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Electron microscope study of surface markings left by unsteady cleavage crack propagation in NaCl single crystals

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ABSTRACT

Surface markings left by an unsteadily propagating cleavage crack in NaCl single crystals have been studied by electron microscopy as a function of accelerations and decelerations of the crack. As a result, a comprehensive description of the sequence of markings present between two successive stopped crack fronts has been obtained. Models are proposed illustrating the manner in which the activation of the longitudinal and transverse slip systems and their interaction can account for the cleavage markings observed.

§ 1. INTRODUCTION

Surface markings left in the path of a propagating crack have been used extensively to study the plastic deformation accompanying the passage of the crack. The understanding of such markings is one of the most direct ways of gaining insight into the processes occurring during cleavage or fracture. The detailed cleavage structure depends mainly on the velocity and local directions of the crack. The relationship has been established largely by optical microscopy, particularly in zones where cleavage cracks have come to rest. Examples of such studies are those of Gilman (1956, 1957), Forty (1957), Robins, Rhodin and Gerlach (1966), Burns and Webb (1966, 1970).

The sub-optical microscopic detail of the surface structure has been studied by electron microscopy usually making use of decoration replicas (Bassett 1958) which reveal surface structure as yet inaccessible by more direct methods, such as scanning electron microscopy. Although surface studies of various ionic crystals with the NaCl structure have been reported (Robins *et al.* 1966, Burns and Webb 1970), most of the research in this field has been performed on NaCl crystals. The literature contains no comprehensive description of the widely different surface markings left by a cleavage crack in NaCl crystals as a function of changes in the velocity and direction of propagation of the crack. The aim of the present work is to fill this gap by studying the changes in surface markings produced by the accelerations and decelerations of unsteady cleavage cracks.

For this purpose the surface regions between two successive stopped crack fronts have been studied by electron microscopy. The most appropriate replica technique is a ‘ shadow-decoration ’ method (Wainer 1974), which has the advantage of allowing the sign of steps to be determined (i.e. to distinguish ‘ up ’ steps from ‘ down ’ steps).

The fine detail of the surface structures at the sub-microscopic level has been correlated with the more macroscopic view of the surface as revealed by optical microscopy. It has been found that such an approach greatly assists the interpretation of the phenomena observed.

Although it is not possible at present to understand every feature revealed by the surface decoration technique, many of the surface markings have been related to the dislocation movements that give rise to the plastic deformation accompanying unsteady cleavage crack propagation.

§ 2. Experimental

The specimens were cleaved from large blocks of high-purity NaCl and LiF crystals (obtained from Hilger and Watts) to give prisms of approximately 15 mm long by 5 mm wide by 3 mm thick.

All the experimental operations were conducted in a dry-box in which the relative humidity was kept to between 5 and 10% by a flow of dry nitrogen. The dry-box included an optical microscope and the bell jar of a 12 in. coating unit. Glove ports were positioned to allow all operations, including cleavage, optical observations, transfer to a vacuum system for decoration-replica preparation, etching, etc., to be completed without exposure to moist air.

A cleavage crack was introduced into a specimen by inserting a single-edge razor blade with a light hammer. The propagation of the crack was then continued with a cleavage jig in which a wedge of approximately 17° angle is slowly inserted into the opening of the crack through a rod connected to a screw driven manually. The propagation of the crack was stopped several times and the instantaneous positions of each stopped crack front were recorded with a photographic camera attached to an optical microscope. When cleavage was completed one-half of the specimen was transferred to the bell jar of the coating unit where a decorated replica of the surface was prepared by evaporating gold and carbon onto it successively.

To ensure that an area of the crystal, previously selected by optical microscopy, appears in an open and identifiable grid square, a 400 mesh ‘ finder pattern ’ electron microscope grid was attached with a light adhesive to the top of the carbon film under the optical microscope. Low-magnification optical micrographs of the replica plus grid were taken ; these always included an edge of the crystal in order to determine the relationship between the main crystallographic directions, the crack propagation direction and the grid bars.

Shadow replicas of the other half of the cleaved specimen after chemical etching were used to correlate the dislocation distribution at the crystal surface with the cleavage-step structures observed in the decorated and shadow-decorated replicas. The crystals were etched in the dry-box with a saturated solution of anhydrous ferric chloride in glacial acetic acid (Wainer and Miguez 1978).

The replicas were examined in a Philips EM 300 electron microscope.

§ 3. Results

Unsteady cleavage crack propagation involves the crack running freely, slowing down, stopping, retreating and healing slightly, then restarting and repeating the entire sequence several times until the crystal is finally cut through. Regions of the cleavage surface between two stopped crack fronts were observed in the electron microscope. There are five different zones to be considered : (1) V or ' lightning ' zone ; (2) transition zone, from V-pattern to tartan pattern ; (3) ' tartan ' pattern zone ; (4) ' stop-band ' zone ; and (5)

Fig. 1

Schematic tracing of the main characteristic features observed between two stopped crack fronts : (1) V or lightning-pattern zone ; (2) transition zone from V pattern to tartan pattern ; (3) tartan-pattern zone ; (4) stop-band zone ; (5) restarting zone with inclined steps.

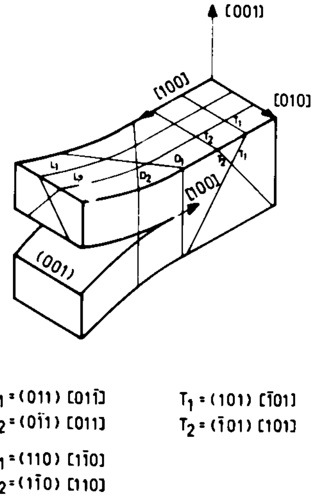
restarting zone with inclined steps. These five zones appear always in the same sequence and are illustrated in fig. 1. This figure is from a mosaic of electron micrographs presented as a tracing, in order to emphasize the main features and because a large area of decoration replica does not reproduce satisfactorily as a half-tone plate. Three of these zones, the tartan pattern the stop band and the restarting zones are shown in fig. 2.

Fig. 2

Composite electron micrograph of a decorated replica of a NaCl crystal showing tartan-pattern, stop-band and restarting zones.

The analysis of these zones will begin with a brief mention of the already well known lightning pattern and the transition zone and this will be followed by a detailed analysis of the other three zones, which have not been previously described. Figure 3 shows the cleavage plane, crack directions and primary slip systems referred to in this analysis.

Fig. 3



Geometry of slip planes and crack propagation direction. L_1 and L_2 are longitudinal slip systems, T_1 and T_2 are transverse slip systems, D_1 and D_2 are diagonal slip systems.

3.1. *V or lightning pattern zone*

The lightning pattern is composed of steps which form well-developed V s and is always present when a cleavage crack is running freely at high velocities in a direction that is not parallel to the [100] direction.

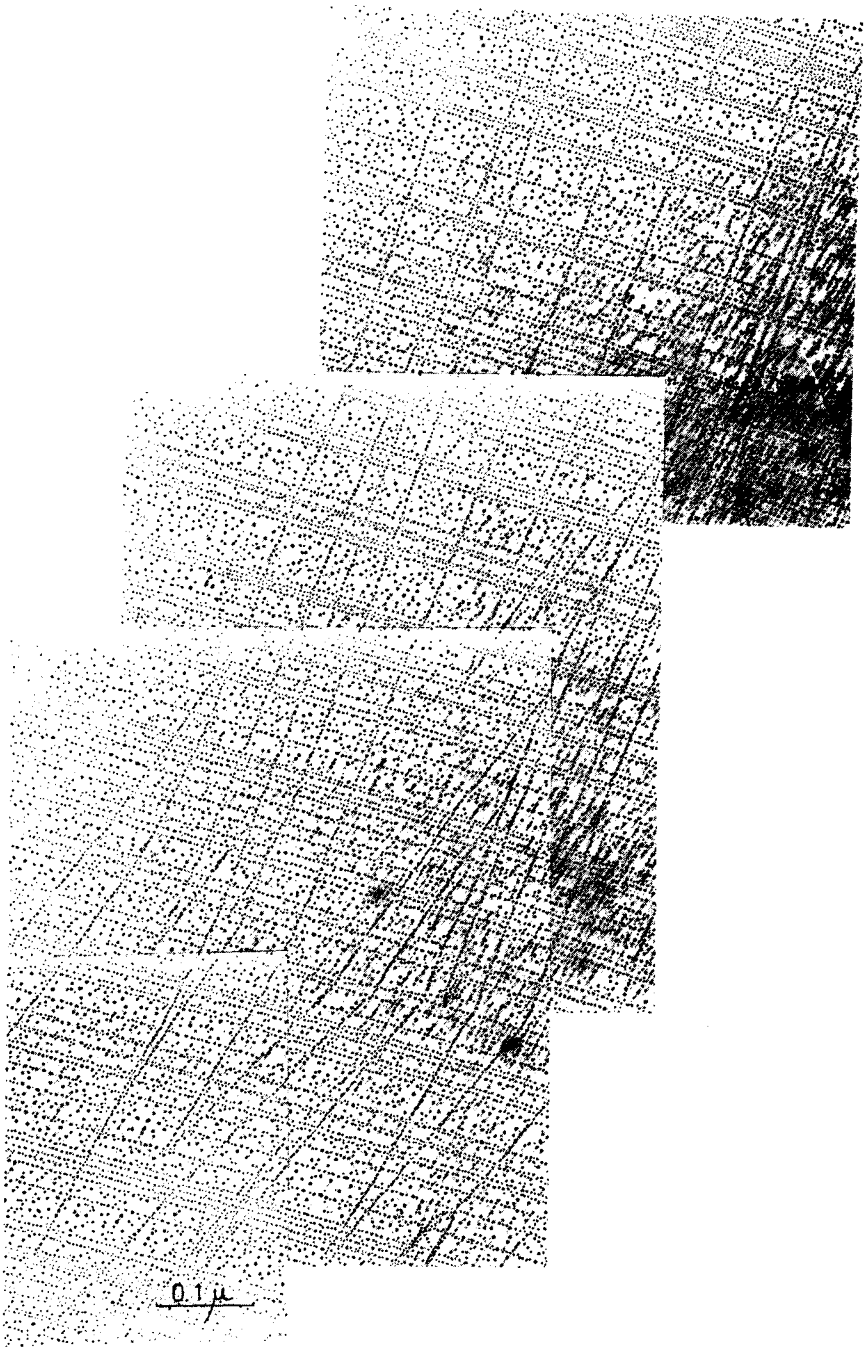
Different mechanisms have been proposed by Bethge (1962), Forty and Forwood (1963), Forwood (1966), Robins *et al.* (1966), Burns and Webb (1970) Levi (1973) to explain the formation of this type of structure. Most of these consider that the V s are formed by the interaction of a cleavage step and a slip step. This accounts for V s in which one arm is parallel to the [100] direction. But for V s in which both arms are curved or follow irrational directions it implies that a large number of dislocations are repeatedly cross-slipping to follow nearly parallel irrational paths. It is difficult to accept the occurrence of such a cooperative effect since, to our knowledge, there is no other situation where a large number of neighbouring individual dislocations cross-slip along parallel curved or irrational paths in this manner. No alternative mechanism can be proposed at present for the formation of this commonly occurring pattern, but it is clear that the mechanisms proposed so far do not explain all the types of patterns observed in practice.

In this zone the relatively high steps present on the surface are inclined with respect to the [100] direction as a consequence of the local direction of crack propagation. This results from a bowing of the crack front which is normally observed when the crack is restarted, as described by Gilman (1957).

3.2. *Transition zone from V-pattern to tartan pattern*

When the crack begins to slow down, transverse slip lines appear and the atomic steps forming the V s tend to align parallel to the crystallographically

Fig. 4

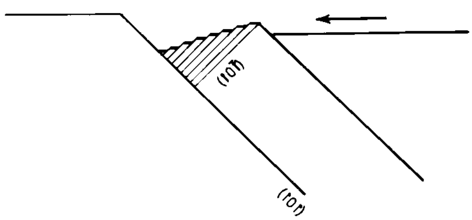


Transition zone from V-pattern to tartan pattern. Standard decorated replica of a NaCl crystal.

preferred [100] direction ; the resultant surface markings are arranged in a manner resembling a tartan pattern illustrated in the extreme upper section of fig. 4, and more distinctly in fig. 5 (*a*).

Fig. 5

(*a*)



(*b*)

(*a*) Electron micrograph of a shadow-decorated replica of a NaCl crystal. Shadowing direction perpendicular to the transverse lines. (*b*) Model proposed to explain formation of transverse band AA.

3.3. *Tartan pattern*

The tartan pattern zone is associated with deceleration of the cleavage crack. When the crack velocity decreases, plastic relaxation of the stresses concentrated at the tip of the crack takes place through the nucleation and multiplication of dislocations of the transverse and longitudinal slip systems. It is worth emphasizing that, under the chosen experimental conditions, the observed cleavage markings are the final result of the deformation processes involved in the deceleration of the cleavage crack and those related to the wedging open of the crystal arms. In effect, each time the crack is restarted, additional plastic deformation occurs in the already deformed part of the crystal, leading to an enhancement of the existing slip bands.

Throughout the tartan-pattern zone, atomic cleavage steps run parallel to the crystallographically preferred [100] direction. Under these conditions it is not possible to distinguish between the elementary cleavage steps and the longitudinal slip lines appearing in this region. No displacement is observed at the intersection of the two sets of perpendicular lines although displacement of the transverse slip lines can be seen at their intersection with high cleavage steps.

The length of this tartan pattern zone depends on the spacing between two consecutive stop bands ; it extends over one-quarter to one-half of the distance between these bands and zone lengths from 50 to 500 μ have been observed.

In order to gain a better insight into the relationship between these surface markings and the activated slip planes, specimens were shadow-decorated (Wainer 1974) in different directions. This technique distinguishes positive, or up-steps, which are characterized by the condensation of larger gold nuclei, from negative or down-steps, which are rendered visible by the condensation of smaller nuclei.

3.3.1. *Analysis of cleavage structures produced by the activation of the transverse slip systems*

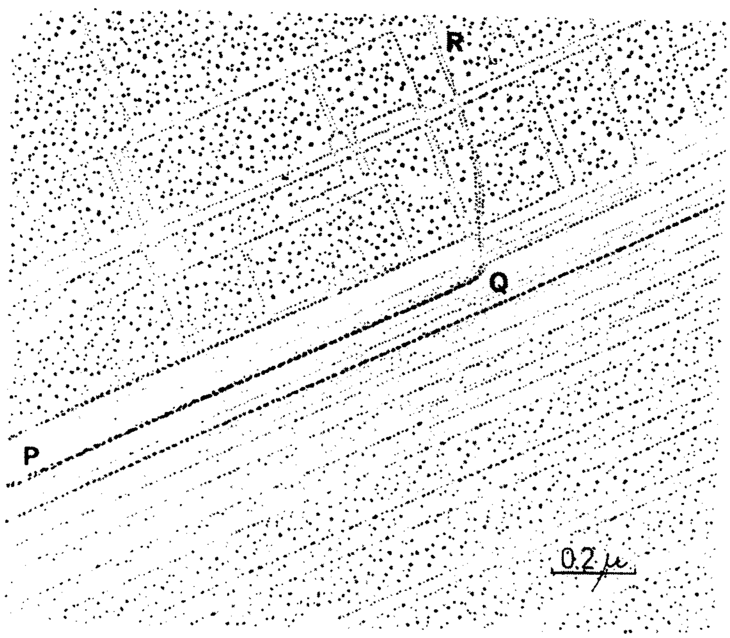
It has been shown from X-ray topography studies (Wainer and Manghi 1977) that numerous dislocation loops are present in the (101) and ($\bar{1}$01) planes as a consequence of the preferential activation of the transverse slip systems ahead of a decelerating crack tip. Transverse slip lines are frequently grouped in bands of variable width. This provides a verification that the most active mechanism for the multiplication of dislocations introduced by cleavage is that proposed by Koehler (1952), and Johnston and Gilman (1960). Further plastic deformation resulting from the wedging open of the crystal arms contributes also to these transverse slip bands.

Both transverse slip systems are activated by a decelerating crack. It is to be expected from the anisotropy of the stress field of the propagating crack that one of them should appear more frequently and this has been confirmed by the presence of a larger number of slip lines belonging to the (101)[$\bar{1}$01] system.

Frequently, narrow transverse slip bands appear as closely spaced groups of down-steps bounded by two up-steps (fig. 5 (*a*)). This distribution can be understood in terms of the preferential activation of the primary (101)[$\bar{1}$01] slip system. Dislocations of the conjugate system produced at or near the surface of the crystal are held up when they impinge on the slip planes of the primary system, and cross-slip and multiply in the narrow zone between these two primary planes (fig. 5 (*b*)). This behaviour is similar to that described by T. Suzuki (cited by Nabarro 1967) for KCl single crystals deformed in compression.

Not all the transverse slip lines are of monoatomic height ; some of them are several atomic units high, and some change their height discontinuously over short distances. Some steps like PQR (fig. 6) follow the general direction of crack propagation until they develop a jog in the [010] direction at a particular step of the band; the cleavage step resumes the general direction of crack propagation after developing another jog; distances between jogs from 0·5 to 5 μ have been observed. It is believed that this remarkable sudden change in the height of some transverse lines and the jogged path of some cleavage steps when traversing the bands can be accounted for by the presence of small (100) cracks nucleated at the intersection of the orthogonal transverse slip systems. A similar mechanism has been proposed for MgO crystals by Argon and Orowan (1964).

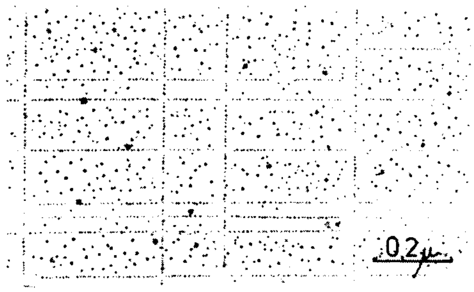
Fig. 6



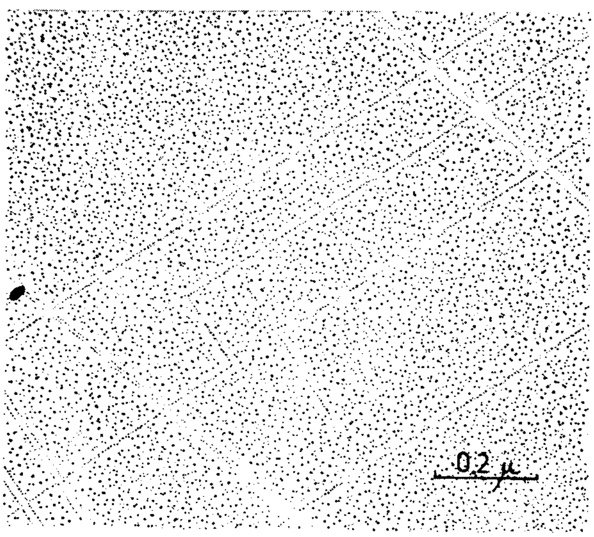
Interaction of a small cleavage step with a dense transverse slip band. Shadow-decorated replica.

Most of the cross-slip structures which are frequently observed in this zone (fig. 7 (*a*)) are obviously related to mobile dislocations. This shows that the statement by Appel and Messerschmidt (1969), that cross-slip of individual dislocations occurs mostly when they are immobile, is true only for surface-induced cross-slip (fig. 7 (*b*)). In order to ascertain whether some of the cross-slip structures were an artifact arising from heating of the specimen during the standard decoration process, a comparison was made between standard decorated and shadow-decorated replicas. It showed that non-crystallographic cross-slip of individual dislocations, though frequently observed in standard decorated replicas, is rarely found in shadow-decorated replicas prepared at room temperature. It is evident from these results that cross-slip structures introduced by cleavage are best studied in shadow-decorated replicas.

Fig. 7



(*a*)



(*b*)

(*a*) Electron micrograph of standard decorated replica of a NaCl crystal showing cross-slip structures. (*b*) Electron micrograph of shadow-decorated replica of a NaCl crystal showing cross-slip structures.

3.3.2. *Analysis of the cleavage structures produced by the activation of the longitudinal slip systems*

It is assumed that most of the isolated longitudinal lines observed in this zone are cleavage steps, monoatomic or only a few atomic units high. Most of them are arms of V s which, after being deflected into the [100] direction, form the tartan pattern. There is also a small number of longitudinal steps which originate from the screw components of transverse dislocation loops. The number of isolated monoatomic cleavage steps which have their origin in grown-in dislocations is negligible. Longitudinal lines that do not originate from the previous mechanisms, can be accounted for by the nucleation of dislocation loops of the longitudinal systems. Longitudinal lines of both signs appear. An important observation from a large number of micrographs is that there are about the same number of lines of both signs, on average. Although steps do not alternate in sign regularly, the presence of pairs of up and down steps was fairly frequently observed. Groups of closely spaced small or monoatomic steps have in general the same sign. The overall level of the cleavage surface is maintained by groups alternating in sign.

The distribution of longitudinal lines along the full extent of the crack front is not at all uniform. It is believed that isolated segments of longitudinal lines are formed when dislocation loops of the longitudinal system emerge at the cleaved surface. Though a very good resolution was obtained in the shadow-decorated replicas, the possibility that these short segments, which are visible as single lines, were formed by unresolved pairs of lines, as described by Robins *et al.* (1966) for MgO crystals, cannot be ruled out. If that were the case, the distance between lines of a pair would be less than 20 Å.

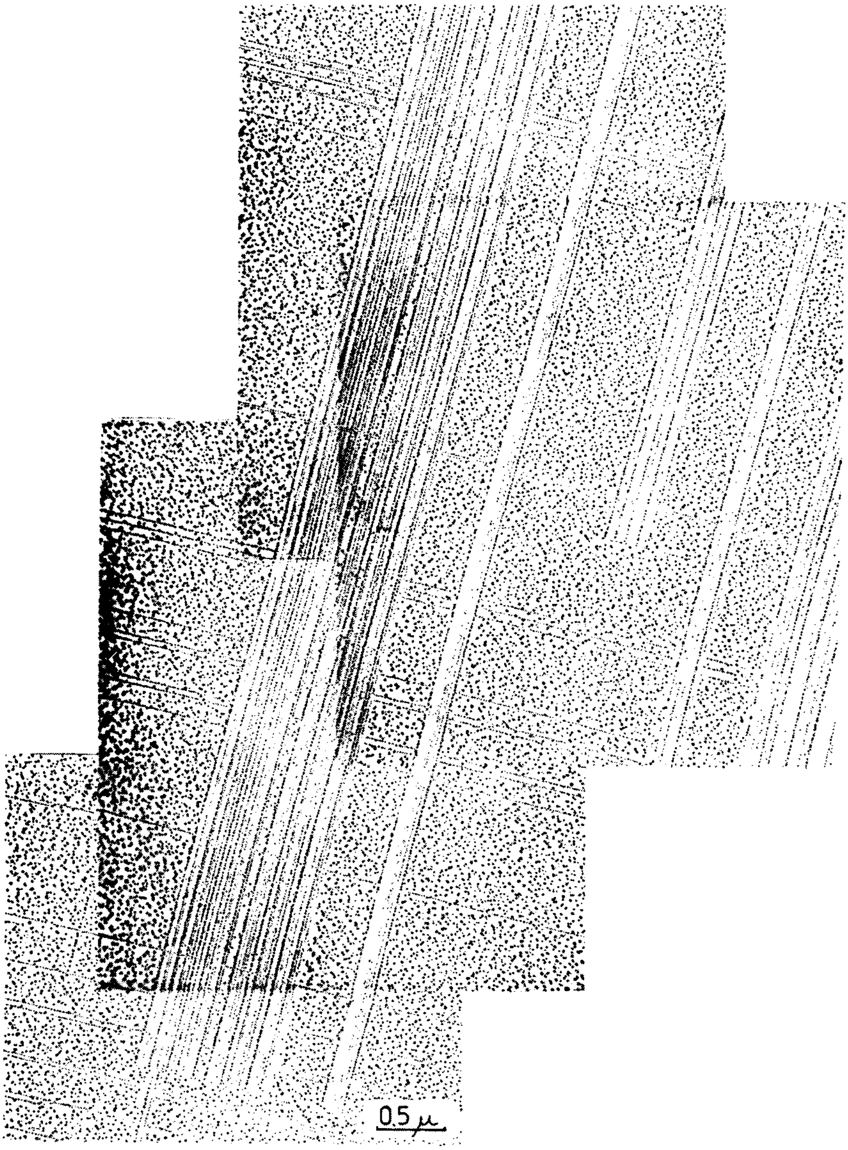
From the large number of shadow-decorated replicas observed, no evidence was found of the presence of more than one longitudinal segment along the same slip trace, unlike the observations of Burns and Webb (1970) on replicas of LiF crystals heated to 125°C. Our observations with the decoration performed at room temperature, rule out the Burns and Webb hypothesis in terms of dislocations moving with the crack tip and support their alternative suggestion that their markings were due to deterioration of the crystal surface before the condensation of the decorating-metal nuclei.

3.3.3. *Bundles : a peculiar structure of the longitudinal lines*

A characteristic feature of the tartan pattern zone is the presence of localized ‘ bundles ’ of parallel longitudinal lines, starting from a transverse slip line and ending in another transverse slip line. Figure 8 is a composite electron micrograph showing the origin of three of these bundles. They stem always from a segment of a single transverse slip line, one end of the transverse line coinciding exactly with the first longitudinal step of the bundle and thus forming an L shape. Sometimes the slip line which gives rise to a bundle has a cross-slip segment and part of the bundle stems from it. There is no noticeable change in the bundles all along their length even when they intersect single or grouped transverse slip lines (see for example group CC in fig. 9).

No acceptable mechanism can be proposed to explain all these features of the bundles if they are assumed to be formed as a consequence of the nucleation and multiplication of dislocation belonging to the transverse slip systems. However, the hypothesis that bundles are formed as a consequence of the activation of the longitudinal slip systems is supported by X-ray topography observations which show (Wainer and Manghi 1977) the localized activation of the longitudinal slip systems in the zone immediately before a stopped crack front. We propose a mechanism by which a side of a longitudinal dislocation loop is cut by the crack and a cleavage step is formed. This step follows the crystallographically preferred [100] direction. The other side of the dislocation

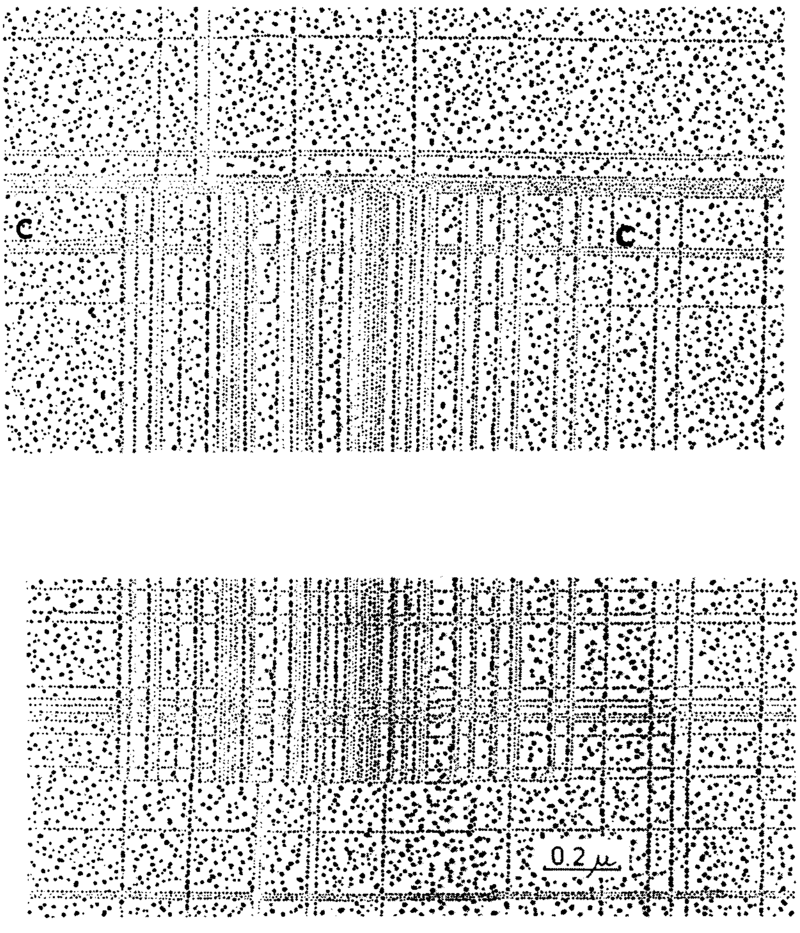
Fig. 8



Composite electron micrograph of a shadow-decorated replica of a NaCl crystal. Origin of bundles of parallel longitudinal lines.

loop is carried along by the stresses ahead of the crack tip, apparently for distances sometimes as long as 200 μ. Eventually the interaction with kinks formed at the intersection of two orthogonal transverse slip systems, or the interaction with the debris left by the multiple cross-slip of dislocations of the transverse systems, slows down the dislocation. This is then cut by the crack, giving rise to a cleavage step of opposite sign which annihilates the previous one (fig. 10 (*a*)). Most frequently, the first side of the dislocation loop cross-slips in the (010) plane. This surface-induced cross-slip (Gilman 1961, Appel and Messerschmidt 1969) gives rise to a [010] slip line, forming the L-shaped base of the bundles (fig. 10 (*b*)). Infection of neighbouring longitudinal planes by multiple cross-slip of a few of these longitudinal dislocation loops can thus

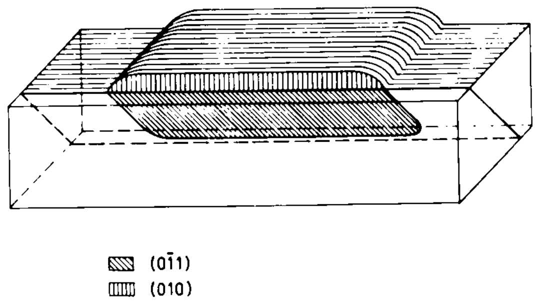
Fig. 9



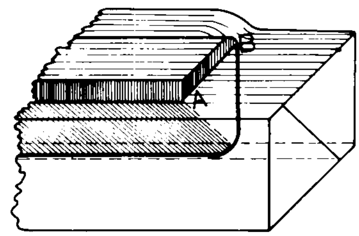
Origin and end of a bundle of parallel longitudinal lines.

account for the appearance of the bundles. Residual stresses and the stresses introduced by the wedging open of the cleavage arms are responsible for this multiple cross-slip ; they also expand the resulting dislocation loops back to the cross-slip segment AB on one side, and to the intersecting obstacle which halted the original dislocation loop on the other side. All the characteristic features of the bundles are explained by this model.

Fig. 10



(*a*)



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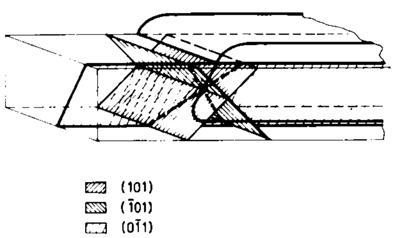


(*c*)

Schematic diagram showing the proposed mechanisms for the origin and end of bundles.

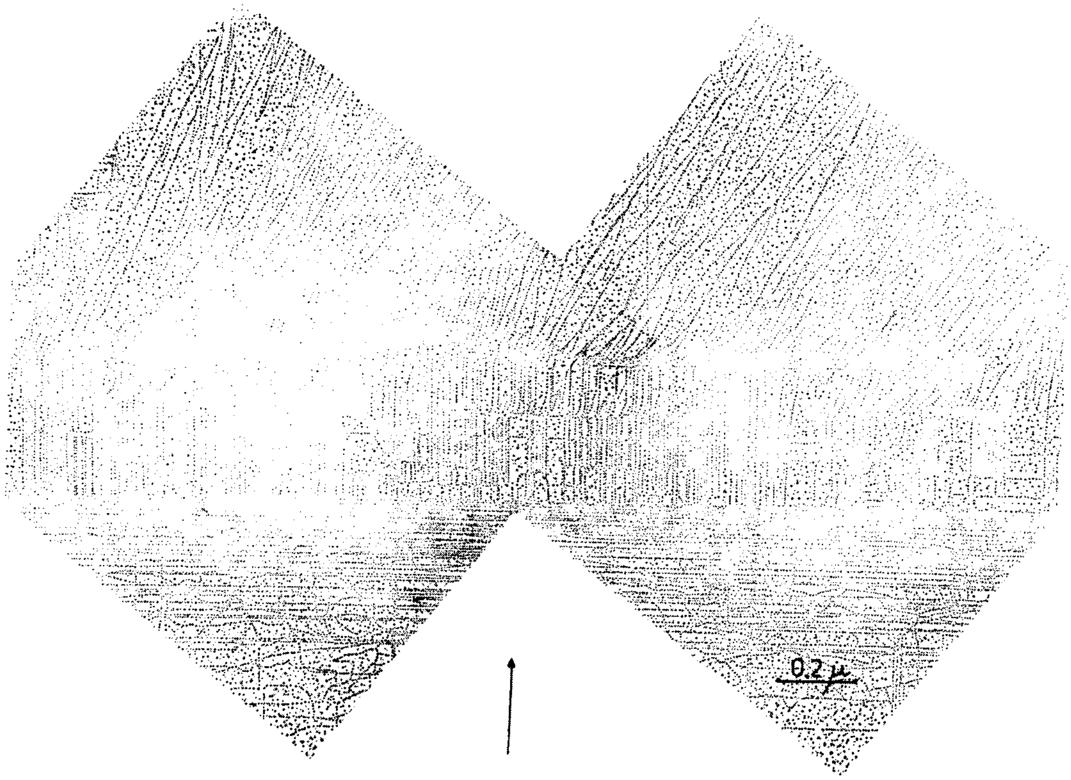
It is proposed that the bands of transverse lines intersected by the bundles have been formed in stages, after the expansion of the longitudinal loops of the bundles has taken place. These transverse bands are formed at each advance of the wedge by multiplication of the existing transverse loops. Therefore, it is quite possible that the expanding longitudinal loops do not actually cross the dense transverse bands.

The fact that bundles, though shorter, appear even for cracks propagating in a [110] direction, further supports the hypothesis that they develop as a consequence of the activation of the longitudinal slip systems.

3.4. *Stop band*

The stop band appears in the last stage of the deceleration process, the immediate healing and the first stage of the restarting process. It is composed of a dense transverse slip band followed by short straight longitudinal lines (fig. 11). A typical value for the length of the stop-band zone is around 2 μ.

Fig. 11



Stop-band zone. Standard decorated replica.

3.4.1. *Transverse slip bands associated with the deceleration processes*

As the deceleration of the crack proceeds, the width and density of the transverse slip bands increases. The stop bands can seldom be resolved into individual slip lines. In the few light bands where this is possible, densities of the order of 10^6 lines/cm have been measured for the transverse slip lines. The stop bands form walls that only cleavage steps higher than 100 Å can transverse with a minor deviation ; atomic cleavage steps and longitudinal slip lines are completely stopped at these bands (fig. 11).

Dislocation processes associated with the formation of transverse slip bands must be the same as those proposed for the tartan-pattern zones : as both transverse systems are active and multiplication by multiple cross-slip of dislocations should occur both by deceleration of the cleavage crack and by the stresses introduced by the wedging open of the crystal arms. These stresses lead to the polygonization of the cleavage halves.

A characteristic feature of the stop bands is the strong interaction between both transverse slip systems, which gives rise to the marked and variable relief best observed in shadow-decorated replicas (not illustrated). This interaction leads even to the upwards or downwards displacement of entire blocks of the crystal. Crack nucleation by the interaction of conjugate systems by the Argon and Orowan (1964) mechanism is considered to be responsible for this roughening of the stop bands.

It is proposed that the main cause of this strong interaction are the stresses introduced by the wedging open of the crystal arms each time the cleavage crack is restarted. However, the influence of the stresses ahead of a decelerating crack tip may also be important.

In fact, cleavage steps are formed at the crack tip, so that the stresses later introduced by the wedging open of the crystal arms cannot be responsible for the observed jogged paths of high cleavage steps.

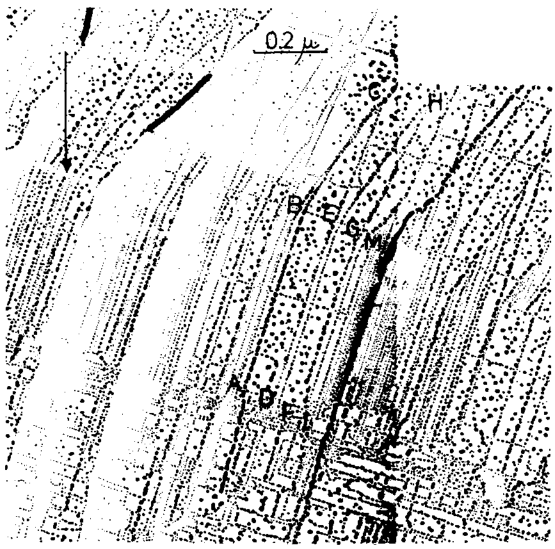
3.4.2. *Short straight longitudinal lines : relationship with dislocations*

Very numerous short straight lines parallel to the [100] direction start at the stop band, in the zones where the local direction of the crack front is roughly parallel to the [010] direction (fig. 12 (*a*)). The length of these short lines ranges from 0·2 μ to about 1 μ. Their density varies from approximately 4×10^5/cm to 7×10^5/cm and their distribution is not uniform. Some of these lines end at small cleavage steps, many change direction and follow the local direction of crack propagation and some of them end at transverse slip lines.

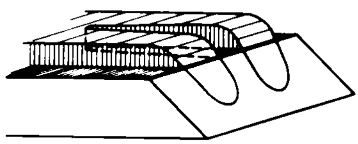
It is proposed that these short straight segments are due to surface-induced cross-slip of one end of the (101)[101] dislocation loops on the (010) plane to reduce their line length. The surface induced cross-slip cannot proceed beyond the place where both cleavage surfaces are united by a healing process. When the crack restarts, a unit cleavage step stems from each cross-slipped dislocation. The observed sequence of signs of the short segments stemming from the stop band, can be understood in terms of this model. If two neighbouring dislocations of opposite sign are very close together, the corresponding steps run parallel for a short time until they annihilate. Pairs belonging to adjacent loops always have the same sequence of signs (positive–negative or vice versa—fig. 12 (*b*)) while the presence of some pairs with the opposite sequence (negative–positive or vice versa) (fig. 12 (*c*)) can be accounted for by the annihilation of steps stemming from the same dislocation loop.

Isolated short longitudinal steps are frequently observed in crack fronts roughly parallel to [010] but not in crack fronts which differ appreciably from [010]. It is suggested that they result from small longitudinal loops nucleated ahead of the crack tip that later expand slightly under residual stresses up to the last transverse slip line. The fact that none of these segments has been observed for inclined stop bands is consistent with this suggestion : the

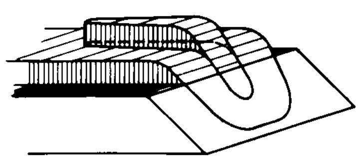
Fig. 12



(*a*)



(*b*)



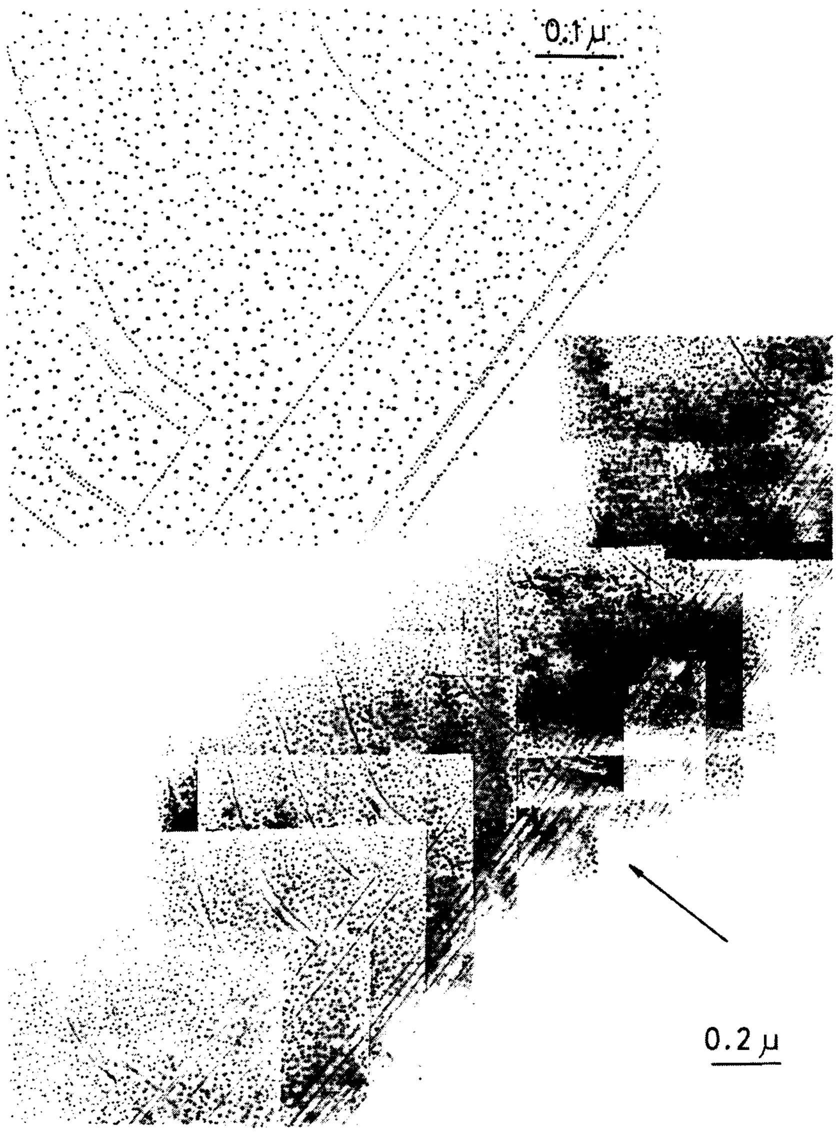
(*c*)

(*a*) Stop-band zone. Electron micrograph of shadow-decorated replica. Shadowing direction perpendicular to the longitudinal steps. (*b*, *c*) Annihilation of pairs of short lines.

longitudinal systems are less active for cracks propagating in a direction inclined to the [100] direction.

A crack front that is roughly parallel to the [010] direction is not strictly straight ; a slight curvature, only visible under the electron microscope is always present. In these slightly curved zones, the short straight lines start always in the [100] direction. This curvature of the crack front can be inferred from the staircase-like ending of transverse slip lines evident in the micrograph in fig. 13. This electron micrograph shows strikingly that a cleavage step originates at the end of every transverse slip line.

Fig. 13



Stop-band zone. Composite electron micrograph of shadow-decorated replica of a NaCl crystal. Shadowing direction perpendicular to the longitudinal steps. Inset : high magnification of part of composite micrograph.

3.5. *Restarting zone with inclined steps*

The zone immediately ahead of the stop band shows an intricate pattern formed simultaneously by a river pattern and something similar to a lightning pattern (fig. 14). This intricate pattern arises from the imperfect healing which usually occurs when the crack is halted. As the crystal surfaces become more intimately united, there is a variable mismatch at numerous sites of the cleavage surfaces. When cleavage is restarted, the crack must cut through these faulted regions producing a multitude of unit cleavage steps. Etching of these faulted regions yields shallow, rounded large pits showing that the 'healed-in' dislocations are of a different nature to those introduced when stressing a crystal either by propagating a decelerating crack or by applying compressive or tensile stresses, confirming the observations of Forty and Forwood (1963).

Fig. 14

Restarting zone with inclined steps. Standard decorated replica of a NaCl crystal.

§ 4. Conclusions

When an unsteady cleavage crack propagates through NaCl, a characteristic sequence of surface markings is observed in a region between two stopped crack fronts. The surface markings can be divided, according to velocity and direction of cleavage into five zones : (1) V-zone present when the crack is running freely in a direction different from [100]; (2) a transition zone related to the initial deceleration of the crack; (3) a tartan-pattern zone associated with the deceleration process; (4) a stop-band zone marking the at-rest position of the crack and including the last stage of the deceleration process and the initial stage of the restarting process; (5) a restarting zone associated with the re-acceleration of the crack. These surface structures are related to the activation of the longitudinal and transverse slip systems accompanying crack propagation.

Some authors (Gilman 1957, Burns and Webb 1970) have presented arguments in favour of the hypothesis that the longitudinal mode of deformation is the one that contributes most to the stop band associated with a stopped crack. However, it is evident from X-ray topography (Wainer and Manghi 1977) and from the electron microscopy results discussed in this paper that what characterizes the deceleration process is activation of the transverse systems.

A simplified model has been constructed illustrating the manner in which the activated transverse systems can account for the cleavage markings observed at the stop bands; it also explains the marked relief appearing as a consequence of their interaction.

In addition it has been established that both longitudinal conjugate slip systems are active. The activity of the longitudinal systems in the deceleration zone is observed mainly through the presence of bundles of longitudinal slip lines. A model has been proposed to correlate the activation of the longitudinal systems with the peculiar bundle structure observed. This takes into account interaction between orthogonal and oblique slip systems.

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