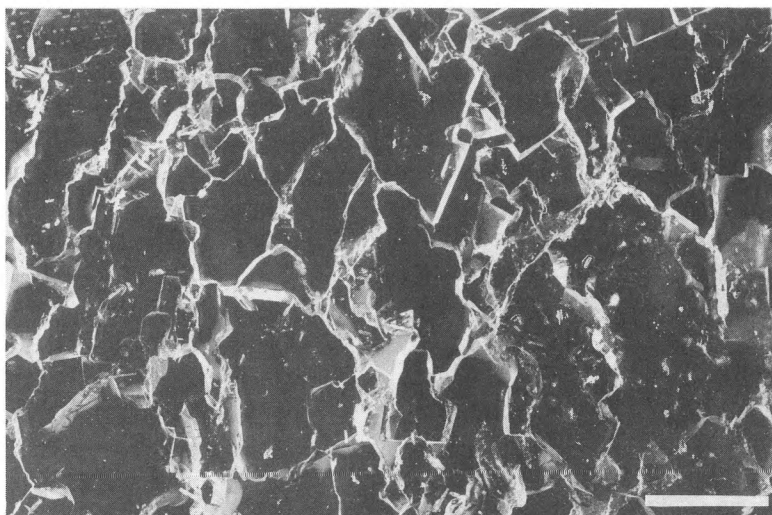


Fig. 1. Dark field, transmission optical micrograph of the general grain structure of the material studied. Note that after careful thin section preparation most grain boundaries were invisible using this illumination. The fine cracks along the grain boundaries were generated by rapidly cooling the thin section with a jet of ether. Scale bar is 5.0 mm.

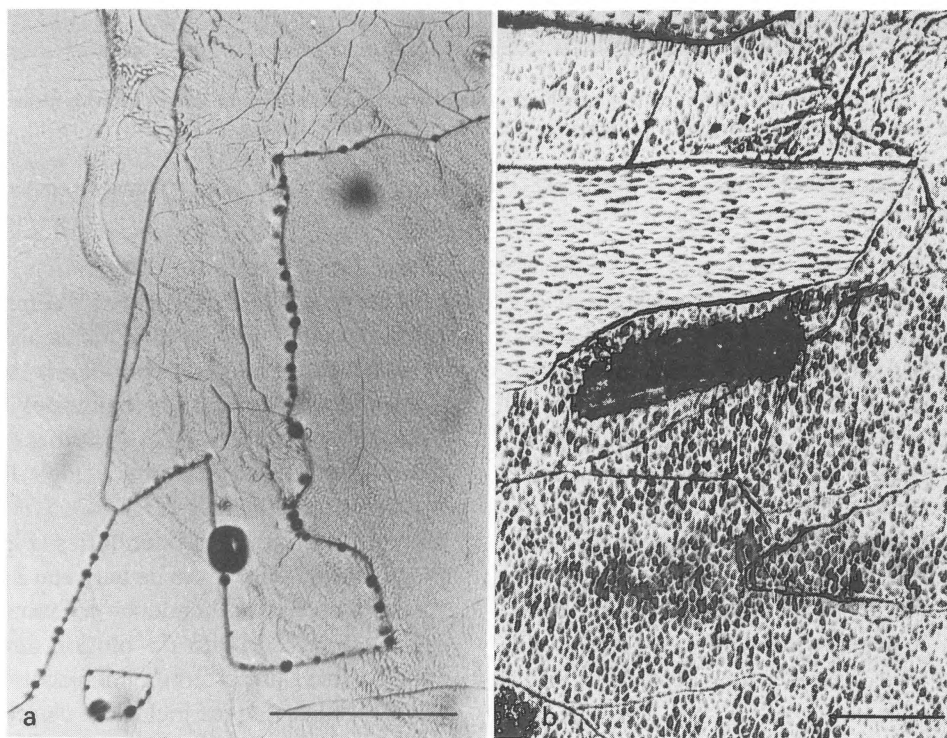


Fig. 2a. Reflection optical micrograph, showing the two types of etch patterns produced: a pronounced pattern with numerous inclusions (cf Fig. 4) outlining grain boundaries, and a less pronounced pattern of subgrain boundaries. Scale bar is 0.2 mm.

Fig. 2b. Epitaxial growth pattern on an etched surface (comparable to the one shown in Fig. 2a), showing small misorientations across subgrain boundaries and large misorientations across high angle grain boundaries. Black areas are polyhalite inclusions. Scale bar is 0.2 mm.

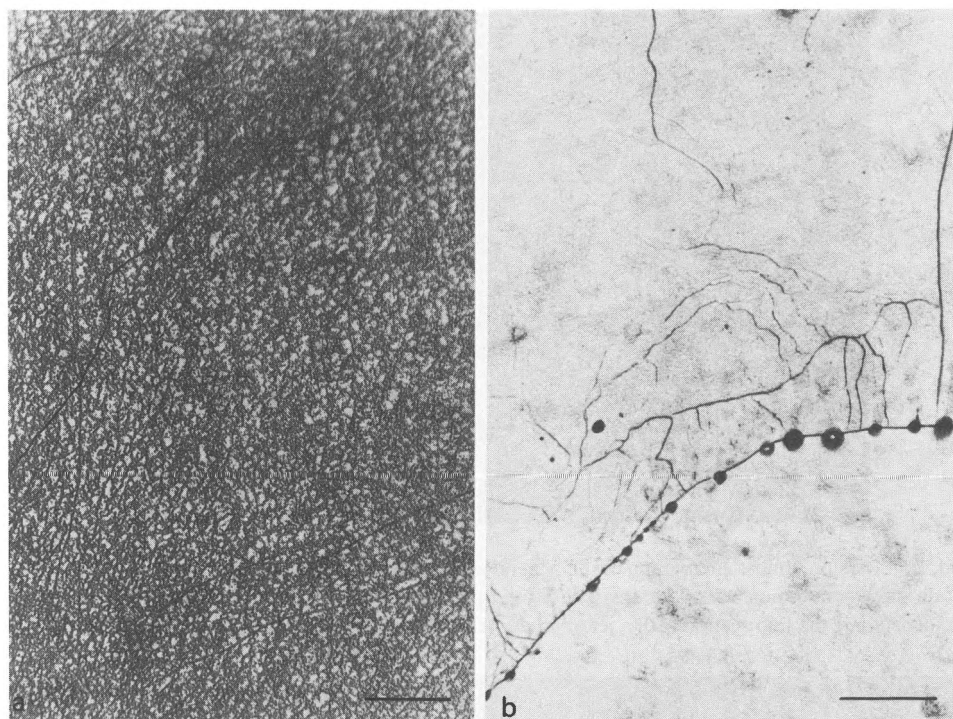


Fig. 3a. Reflection micrograph of the etch pit pattern observed on (001) cleavage planes of some grains, showing dislocation etch pits, part of which are arranged to form subboundaries. Scale bar is 0.05 mm.

Fig. 3b. Reflection micrograph showing a grain with a well developed subgrain structure, being replaced by another grain, which contains no discernible substructure. Scale bar is 0.1 mm.

network of subgrain boundaries consisting of arrays of dislocations, with the same cell size as defined by the less clearly etched lines mentioned above.

Grain boundaries (see also Spiers et al. 1986) range in morphology from euhedral to irregular, lobate or serrated, with angles at triple junctions often approaching 120 degrees. The grains generally have an irregular, lobate shape. Only about half of the grains in the sample show a subgrain structure, which often seems to be truncated along the boundary of a substructure – free grain (Fig. 3b).

Grain boundary structure and brine content

In transmitted light, the above mentioned small bubbles along high angle grain boundaries can be

seen to be the terminations of a complicated network of grain boundary fluid inclusions, present on at least 80% of all grain boundaries (similar structures are also present at halite-polyhalite interfaces). The inclusions have all shapes from subcontinuous films through worm or finger like tubes, to isolated bubble arrays (Fig. 4a). S.E.M. examination of 'parted' grain boundaries (Fig. 4b) shows the tubes and bubbles to be between 2 and 10 μm in size and to have a tendency to assume negative crystal shapes. In situ dissolution and disruption experiments under the microscope (Roedder 1984b) reveals that the inclusions can contain brine, or brine and gas. This gas can be at higher than atmospheric pressure and is soluble in water (observations + odour indicate the presence of H_2S). Intragranular fluid inclusions are sometimes observed in the material studied. When present, they can sometimes be seen to define arrays outlining

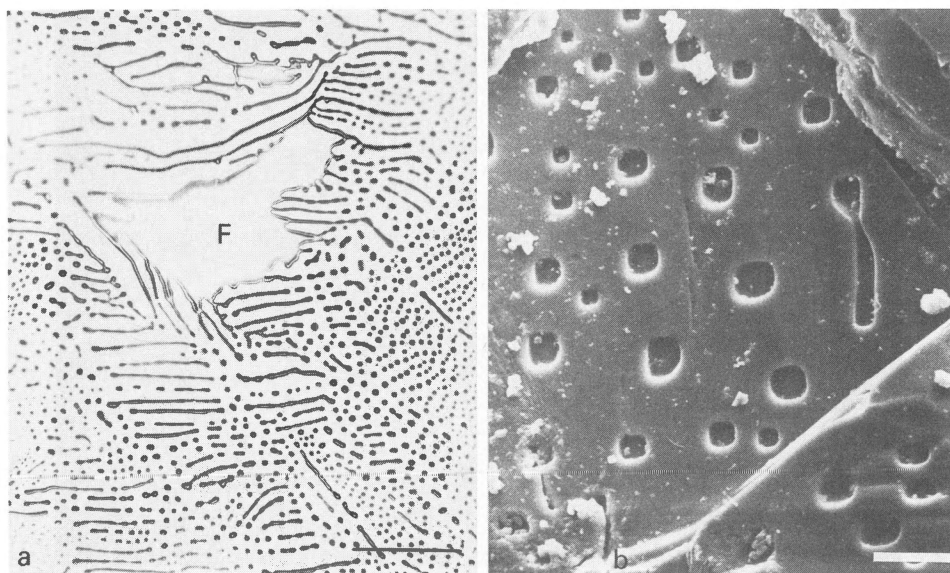


Fig. 4a. Transmission optical micrograph of grain boundary in plan view, showing fluid inclusion morphologies (containing mainly gas in this case). Note how the conspicuous film segment (F) 'fingers out' into arrays of tubes and bubbles. Scale bar is 0.2 mm.

Fig. 4b. Scanning electron micrograph of a mechanically parted grain boundary, showing the tendency of grain boundary fluid inclusions to assume negative crystal shapes. Scale bar is 0.02 mm.

'ghost' grain boundaries (i.e. former grain boundary positions, cf. Bachmann 1985), or healed cracks. Similar structures are sometimes formed by polyhalite grain networks, and identical structures formed by kieserite grains in carnallite and bischofite have also been described (Leng 1945; Urai 1983; Urai & Boland 1985).

Total water content of the samples is about 0.05 wt%, at least half of which represents the water of hydration of polyhalite, a substantial part of the remainder being the grain boundary fluid. Observations agree in order of magnitude with measurements of the water content of the Speisesalz (Jockwer 1981), but while Jockwer (1981, page 14) only mentions 'adsorbed water' on grain boundaries, our observations clearly show the existence of a true liquid phase.

Microstructural observations in irradiated samples

The irradiated samples were opaque in sections thicker than 0.02 mm, in thinner sections they had a

deep blue colour (Przibram 1956), due to irradiation induced lattice defects (Levy et al. 1973). This blue colouration was heterogeneously distributed on all scales, decorating microstructural features which were invisible before irradiation.

At high magnification the colouring was found to be distributed in two ways: (a) in an irregularly developed cellular pattern, with a cell size of a few microns (Fig. 5), and (b) in sharply defined planar arrays (in a number of cases shown to be parallel to (110)), often present in two perpendicular sets (Fig. 6).

Then, superimposed on a coarser scale, a roughly equiaxed, cellular pattern of curved bands was developed in about half of the grains. These bands were either more (Fig. 5) or less (Fig. 6) intensely coloured than the enclosed area, these two types sometimes being laterally transitional (see Fig. 5). The 'light' bands could be shown to correspond to the subgrain boundaries which were revealed by the etching procedure, while the 'dark' bands were not etched.

Grain boundary regions were generally less in-

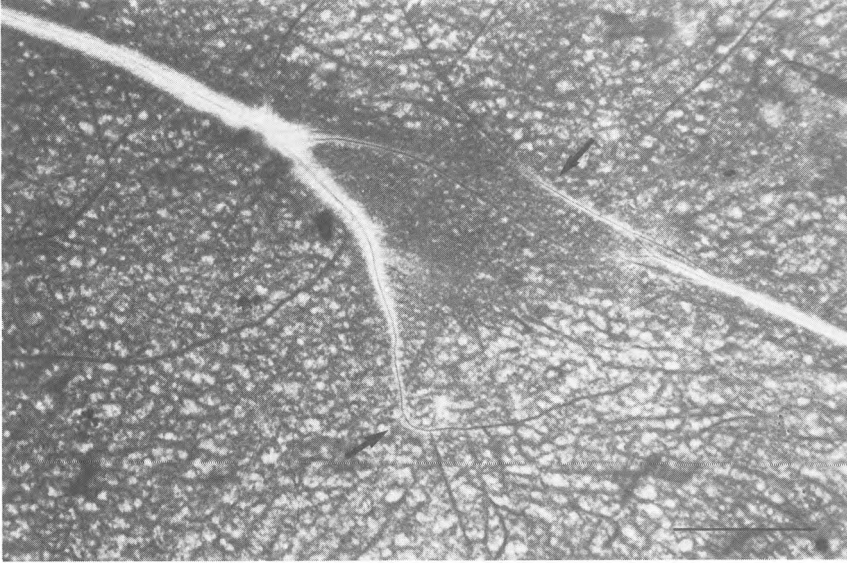


Fig. 5. Irregularly developed pattern of irradiation colouring, defining a fine cellular pattern of a cell size of a few microns. Part of the larger scale network shown in Figs. 7 and 8 can also be seen, with a 'light' band laterally changing into a 'dark' one (arrow). Scale bar is 0.1 mm.

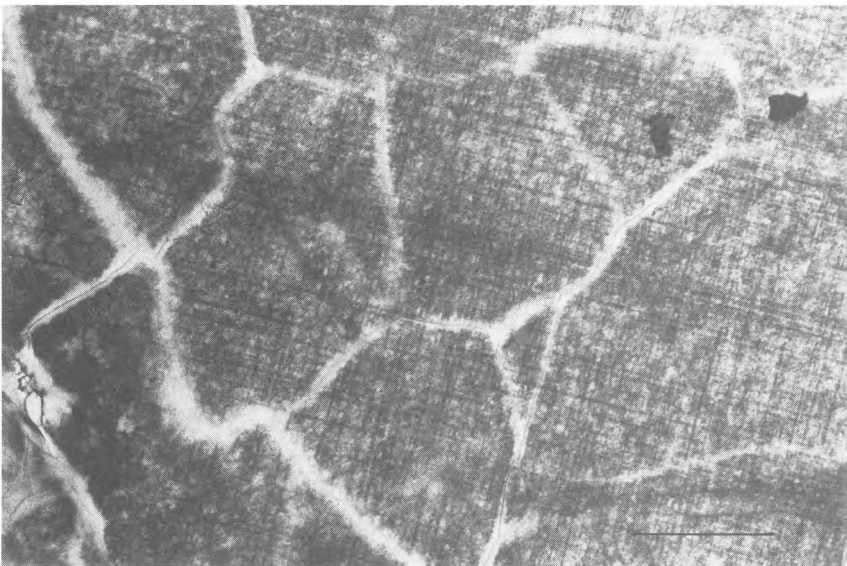


Fig. 6. Irradiation colouring, developed in two perpendicular planar arrays. (In this photograph the planes are not exactly parallel to the line of sight; the bands are in fact more discrete.) Note also the conspicuous pattern of less intensely coloured bands, outlining subgrain boundaries. Across some of these, small changes in direction of the planar arrays can be detected. Scale bar is 0.1 mm.

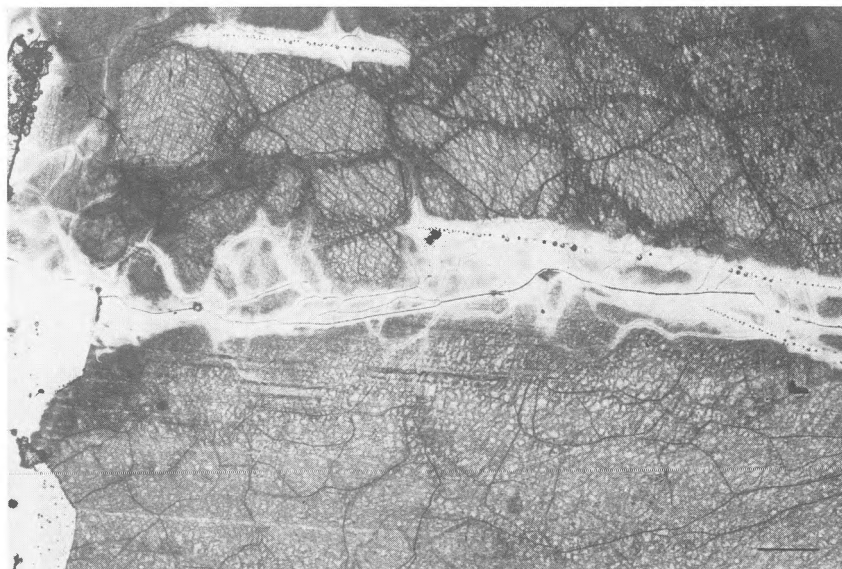


Fig. 7. Irradiation colouring, showing the fine cellular pattern of Fig. 5, a coarser pattern of bands which are more intensely coloured than the areas they enclose, and a less intensely coloured grain boundary. Scale bar is 0.1 mm.

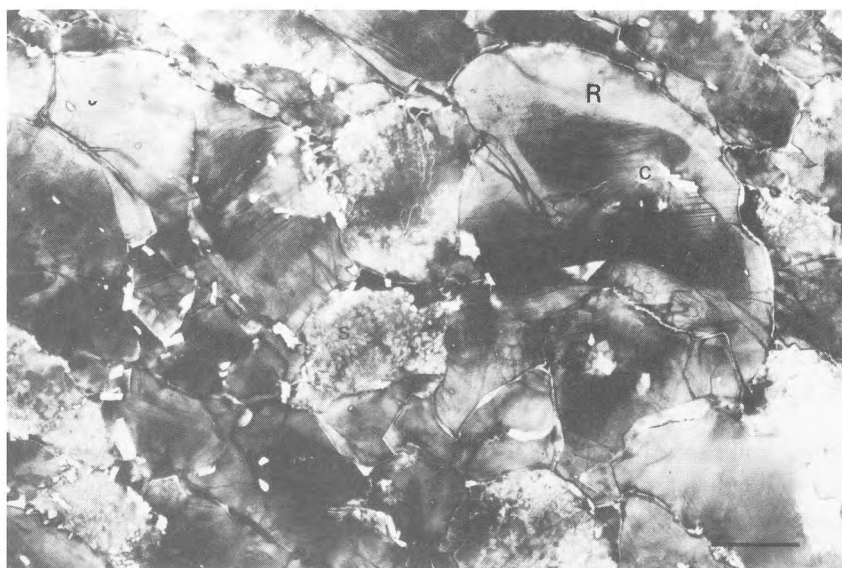


Fig. 8. Transmitted light micrograph of an irradiated sample, showing a grain with a dark core (C) and a less intensely coloured rim (R), which is interpreted as an overgrowth structure; and grains with a well developed subgrain structure (S). Scale bar is 2 mm.

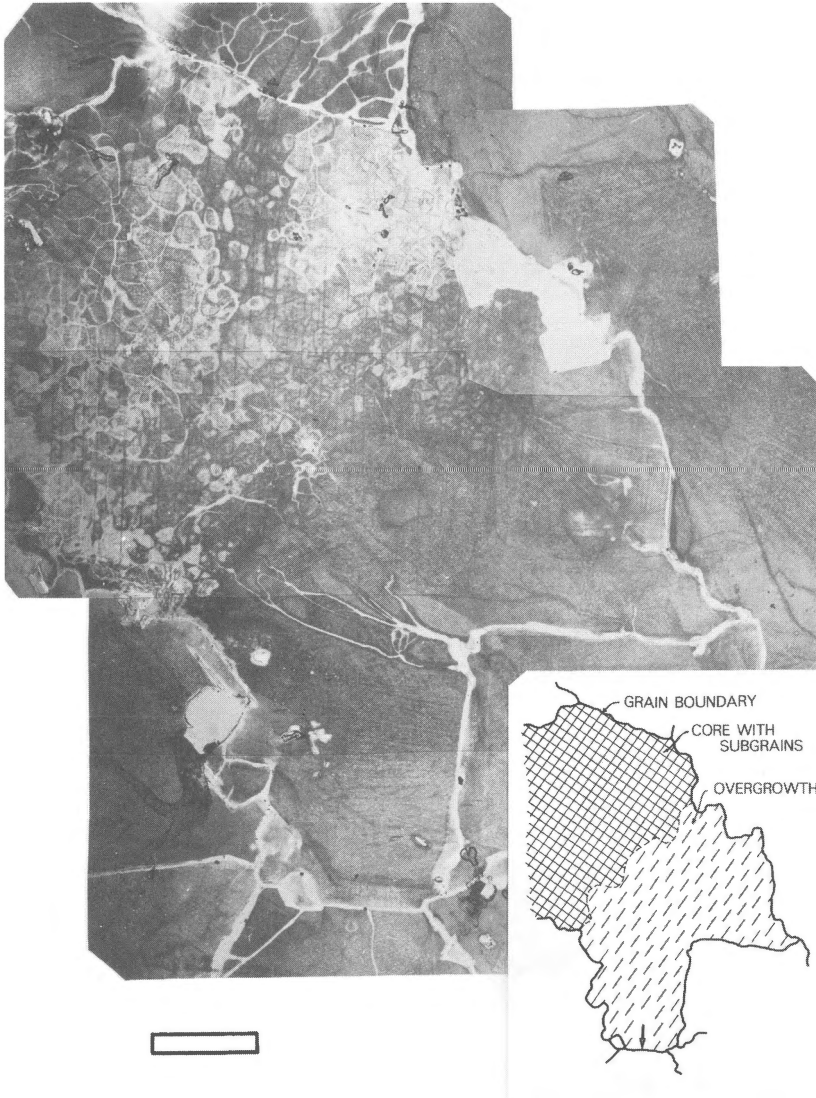


Fig. 9. Transmitted light micrograph of an irradiated sample, showing a grain with a core containing subgrains and decorated slip bands, and an overgrowth without subgrains (see line drawing). Scale bar is 1 mm.

tensely coloured than grain interiors (Fig. 7), as were haloes around polyhalite grains and regions around healed cracks. Similar structures are also described in Holdoway (1979), Levy et al. (1973) and Wilkins et al. (1981).

Finally, on a slightly larger scale, almost all grains show variations in the intensity of colouring, defining the following patterns: (a) in many grains a darker 'core' (with or without subgrains) and a

lighter 'rim' (Fig. 8, Fig. 9) was present; (b) less frequently subgrains were developed in the rim (or overgrowth) part of a grain (Fig. 10); and (c) grains with overgrowths appeared to be replaced by a neighbour in a number of cases (Fig. 10).

The intensity of irradiation induced colouring (at constant dose rate, total dose and temperature) is known to be very sensitive to small changes in dislocation density and/or the concentration of

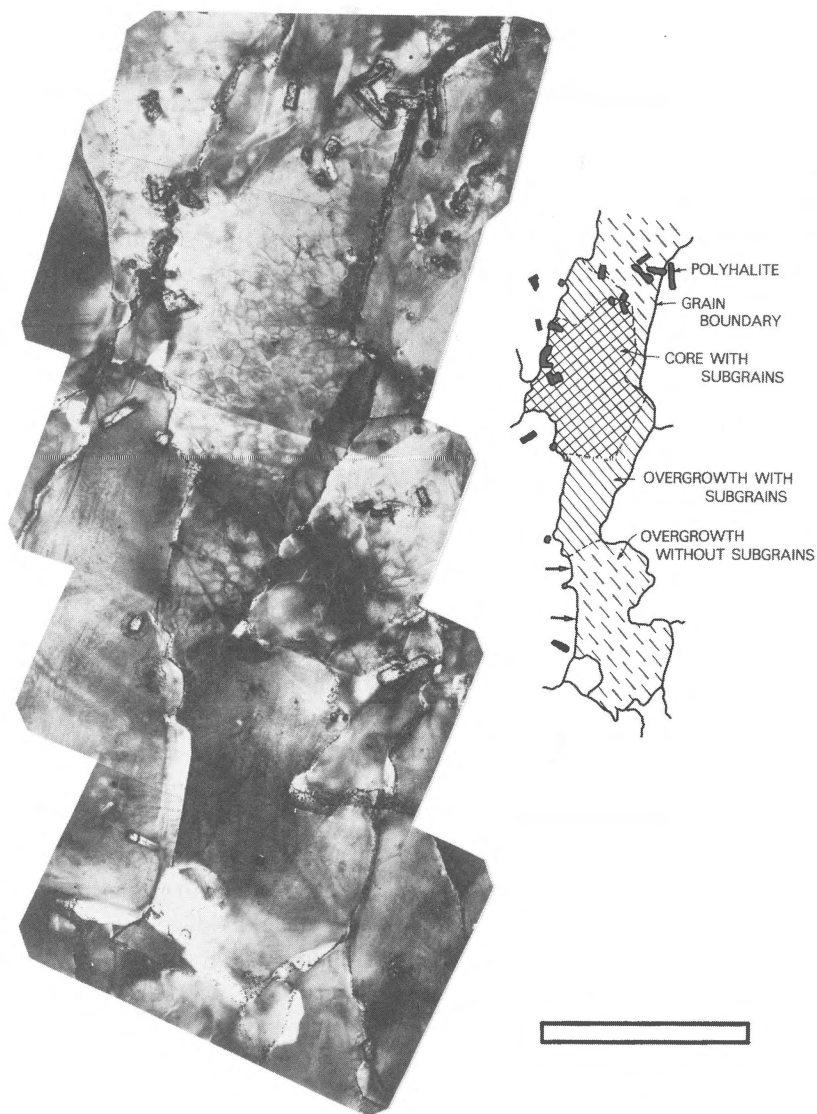


Fig. 10. Transmitted light micrograph of an irradiated sample, showing a grain with a core containing subgrains, an overgrowth with subgrains and an overgrowth without subgrains (see line drawing). These structures are interpreted to be diagnostic for dynamic recrystallization. Scale bar is 2 mm.

solid solution impurities (Levy et al. 1973). We interpret the observed microstructures accordingly: the planar arrays parallel to (110) outline slip bands, and part of the subgrain structure is decorated because of the concentration of dislocations in subgrain boundaries. On the other hand, the less intense colouring of other subgrain boundaries and the high angle grain boundaries may well be due to

impurity (e.g. Ca^{2+}) segregation. Similarly, the concentric bands of colouring (see Figs. 8, 9 and 10) are interpreted to represent very small differences in impurity concentration, marking successive positions of a grain boundary during its migration, possibly due to changes in grain boundary (fluid) composition during recrystallization (see for example Ten Have & Heijnen 1985).

Discussion

Grain boundaries

The fluid films and the fluid inclusion morphologies found on almost all grain boundaries provide clear evidence for the operation of a surface energy driven solution-precipitation process in which a subcontinuous fluid film breaks up into an array of tubular, and later equiaxed, fluid inclusions (Urai et al., 1986a). The process (see Fig. 3 in Urai et al. 1986b) is analogous to the formation of fluid inclusions by the healing of a crack (Lemlein & Kliya 1960). The grain boundaries outlined by fluid inclusion trails are therefore interpreted to have contained fluid films during deformation. Such fluid films have been shown to dramatically enhance grain boundary mobility during experimental deformation of the material studied (Spiers et al. 1986; Urai et al. 1986b) and also in carnallite (Urai 1985) and bischofite (Urai 1983).

Deformation mechanisms

The samples investigated in the present work have a deformation history consisting of at least two stages:

1. the formation of the Asse salt ridge, in which large strains were imposed on the material at temperatures probably below 100° C. This stage may have consisted of several episodes (Kokorsch 1960, Urai & Boland 1985), and
2. convergence of the mined galleries at the site of extraction of our samples. This has occurred at about 40° C, with permanent strains less than a few %.

Keeping this in mind the microstructural observations reported above are interpreted as follows:

The subgrain and etch pit structures found in many grains clearly point to the operation of dislocation creep processes, involving slip and subgrain formation (polygonization). This is also consistent with the presence of the crystallographic preferred orientations in Asse salt (Brokmeier 1983). Comparison of the subgrain diameters with the subgrain size vs. deviatoric stress plots of Car-

ter et al. (1982) indicates that the deviatoric stresses involved were of the order of 0.8 MPa (in agreement with the results from Carter et al. 1982 who studied Asse samples from different locations). The low misorientations across subgrain boundaries indicate that 'rotation recrystallization' (Guillopé & Poirier 1979; Urai et al. 1986a; Poirier 1985) was not a dominant process. However, the material has clearly undergone extensive recrystallization by grain boundary migration. The observations of lobate grain shapes, grains with subgrains developing overgrowths, subgrains developing in overgrowth structures, and grains with overgrowths being replaced themselves (Figs. 8, 9, 10) are characteristic of a dynamic (syntectonic) recrystallization process, associated with the large deformations during formation of the Asse anticline, with a less important metadynamic (post-tectonic) overprint.

The process envisaged is similar to that found in bischofite (Urai 1983; 1987) and octachloropropane (Means 1983 see also Urai et al. 1986a), where the dominant process during dynamic recrystallization is simply the migration of pre-existing high angle grain boundaries, without the formation of new grains *sensu stricto*. In these materials the formation of amoeboid grain shapes, cyclic changes in the size of individual grains and reversals of grain boundary migration direction are common microstructural processes.

In rock salt deforming under dry conditions such dynamic recrystallization processes only operate at relatively high homologous temperature (T/T_m) conditions. In the rocksalt samples studied the strong increase in grain boundary mobility due to the presence of fluid films resulted in very similar processes, but at much lower temperatures. With the exception of Spiers et al. (1986) and Skrotzki & Welch (1983) experimental work on rocksalt to date (see Carter & Hansen 1983) failed to reproduce these recrystallization processes and therefore extrapolation of these mechanical data to the low strain rates characteristic of natural salt flow should be treated with caution.

Finally, some of the slip bands found in many grains and some of the free dislocations may be associated with gallery closure after mining.

Solution-precipitation creep

In an investigation of naturally deformed carnallite samples from a nearby locality in the Asse mine Urai & Boland (1985) have demonstrated the operation of recrystallization processes very similar to the ones described above. In addition, they have provided evidence for the operation of solution transfer processes, in combination with dislocation creep. Based on a model of solution-precipitation creep Spiers et al. (1986) have argued that during deformation of rocksalt in nature, solution-precipitation creep processes (accompanied by grain boundary sliding and migration) should also be important deformation mechanisms. Similar processes were inferred to operate in naturally deformed bedded salt by Borns (1983).

The microstructural observations reported in this paper do not provide direct evidence for solution-precipitation creep (e.g. preferential overgrowth sites, Squires et al. 1963; Harris & Jones 1963). However they do provide evidence for the presence of a thin brine film network along grain boundaries, and for paleostress levels at which in such materials, according to the model proposed by Spiers et al. (1986) deformation occurs by the combined operation of solution-precipitation and dislocation processes. The absence of direct evidence for solution-precipitation creep processes in the samples studied is not a strong argument against this interpretation, because in an aggregate deforming by the combined operation of solution-precipitation creep and dislocation processes, accompanied by extensive grain boundary migration, the chances of preserving a system of preferential overgrowth sites are small (see Urai 1983).

Finally it should be noted that the Na₂SP samples studied in this work are quite 'dry' compared with other salt rocks (Roedder 1984a), although they may have contained more brine during deformation. Therefore, similar brine enhanced dynamic recrystallization processes are probably common during rocksalt deformation in nature.

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