INVESTIGATION OF THE INITIAL STAGES OF PLASTIC DEFORMATION OF ROCK SALT CRYSTALS

R. I. GARBER and L. M. POLYAKOV

Physico-Technical Institute, Academy of Sciences, Ukrainian S.S.R.

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Plastic deformation processes in rock salt, involving the formation of elementary displacements that constitute a special deformations stage, are investigated. Further deformation results in single slip bands. The activation energy required for the annealing of the residual stresses of the elementary displacements is half that of the slipping bands. An additional attenuation of light has been detected near the traces of the elementary displacements. It is proposed to attribute this to the influence of systems of line inhomogeneities differently oriented on both sides of each trace. It is established that certain traces of elementary displacements contract after removal of the load, similar to the case of elastic twins of sodium saltpeter. It is established that the trace of the elementary displacement on the lateral surface of the crystal has a smooth profile, extending over a distance of approximately 1500 A. The smooth form of the profile is well explained by the influence of surface-tension forces, which are in thermodynamic equilibrium with the additional residual stresses.

SLIPPING in plastic deformation is usually described as simple shear, shear with bending of the glide planes, twinning over irrational planes, displacement, and multiplication of the dislocations. However, the large number of observed phenomena makes it impossible to describe this complicated process with any one mechanism alone. For a detailed analysis it is best to subdivide the entire process into individual stages, as was done in the investigation of mechanical twinning.[1,2]

The stages of the process are best established in accordance with the type of the deformation elements and the magnitude of the stress at which they appear. In the deformation of rock-salt crystals, we can distinguish the following stages (σ is the normal stress in g/mm^2):

Elastic deformation	$\sigma < 70$
Elementary shear	$\sigma \approx 70$
Single slip band	$\sigma \approx 100$
Stacks of slip bands	$\sigma > 100$
Isotropic scattering of light (Tyndall cone)	$\sigma > 120$
Asterism	$\sigma > 600$
Failure	$\sigma > 2000$

In the present paper we consider the initial stages of deformation, which terminate in formation of a single slip band. The first stage is elastic deformation. It is characterized by homogeneous distribution of the strains in a homogeneous stressed state, and by the absence of other changes of shape, dimensions, or physical properties of the specimens. In elastically-deformed specimens we can observe, under crossed polarizing prisms, homogeneous transmission of light, the intensity of which increases with increasing stress. The initial illumination is restored after removal of the load. When an elastically-stressed specimen is rotated, the illumination is restored every 90°. For well annealed rock-salt crystals without imperfections, cut along the (100) cleavage planes, these stages correspond to a compression or tension stress $\sigma < 60-70$ g/mm^2. Changes in photo-conductivity and color at lower stresses, described in references 3 and 4, must be attributed to uneven distribution of stresses over the volume of the specimen. The yield point as determined by this method cannot be connected directly with any element of deformation, and characterizes merely the presence of local overstresses in the specimen. Elementary shears occur in annealed specimens that have no defects, under slow smooth loading. In experiments with a rigid loading device, shears of this type appear at a deformation rate up to 0.5% per minute. At a higher rate of deformation, this stage does not appear.

The appearance of elementary shear in a specimen is manifest by a characteristic clear strain pattern in polarized light, which is retained in a somewhat modified form (the illuminated portions become noticeably darker) also after the load is removed from the specimen. The appearance of intersecting elementary shears changes radically the picture of the stress distribution. Stresses of opposite sign cancel each other, while those of equal sign add together. The elementary shears, as a rule, originate on the surface; as the stress increases, the region of shear penetrates deeper into the specimen and gradually grows through the entire specimen. The shear appears on a surface defect, capable of causing the necessary stress concentration. Stepanov[5] has shown that the shear can be localized by producing a scratch several microns deep on the surface of the specimen. It is characteristic, that annealing at 600°C for six hours does not eliminate the effects of the scratch.

(110)

FIG. 1. Crystal with incomplete traces of elementary shear along the (110) plane, which break off inside the specimen. Polarization microscope, crossed prisms. Compression stress along the (010) axis – 70 g/mm² (× 25).

Figure 1 shows a photograph of a rock-salt crystal in transmitted light, under crossed polarization prisms, after several traces of the elementary shear have been produced in it. Some of the traces do not pass through the entire specimen. A small increase in stress in the specimen results in new traces and a certain elongation of those already existing. Electron-microscope photographs of the cleavage surface perpendicular to the slip planes show curving and other distortion of the tapering trace of the elementary shear near its vertex (Fig. 2). The increase in the resistance to further spread of the shear can be attributed to redistribution of the stresses. The traces of the elementary

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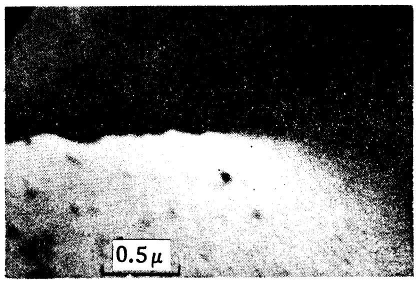
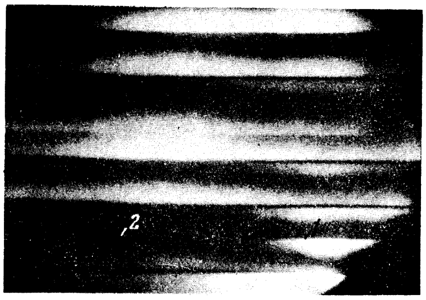


FIG. 2. Distortions that lead to the tapering of the trace of the elementary shear. Electron microscope, lacquer replica contrasted with chromium (× 25,000).

shear have a higher illumination on the surface of the specimen, corresponding to higher values of the stress remaining after removal of the load. It is in these places that the shears, which penetrate into the crystal where the stresses are considerably lower, originate. As the stress increases to $\sigma \approx 100$ g/mm², the elementary shear develops into a single slip band (Fig. 3).



—(110)

FIG. 3. Transformation of elementary shear traces, 2 into single slip bands, 1 (so called "pencil lines"), after the shear is increased. Crossed prisms (× 25).

If the lateral surfaces of the specimen are coated with warm water prior to loading, the elementary shears may occur inside the specimen in the form of short lines, which are brightest in the middle. The water dissolves and "cures" the surface imperfections. As a result, the weakest places are the imperfection regions inside the specimens. As the stress is increased, the trace of such a shear spreads also to the lateral faces of the specimen. Sometimes the traces of the shear pass through the vertices of the two-faced angles of the pores, which are bounded along the cleavage and serve to concentrate the stresses.

The elementary shears are sensitive to variable stresses. If a specimen with traces of elementary shears is loaded (10-15) times and the stress is raised to approximately 70 g/mm², slip bands are formed in the region of the elementary shears. In an investigation of reverse slip in rock salt[6] it was noted that certain slip bands were partially shortened after unloading. This phenomenon, similar to elastic twinning, was observed in calcite, sodium saltpeter, and antimony; it was also observed during slip in elementary shear production. To observe this phenomenon it is necessary to employ a rigid loading device to produce the strain at a rate up to 0.5% per minute, and to remove the load immediately after loading. The vertical shear traces of Fig. 4 are shortened after unloading, while the horizontal traces can serve as reference lines.

The crystallographic orientation of the elementary shears is determined not only by the fact that

FIG. 4. Shortening of two longitudinally incomplete traces after unloading and formation of tapered elementary shear traces. Crossed prisms (×25).

the maximum tangential stress occurs in the {(110)} planes in compression along the edge of the cube. In specimens variously oriented with respect to the crystallographic axes, the elementary shear traces are always located in the planes of the rhombic dodecahedron {(110)}. If the specimen axis is oriented along the [110] direction then the greatest shear stresses are in the (100) plane and are directed along the edge of the cube. However, in this case too, the elementary shears occur in the plane of the rhombic dodecahedron, the normal to which $[0\bar{1}\bar{1}]$ makes an angle of 60° with the axis of the compression forces, and the shear stress is one-half or one-third the shear stress in the (010) plane along the [100] direction. As can be seen, the law of tangential stresses is obeyed also in the formation of elementary shears.

The difference between the elementary shear stage and the later stages of the deformation is also disclosed by an investigation of the residual stresses during the annealing process. Samples of rock salt were deformed until elementary shears ($\sigma_0 \approx$ 70 g/mm^2) were formed or until slip bands and packets of slip bands were formed ($\sigma_0 \approx$ 120, 250, and 500 g/mm^2), and were then annealed at 350—750°C. The time interval τ needed to eliminate the residual birefringence in the specimen was determined during the annealing. The experimental data were used to plot curves of ln τ (1/T) (Fig. 5). The linearity of these curves is the reason for believing that the removal of the distortions that remain after deformation is an activation process.

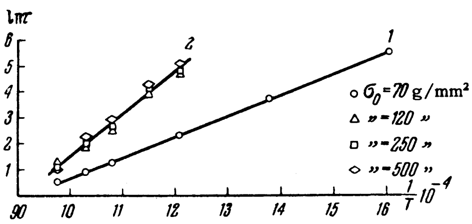


FIG. 5. Logarithm of the relaxation time of the residual stresses as a function of the reciprocal of the absolute annealing temperature and of the maximum stress during deformation, σ_0. 1) elementary shears, 2) later stages of deformation.

All the experimental points of Fig. 5 fall on two straight lines with different slopes. The points on line 1 correspond to annealing of crystals with elementary shear traces, while those on line 2 pertain to subsequent stages of deformation. If the activation energy of strain relief is determined from the slope of the ln τ (1/T) lines, we obtain for line 1, corresponding to elementary shears, E_1 = 14 kcal/g-mole, and for line 2, pertaining to slip bands and packets of slip bands, E_2 = 30 kcal/g-mole. The obtained results lead us to believe that to remove the residual stresses in the former case it is enough to have a diffusion displacement of the molecules in the slip plane, while in the latter case volume migration is essential.

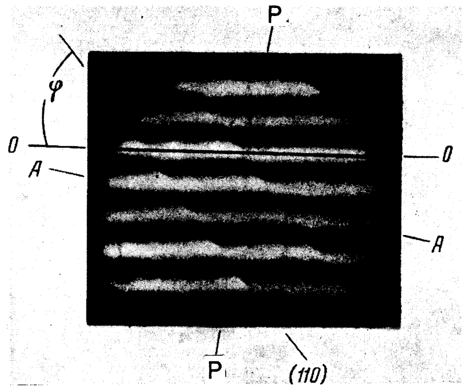


FIG. 6. Curving of compensator interference fringes on the elementary shear traces. The residual stresses on both sides of the elementary shear traces are of equal magnitude and opposite sign. Crossed polarization prisms. A quartz wedge — compensator — is installed in the focal plane of the eyepiece. A—A) analyzer, P—P) polarizer, O—O) hairline on the compensator.

Figure 6 is a photograph of a specimen with two elementary shears taken in transmitted monochromatic light under crossed polarization prisms and a compensator in the form of a quartz wedge, located between the prisms and made coincident with the image of the crystal. The interference fringes are displaced symmetrically from the normal position, which has been fixed with the aid of the hairline O—O. Consequently, on each elementary shear trace the residual stresses are approximately equal in absolute magnitude and opposite in sign. The normal stresses are determined in this case from the known formula[10]

$$\sigma = a \sin \varphi / ALd, \qquad (1)$$

where A is the distance between neighboring interference fringes, a the displacement of the interference fringe in the stressed portion, φ the angle between the shear trace and the edge of the quartz wedge, L the corresponding piezo-optical constant of rock salt, and d the thickness of the specimen along the beam. The residual stresses calculated from (1) are approximately equal to the average stresses in the specimen during the production of elementary shears ($\sigma \approx 66$ g/mm^2).

On the whole, the picture agrees with the scheme proposed by Obreimov and Shubnikov.[7] However, if the density of the image of a crystal with elementary shears, obtained under crossed polarization prisms, is measured along a straight line perpendicular to the shear traces, a clearly asymmetrical curve is obtained. The latter is in clear contradiction with the conclusions made on the basis of Fig. 6, where it is assumed that the illumination in Fig. 1 is determined only by birefringence due to residual stresses. This contradiction appears also in observing the variation in the illumination on both sides of the shear trace upon rotation of the crystal relative to the beam. By placing the crystal between crossed polarization prisms, it is easy to verify that the illumination on either side of the elementary shear trace changes from maximum to minimum as the table with the crystal is rotated 90° relative to the beam. The initial distribution of the illumination is restored only when the specimen is rotated 180°. Were the illumination produced only by birefringence, it would be restored in crossed prisms by rotating the table 90°, i.e., four times per revolution. The contradiction can be resolved by assuming that the birefringence is accompanied by additional scattering, reflection, or absorption, leading to the attenuation of the light of definite polarization at definite crystal positions. Such an effect can be expected from a system of scratches, cracks, or groups of dislocations. It must be assumed that these imperfections are differently oriented on the two sides of the shear trace, since the minimum illumination on one side of the trace corresponds to the maximum illumination on the other side. The orientations of the imperfections may, for example, coincide with the planes of the cube.

An investigation of the illumination near the trace upon rotation of the crystal has in fact disclosed additional attenuation of the light, possibly as a result of diffraction by the system of scratched irregularities of the type indicated above.

In a previously published paper by one of the authors and Kovalev,[8] it was shown that electron-microscope photographs of deformed rock salt show systems of cracks oriented along the cube planes in the slip-band packets.

Interesting information on deformation can be obtained by studying the shape of the lateral surface of the specimen at the place where the elementary shears emerge. A sample of rock salt was deformed until elementary shears were formed, and a collodion replica of the cleavage plane parallel to the lateral surface was made and contrasted by a molecular beam of chromium, directed at a small angle to the surface. The replica was then viewed in an electron microscope (Fig. 7). The density of

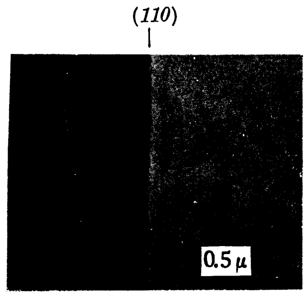


FIG. 7. Emergence of elementary shear trace on the (100) plane. Lacquer replica contrasted with chromium, at an angle of 12° to the direction (010). (×25,000)

the negative of the electron-microscope print was measured along a line perpendicular to the slip trace. Using the microphotogram and knowing the contrasting condition, it is possible to plot the profile of the lateral surface of the specimen by the procedure described in reference 9. The height of the profile in section x is

$$h_x = \cot\theta \left[\int_0^x \frac{\ln (D_0 / D_x)}{\ln (D_0 / D_h)} \, dx - x \right], \qquad (2)$$

where θ is the angle between the contrasting direction and the normal to the surface of the replica ($0 < \theta < 90°$), D_0 the density of the electron-diffraction negative in the portions of the print not covered by chromium, D_x the density in section x, D_h the density in the horizontal sections located far away from the curved portion of the surface of the replica. In the derivation of Eq. (2) it was assumed that the distance from the source of the contrasting molecular beam to the print is much greater than the dimensions of the print, and that absorption and scattering of the electrons occurs only in the chromium contrasting layer.

The profile of the lateral surface of the specimen (Fig. 8) has a smooth shape at the place of

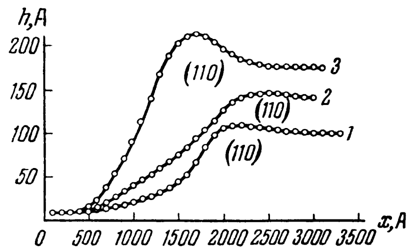


FIG. 8. Relief of the cleavage surface along the (100) plane of crystals with elementary shear traces: 1) $\sigma = 66$, 2) $\sigma = 80$, 3) $\sigma = 95$ g/mm^2.

emergence of the elementary shear. The lateral surface is concave on one side of the shear trace and convex on the other. The small steps, usually employed to represent schematically the emergence of dislocations and similar phenomena in describing pure shear, are obtained at later stages of the deformation.

The shape of the obtained profile makes it possible to state that the shear has caused one portion of the crystal to become displaced relative to the other. The major part of the step is smoothed by the concave part of the profile. Adjacent to this section is a convex bead, the height of which is approximately one-tenth that of the step. The causes of the appearance of such a bead are still not clear. It is possible that the residual stresses cause elastic bulging of the region of the crystal adjacent to the shear trace. This recalls to some extent the elastic deformation of the surface near a forming elastic twin, as described in reference 1. Such a formation was recently called the accommodation region.[10]

If the smoothness of the concave portion of the profile is due to surface-tension forces, stresses τ should occur inside the deformed region ABC (Fig. 9) and in its direct vicinity. The average density of elastic energy is

$$\bar{E} = \bar{\tau}^2 / 2G, \qquad (3)$$

where G is the shear modulus. The condition of stability of the profile can be written by equating the elastic energy in the deformed volume ABC to the surface energy difference connected with the change in the area of the lateral surface in the region of the shear (see Fig. 9). We have

$$\alpha_{(100)} \overset{\frown}{AB} + \bar{E} S_{ABC} \geqslant \alpha_{(100)} \overline{AC} + \alpha_{(110)} \overline{BC}. \qquad (4)$$

(S_{ABC} is the area of the triangle ABC). Inasmuch as the curvature of arc AB is small, we can put

$$\overset{\frown}{AB} = \overline{AB}. \qquad (5)$$

The tangent of γ — the slope of the surface for the elementary shears—ranges from 0.03 to 0.12, while the height h of the profile is from 10^{-6} to 2.5×10^{-6} cm. Inserting these values into (4), (3), and (5), we determine the magnitude of the critical tangential stresses under which the profile still remains smooth

$$\bar{\tau}_c \geqslant \left[\frac{4G\,(\alpha_{(100)} + \sqrt{2}\,\alpha_{(110)})\,\gamma}{(1+\gamma)\,h} \right]^{1/2}. \qquad (6)$$

Putting $\alpha_{(100)} = 500$ erg/cm^2, $\alpha_{(110)} = 1250$ erg/cm^2, and G = 1.26×10^{11} dyne/cm^2 we obtain an average value $\bar{\tau}_c \gtrsim 10^{10}$ dyne/cm^2 for h = 250 A and $\gamma = 0.12$.

Equating the theoretical strength of the crystal with the local stresses at the emergence of the elementary shear, as calculated by Eq. (6), we note that in practice the profile remains curved until the crystal fails in the region of emergence of the shear.

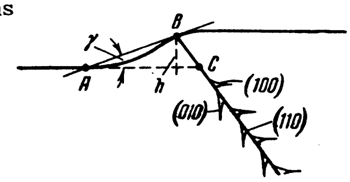


FIG. 9. Arrangement of elementary shear trace of distortions on the cleavage surface (100), and of the systems of scratches which possibly lead to the observed attenuation of light on one side of the trace under crossed prisms of a polarization microscope.

Observations in polarized light show that the trace is much brighter at the place of emergence on the lateral surface. This can be attributed to the stresses due to curving of the surface. If the radius of curvature of the convex portion of the profile is denoted by R, the magnitude of the stresses, $\tau = \alpha/R = 6.2 \times 10^6$ dyne/cm^2, is indeed approximately equal to the residual stresses near the elementary shear trace. It is seen from this that the increased brightness can be explained by superposition of stresses due to curvature of the surface on the residual stresses that arise during the formation of the elementary shear.

The resolving power of the method used here is different along the x axis and the h axis (see Fig. 8). It amounts to 100 A along the x axis, and therefore one of the results is the conclusion that in the elementary shear the plastic deformation is localized in a layer not more than 10^{-6} cm thick parallel to the shear trace.

In conclusion the authors express their gratitude to V. K. Sklyarov and I. M. Fishman for their help in performing these experiments.

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