CHAPTER VI

FRACTURE IN SODIUM CHLORIDE

The previous chapters have been mainly concerned with the studies of various aspects of dislocations in sodium chloride. To prepare the specimens from the as grown single crystal, one has to cleave it. It is well known that sodium chloride cleaves along the \{100\} planes when a small force is applied with a cleavage knife along the [100] direction. Contrary to the expectations, the cleaved surfaces are not found to be perfectly smooth. Instead, they contain cleavage steps which are popularly known as river patterns or tear marks. The density of the lines in the river pattern is known to vary with the mode of cleavage and the perfection of the crystal. One explanation for the observation of these furrows on the cleaved surfaces is the presence of screw dislocations in the crystal. According to this hypothesis put forward by Gilman (1956), the steps in the cleavage surface of a crystal are formed by encounter with a screw dislocation, the Burgers vector of which does not lie in the cleavage plane. Recently, Prinkel’ et al (1964) have reported that this mechanism was either absent or not predominant in their studies of crack propagation in ionic crystals. A study has been made here to throw
Fig. 1. Cleavage steps on sodium chloride single crystal. (x150)

Fig. 2. Interference pattern on a river pattern. (x150)
more light on these river patterns with special reference to plastic deformation present at the tip of a crack and its effect on the unevenness of the cleaved surface.

Another aspect that has been studied in the present investigations on cleavage of deformed sodium chloride single crystals is the propagation of cracks in the planes other than \{100\}. (110) crack and its possible origin have been discussed.

Results and Discussion

It is quite usual to observe the cleavage steps on the cleaved surface of single crystals as is shown in figure 1 for sodium chloride crystal. Figure 2 is the interference pattern of the river pattern. It is quite obvious from the contour of the fringes that the lines making up the river patterns represent a level difference in the cleavage surface which results because the cleavage crack is not following a single crystallographic plane. A crack that may start as an ideal single crack, after some growth, breaks up into a set of parallel cracks propagating simultaneously and joining along lines when these overlap, either by secondary cleavage or by shearing of a thin strip of the crystal. The cleavage steps so produced require additional energy for their formation and thereby
Lmt (1953) has given a number of causes that may be responsible for the nucleation of these cleavage steps. The most common of these is the presence of screw dislocations that intersect the cleavage plane (Gilman, 1955, 1958). When the cleavage crack cuts a screw dislocation, the level of the crack on either side of the dislocation is shifted with respect to each other and the cleavage crack after crossing the screw dislocation, moves on two different levels which are joined by a step which is of the order of the Burgers vector of the screw dislocation. Such cleavage steps propagate together and are capable of annihilating each other if they are of opposite sign or producing a larger step if they are of the same sign.

The screw dislocations responsible for the cleavage steps can be present in the crystal as randomly distributed grown in dislocations or in the form of regular arrays in a twist boundary. These can also be introduced in the crystal by plastic deformation before the cleavage occurs or by glide at the tip of the cleavage surface. It has been observed in the present studies that the density of the cleavage steps is much higher in an
Fig. 6. Multiple beam interference fringes across a high density of cleavage steps. (x120)
initially deformed crystal. This is made clear in figure 3. This observation supports the view that the cleavage steps are produced in the sodium chloride by the presence of screw dislocations. As can be seen in figure the tilt boundary, consisting of edge dislocations, only changes the direction of propagation of these steps.

Cleavage Surface and Crack Velocity

Finkeln et al (1964) have recently reported that screw dislocation hypothesis may not be applicable to ionic crystals. According to these authors as the velocity of propagation of the crack increased, the structure of the surface on the average, became more complicated. In conformity with the earlier observations, it was observed by them that with increase in velocity of crack, the ability of the latter to generate dislocations in front of itself decreases. This decrease is attributed to the time required for nucleation of a dislocation loop or the release of dislocation sources locked by impurities. Gilman et al (1968) have established the existence of a threshold velocity, above which the crack did not create dislocations at its tip. Campbell et al (1961) showed that a time of the order of $10^{-9}$ sec. was
process being a fluctuation one, requires a definite time. With increase in velocity of crack, the time of action on the source diminishes, resulting in a decrease in plastic deformation close to the crack and ultimately in the fracture cavities. This interpretation is capable of explaining the existence of plastic deformation even at very high velocity of the crack, in insufficiently pure, commercial, previously deformed or polycrystalline material. If there were no sources close to the crack, the fluctuation mechanism would not be operative and there would be a critical speed, above which plastic deformation would be absent.

Contrary to the observation of Frinkel et al., it has been observed in the present case that a higher crack velocity results in a smoother surface and this supports the screw dislocation hypothesis. Let us consider the propagation of Wallner waves along fracture surfaces. These were discussed and studied
by Saekal (1940), Wallner (1939) and Kerkhof (1963). These waves are formed at the front of a crack in the case of fracture of regions having a disturbed structure. Stress concentration is particularly high near such regions, and on fracture, they emit high frequency waves in the ultrasonic or hypersonic frequency range. According to the view of Pringle (1908), it must be admitted that Wallner waves are surface Rayleigh waves, produced in the cavities of sufficiently rapid crack. When such waves intersect the front of the crack, the latter is deflected and forms furrows or steps on the fracture surface. A family of such steps can be used to determine the speed of propagation of a brittle fracture (Saekal, 1959). A similar relief has also been produced artificially by introducing into the fractured body ultrasonic vibrations from an external source (Kerkhof, 1953, Schardin 1959). The frequency of the Rayleigh surface pulses ought to increase with increase in the velocity of crack, resulting in an increase in the uneveness of the surface.

There is sufficient evidence to show that Wallner waves are present in certain crystals. However, in the case of sodium chloride these were not observed as an increase in the crack velocity did not produce more uneveness of the surface. Indeed reverse was the case. Further more, interference pattern of a surface due to
Fig. 4. Birefringence pattern on a $(100)$ crack that has been made to stop. (x125)
Wallner waves and the lines themselves show that Wallner waves are distributed in a regular fashion. Interference pattern on sodium chloride river pattern does not show this. It appears that screw dislocation mechanism is operative in case of sodium chloride crystals.

**Glide at the Crack Tip**

It has been mentioned above that the screw dislocations capable of producing cleavage steps may be introduced into a crystal by the glide at the tip of the cleavage crack. That the glide is produced at the tip of the crack has been observed in the case of sodium chloride using stress birefringence technique. Figure 4 shows the birefringence pattern on a (100) crack that has been allowed to slow down and stop. New glide bands can be seen starting from the tip of the crack. This clearly illustrates that the glide is caused by the tip of a moving crack (also see figure 11). The necessary condition is that the velocity of the crack must be low enough so that the time of loading of a unit volume at the tip of a moving crack is long enough for dislocations to move and multiply. On this basis it is expected that at lower crack velocity, as it would produce more screw dislocations, should result in
Fig. 5. *X-ray* scatter pattern on the two cleavage surfaces of a pre-stressed crystal.
Fig. 7. Birefringence pattern after 20 minutes of crystal at 30°C. (x100)

Fig. 8. Birefringence pattern after 40 minutes of crystal. (x100)
larger number of cleavage steps and this has been confirmed to be true in the present studies.

To test the screw dislocation hypothesis still further, the river patterns produced by cleavage in as grown and pre-stressed crystals were studied. The deformation before cleavage was also obtained by dull cleavage. Figure 3 represents a highly furrowed surface that has been obtained on a crystal that was stressed before cleavage occurred. The vertical line on the right hand side of the figure, where the cleavage steps have changed their direction without undergoing a change in density is a single tilt boundary. The idea of the step heights of the cleavage steps can be had from the contour of the multiple beam interference fringes which are crossing the steps. The various parts of the crack lie in different planes and it appears that in the middle portion at least plastic shearing of the material has taken place when the various parts of the cracks joined together. This has resulted in further glide as is obvious from the birefringence pattern (figures 5 & 6) obtained on the river pattern. However, the plastic deformation limits itself mainly near the surface as on annealing the birefringence bands disappear. Figure 7 shows birefringence pattern after 20 minutes anneal at 600°C and figure 8 after 40 minutes of anneal. The bands vanished completely.
when the crystals were annealed for one hour. Incidentally, this observation stresses the importance of a sharp cleavage knife to avoid plastic deformation and a furrowed surface which are produced by dull cleavage.

It has thus been seen that the cleavage steps result as the propagating crack intersects screw dislocations. A simple tilt boundary results only in the change in direction of the cleavage steps. There is definite evidence of the presence of glide at the tip of a slowly moving crack. Further, dull cleavage results in highly furrowed surfaces and this introduces plastic deformation in the cleaved surfaces. Wallner waves are not observed to be effective in sodium chloride cleavage.

Non-Cleavage Plane Fracture

So far we have restricted our discussion to the usual (100) cracks in sodium chloride crystals. It is well known that single crystals cleave on definite crystallographic planes which can be predicted. Various criteria have been suggested for their prediction. Hauy suggested that the unit cells of crystals are bounded by cleavage planes; this fails for fluorite. Tutton's suggestion that cleavage planes are most closely packed planes (Wooster, 1932) does not hold for many crystals such as zinc blende, fluorite and calcite. Huggins (1923)
suggested that the cleavage plane is the plane that
cuts a minimum number of chemical bonds per unit area. This criterion suffers because of rather vague meaning
of the "chemical bond" especially in the case of ionic
crystals. The best criterion that has been put so far
appears to be that the plane of minimum surface energy
should be the cleavage plane. This may not apply for
b.c.c. crystals like iron and tungsten.

In the cleavage of an ideal crystal the energy
required to cleave is twice the surface energy of the
resulting cleaved surfaces. For this reason almost
all the crystals cleave on lowest surface energy planes.
Crystals not cleaving on minimum surface energy planes
are rather unusual and their behaviour requires
explanation. The fracture of sodium chloride along
non-cleavage planes has been studied here.

Sodium chloride has (100) as the normal fracture
plane. It has, however, been observed that rock salt
type crystals sometimes fracture parallel to (110)
planes. Stokes et al. (1958, 1959 & 1961) and Koh (1959
& 1960) have observed (110) fracture in case of MgO.
They have attributed these cracks to the piling up of
dislocations when two (110) slip bands meet. Gilman
(1961) has reported (110) cracks associated with (100)
main crack in pre-strained lithium fluoride crystals.
As he did not observe any evidence of significant dislocation motion or any obstacle against which dislocations might pile up, he attributed, in view of the fact that according to the ionic crystal theory \{110\} planes have a surface energy about 2.5 times greater than \{100\} planes, these cracks to a lowering of the cohesive energy of (110) surfaces by plastic deformation. Wiederhorn (1962), elaborating on Gilman's idea, calculated the elastic energy released by a crack propagating parallel to a slip band and found that the elastic energy released could be large enough to explain (110) crack propagation in LiF. Wiederhorn (1962) described a second mechanism of (110) crack formation which involves the elastic interaction between a slip band and a moving (100) crack. He contended that a crack propagating on a cleavage plane experiences a repulsive force from a slip band and that this force may be large enough to cause the crack propagation on a (110) slip plane.

Recently Wiederhorn (1963) has reported that (110) fracture observed in all the cases runs parallel to birefringence band. He has suggested that the stresses associated with these bands are instrumental in the formation of (110) cracks. The elastic stress of the birefringence bands may either aid or hinder the crack propagation. Compressive stress may hinder crack
11. 0. ... crack propagation along its bifurcation front. (x12.)
propagation, since extra energy is required to expand the crack walls against the stress, whereas tensile stress aids crack propagation if the elastic stress is to relax on the propagation of the crack parallel to the birefringence band as in this case the elastic energy would be released and it would reduce the energy required for fracture. Walderhorn has suggested that both these mechanisms might be operative to cause (110) fracture in lithium fluoride.

Earlier Kuznetsov (1957) has cast doubts as to if the observed cracks are actually (110) cracks. While discussing the (110) fracture in rock salt, he has noted that on the considerations of energy (110) cracks may be stepped (100) cracks and not as truly flat (110) cracks. He has calculated that 1.75 times as much energy is required for a crack to form a flat (110) surface as is required to form a stepped (100) surface. In view of the controversy regarding the origin of (110) cracks, efforts were made to study the non-cleavage plane fracture in sodium chloride single crystals.

It has been observed that normally cleavage occurs along (100) planes but the fracture has also been observed in planes other than (100), usually in pre-strained crystals. Figure 9, shows a crack propagating along its birefringence band.
Fig. 10. A crack deviating from its birthingence
band. (x12)
Figures 10 & 11 are very unusual. It is clear from the birefringence bands that the (110) crack is not running parallel to the bands but instead is inclined to these. To the author's knowledge, such an observation has not been reported earlier and is very significant for throwing more light on the unusual cracks observed so far. The mechanisms put forward by Gilman (1961) and by Wiederhorn (1962 & 1963) are incapable of explaining the presence of this crack which is inclined to (110) plane. Their explanation in fact was to explain the (110) cracks observed in lithium fluoride.

In the opinion of the author such a crack can be explained as being composed of stepped (100) cracks. From energy considerations, one would expect this to be consisting of (100) surfaces as the surface energy of (100) surface is minimum. Indeed if Kuznetsov (1958) explanation of (110) cracks as stepped (100) cracks is to be accepted, one would expect the cracks to form in planes inclined to (110) planes as well. This is supported by the observation of the crack of figure 11. Figure 11 is most interesting from this point of view. The crack is clearly seen to be of stepped nature and the steps are quite favourably inclined to (100) directions.

The present observations, though may be insufficient to rule out the mechanism put forward by Gilman and Wiederhorn, provide sufficient evidence to believe that
the non-cleavage plane cracks observed in sodium chloride may in reality consist of (100) stepped surfaces. Repulsive forces encountered by a (100) propagating crack in a pre-strained crystal may cause the additional (100) stepped surfaces to give a general appearance to the crack of a (110) crack or of a crack which is inclined to the (110) planes.

Summary

A number of sodium chloride single crystals have been cleaved by sharp as well as dull cleavage and a study of the cracks and the resulting cleavage surfaces has been made. The observations support the Gilman's hypothesis of screw dislocations for the formation of cleavage steps and a higher crack velocity is seen to result in greater evenness of the surface. Birefringence patterns of the cracks that have been made to stop in the crystal, reveal the presence of new glide bands at the crack tips. Apart from the normal (100) fracture, unusual fracture in (110) as well as other random planes has been observed. New and interesting observations regarding these cracks have been made. The evidence obtained indicates that the non-cleavage plane fracture may indeed consist of stepped cleavage plane surfaces.
Bibliography

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