DISLOCATIONS IN LITHIUM FLUORIDE

A microscopical exercise in plastic deformation using dislocation etch pit and birefringence techniques, with appropriate crystallographic background

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I. INTRODUCTION

DISLOCATION GEOMETRY

With very few exceptions, solids are crystalline. A crystal consists of atoms arranged in a regular three-dimensional array or lattice. Crystals may be classified in terms of their lattice geometry, but often it is more convenient to base classification on the predominant type of atomic bonding, for example, ionic, covalent, or metallic.

When crystals are stressed they undergo a change of shape which is elastic i.e. the crystal reverts to its original dimensions when the stress is removed. Above a critical stress, known as the yield stress, this elastic deformation may be accompanied by permanent or plastic deformation which remains when the stress on the crystal is relaxed.

Many types of crystal exhibit little or no plasticity before fracture occurs, and such crystals are said to be brittle. On the other hand, crystals of metals and alloys frequently possess a reasonable degree of plasticity or ductility, and this is one reason for their great importance as constructional materials. Their ductility not only enables them to be fabricated into many complicated shapes but it also allows them to relieve high local stress concentrations in service without fracturing.

Other types of crystal may undergo plastic deformation; among ionic crystals, examples are AgCl, NaCl, LiF, MgO, TiO$_2$ and CaF$_2$ and in covalent crystals Ge, Si, and InSb. Very few of these however, have ductilities approaching those of metals, and then only under very special conditions.

The Slip Process

The most important mechanism by which plastic deformation occurs is that of slip. In this process (Fig. 1), one part of the crystal shears or slips over the other on a specific crystal plane (the slip plane), and along a specific crystal direction (the slip direction) which lies in the slip plane.

The combination of a slip plane and a slip direction lying in that plane constitutes a slip system. The smallest distance or vector describing the slip movement is equal to the distance between equivalent atoms along the slip direction; this means that the crystal structure is restored after the slip process. A characteristic of slip, as Fig. 1. shows, is that steps are produced on the crystal surface; these steps appear as slip lines or traces. It should be noted (Fig. 1) that slip steps are not produced on crystal faces which lie parallel to the slip direction.

In real crystals, particularly for small amounts of plastic deformation, it is observed experimentally that the deformation is heterogeneous. The deformation occurs in localized regions of the crystal where slip takes place on a number of parallel and closely spaced planes. These regions are called slip or glide bands.

If a crystal has more than one slip system, the system which operates initially is that on which the resolved component of the applied stress (that is the shear stress acting in the glide system) along the slip direction is greatest. At the yield stress, if
there is an equal resolved shear stress on a number of systems, they will be equally favoured for slip.

**Dislocation Mechanism of Slip**

![Diagram of slip process](image)

*Fig. 1. The slip process under a compressive stress (under tension the slip direction would be the reverse of that shown).*

Some years ago it was realised that slip involved the movement of a particular type of imperfection which is present in crystals. This defect is called a dislocation. The properties of dislocations, their ability to multiply under the action of a stress together with their interactions with one another and with other types of crystal defect, particularly solute atoms, precipitates, and grain boundaries, largely determine such mechanical properties as yield stress, creep strength, and ductility.

A dislocation may be pictured quite straightforwardly by considering that the slip process does not involve the simultaneous shearing of one part of the crystal over the other, but rather, that the process takes place as a consecutive motion beginning in a small area on the slip plane and then expanding across the slip plane. At any stage during this movement a boundary can be specified which separates that area of the slip plane which has slipped from that which has not. This boundary is the dislocation line. On an atomic scale it is the region in the crystal where the atoms are in transitional positions between the equilibrium sites of the slipped and the unslipped parts of the crystal. In real crystals this region extends over many atoms and it is convenient to consider the dislocation as the line which runs through the centre of the disturbed region.
Since a dislocation bounds an area, it is always continuous unless the area includes the crystal surface. Consider Fig. 2 where, under the influence of a shear stress \( \tau \) the top half of the crystal has slipped relative to the bottom half over an area SBEF of the slip plane ABCD in the direction of, and by a magnitude equal to, the vector \( b \). The line EFS then represents a dislocation line. The vector \( b \) is called the Burgers vector of the dislocation. For a single dislocation which produces slip, the Burgers vector is equal to the slip vector, that is the shortest distance between equivalent atoms along the slip direction (p. 1). Single or unit dislocations of this type are known to be responsible for slip in lithium fluoride. However, in certain structures, for example, many metals, graphite, and \( \text{TiO}_2 \) a given slip vector can be achieved by a number of dislocations having Burgers vectors which add up to the Burgers vector of an equivalent unit dislocation. Such dislocations are known as partial dislocations and it follows from their nature that they are separated from each other by regions of faulted structure. The combination of partials and faulted regions is known as an extended dislocation.

Fig. 2. Quarter loop of dislocation EFS on slip plane ABCD

Returning now to consideration of unit dislocations, the structure and properties of a dislocation vary with the angle it makes with the Burgers vector. As Fig. 2 shows, this angle may lie between 0 and 90°. The two extreme cases are known as an edge dislocation (segment EE' in Fig. 2) which lies perpendicular to the Burgers vector, and a screw dislocation (segment SS') which is parallel to the Burgers vector. Theoretical considerations of dislocations are usually confined to the edge and screw configurations since a dislocation of any other inclination to the Burgers vector (called a mixed dislocation) may be considered to be a combination of edge and screw dislocations. Edge and screw dislocations will now be analysed in more detail.

**Edge Dislocations**

A shear stress is applied to a crystal block with a slip plane ABCD so that part of the top half slips over the bottom half by an amount described by the vector \( b \) (Fig. 3(a)). The line EF represents the boundary between the slipped and unslipped areas on the slip plane and, since it is at all points at right angles to the Burgers vector, it is entirely an edge dislocation. When further slip occurs the dislocation EF will move in...
the direction of \( b \) until it passes out of the crystal along CD. The two halves of the crystal will then have slipped by an amount \( b \) as shown in Fig. 3(c).

![Diagram](image)

**Fig. 3.** Edge dislocations of opposite sign: (a) with the half-plane above the slip plane, and (b) with the half-plane below the slip plane. Under the same shear stress they produce the same deformation of the crystal by moving in opposite directions—see view (c).

Fig. 4 shows an idealized representation of the distribution of atoms around an edge dislocation in a simple cubic structure. The dislocation lies on a (001) plane, that is parallel to one of the cube faces, and has a [010] Burgers vector, i.e. along a cube edge as indicated. The plane of atoms depicted in the diagram is indexed (100) and is equivalent but normal to the (001) plane containing the dislocation.

It will be seen from Fig. 4 that the edge dislocation lies immediately beneath an incomplete plane, or half-plane of atoms (the plane EFGH in Fig. 3(a)). This extra material produces a distortion in the crystal; the top half of the crystal in Figs. 3(a) and 4 is in a state of compression and the bottom is in tension. Thus a small misorientation or bend exists about an edge dislocation line. An edge dislocation may be denoted by the symbol \( \psi \) (Fig. 3(a)), where the horizontal line represents the slip plane and the vertical line the position of the half-plane. The deformed crystal of Fig. 3(c) may also be produced by an edge dislocation, which has its half-plane lying
below the slip plane (Fig. 3(b)), being initiated along CD and moving to AB. Such a
dislocation would be denoted by the symbol T. These dislocations υ and T are said to
be of opposite sign (positive and negative, respectively) and under the same shear
stress will move in opposite directions, as shown in Fig. 3. It will be appreciated that
when dislocations of opposite sign coincide on a given slip plane, they cancel out or
annihilate each other.

![Diagram of edge dislocation](image)

**Fig. 4.** Edge dislocation in a simple cubic structure. This dislocation has a
[010] Burgers vector and a (001) slip plane. The diagram shows the distribution of atoms in the (100)
plane which is normal to the slip plane.

### Screw Dislocations

Fig. 5 demonstrates how a screw dislocation may be introduced into a crystal block.
Here the two halves of the crystal have sheared relative to each other over the area
EBCF of the slip plane ABCD. EF is again the boundary between the slipped and the
unslipped areas and, since it lies parallel to the Burgers vector, it is a screw
dislocation. Unlike the edge dislocation, no incomplete planes of atoms are
associated with a screw dislocation; the distortion it produces is one of pure shear.
When the crystal undergoes further slip, the screw EF will move at **right angles to the
Burgers vector**, towards AD. When it passes from the crystal the configuration of Fig.
3(c) will again be produced.

A screw dislocation in a simple cubic structure, equivalent to the edge of Fig. 4, is
deplotted in Fig. 6. Slip has occurred as indicated by the shaded region. To the right,
unit translation or movement has been made, but this translation decreases to zero
at the boundary between the slipped and unslipped regions. The present boundary is
called a screw dislocation because the displaced atoms in the neighbourhood of the
boundary e.g. a, b, c, d, e, follow a spiral or screw like sequence which continues and
extends along the dislocation providing it remains of screw character. The spiral in
Fig. 6 follows a right-handed sense. Screw dislocations may be of opposite sign since
the spiral may follow a left-handed sense. As for edge dislocations of opposite sign
screw dislocations of opposite sign (or sense) produce the same deformation by
moving in opposite directions, and they will also annihilate one another when they
coincide.
The ways in which dislocation movement produces plastic strain are demonstrated schematically in Fig. 7. for a crystal under (a) compression and (b) tension. For convenience edge dislocations only are considered and in both cases slip is taking place on two different but equivalent planes. During compressive strain the length of the crystal decreases and this may be accomplished by the dislocation motion shown in (a). On the other hand, tensile straining lengthens the crystal, by the dislocation movements illustrated in (b). Comparison of (a) and (b) shows that dislocations of a particular sign produce compressive or tensile strain by moving in opposite directions.

Returning to Fig. 2., it may now be seen that, as the crystal is deformed, the edge segment EE’ and the screw segment SS’ of the dislocation line EE’ will move towards the surfaces CD and AD respectively, that is, the quarter loop of dislocation will expand across the slip plane ABCD. In Fig. 8 it has expanded such that EE’ has passed out of the crystal on part of the slip plane and has produced the slip step CG; the screw SS’ has moved towards AD and the step SB has been extended. When the dislocation passes across the whole slip plane the crystal will have been deformed in an identical manner to that of Fig. 3(c). The shape of the dislocation SFE depends on the relative velocities of screw and edge dislocations in a particular crystal structure. For example, if at a given stress, edges moved faster than screws, the dislocation loop would be predominantly of screw character.

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**Fig. 5.** Screw dislocation EF on slip plane ABCD.

**Fig. 6.** Screw dislocation in a simple cubic structure. It lies in the (001) plane and has a [010] Burgers vector.
Fig. 7. Schematic representation of the edge dislocation movement required to produce equal increments of slip on two different slip planes in a crystal undergoing: (a) compressive, and (b) tensile deformation.

Fig. 8. Dislocation loop GFS on slip plane ABCD.

As a result of the distortion or strain produced by a dislocation, the neighbouring atoms cannot occupy their normal equilibrium positions in the structure. The situation is illustrated in Figs. 4 and 6 where the atoms in the immediate vicinity of the dislocation do not conform to the simple cubic structure. The atoms occupy positions of higher energy which means that dislocations possess strain energy. Calculations show that the strain energy of a dislocation is proportional to the square of its Burgers vector. Thus the unit dislocation with the smallest possible Burgers vector will have the lowest strain energy and, being the most stable, will therefore be responsible for slip.

In some crystals, slip results from the movement of extended dislocations (p. 3) because they have a lower energy than that of an equivalent unit dislocation. Extended dislocations are not involved with lithium fluoride as this structure does not readily form the faulted regions necessary for the dissociation of the unit dislocation into partial dislocations.
Special Motion of Dislocations

The above discussion has been concerned with a description of dislocations and their movement on one specific slip plane under the influence of an applied stress. In real crystals the movement of dislocations is more complicated and it is useful to point out some examples of this. It follows from the definition of slip that a dislocation only produces slip when it moves in a plane which contains its line and its Burgers vector. This criterion limits the movement of an edge dislocation to one unique plane whereas it in no way restricts the motion of a screw dislocation. Since the line and the Burgers vector of a screw are parallel, in principle it can slip on any plane on which it lies. Thus it is possible for screw dislocations to slip from one plane to another; this phenomenon is called cross-slip and is illustrated in Fig. 9 (compare with Fig. 5). A screw dislocation which was initiated along AB has slipped across the plane ABCH and then moved on to a parallel plane DEFG via the plane CDGH. The plane CDGH is called the cross-slip plane.

Edge dislocations may move in a direction normal to their slip plane as a result of a diffusion controlled transfer of point defects, either lattice vacancies or interstitial atoms, to and from the dislocation half-planes. Such motion is called climb and occurs predominantly at elevated temperatures. In this way drastic rearrangements of dislocations can occur. These rearrangements involve the annihilation of many dislocations (as dislocations of opposite sign cancel each other out when they coincide), and the formation of stable dislocation arrays (like those in sub-grain boundaries). By these processes the strains in a deformed crystal are reduced and the crystal is said to undergo recovery. Dislocation movements during recovery are further described in Section IV.

![Fig. 9. Illustrating the cross-slip of a screw dislocation](image-url)
II. DEFORMATION STUDIES WITH LiF

Some of the alkali halides such as LiF, may be prepared in states of high purity and structural perfection. Suitable specimens may be obtained by cleavage rather than cutting, and the nature of the material lends itself to the ready detection of the presence of dislocations. Such halides are therefore very suitable for experimental studies on the properties of dislocations. In particular, a number of significant advances in understanding have followed the development of techniques for the detection and observation of dislocations in LiF, which for the reasons given above has been adopted for the present exercise.

LiF forms an ionic crystal with the NaCl structure; the accompanying ball model represents an elementary cubic cell which also is depicted in Fig. 10. The lithium and fluorine are present respectively as positively and negatively charged ions. The binding force holding the ions together in the structure is predominantly the electrostatic force between ions of opposite electrical charge.

LiF slips on planes of the {110} type i.e. the body-diagonal planes; the six possible planes are shown in Fig. 11. The slip vector is of the face-diagonal, <110> type; each of the possible vectors lies in one of the {110} planes and so the crystal has six slip systems. If the crystal is compressed in an [001] direction, as in Fig. 11, it may slip on four of the possible slip planes: (011), (011), (110) and (101); all are equally inclined to the stress axis and will therefore have equal resolved shear stresses acting on them. The (110) and (110) planes lie parallel to the stress axis and the resolved shear stress acting on them is zero.

LiF of high purity may readily be produced in single crystal form and accurately parallel faces may be prepared by cleavage on the {100} planes. Two particular ex-
experimental techniques can be used on these crystals which, in combination, form a powerful method of examining deformation processes. These are the ‘etch pit’ technique of revealing the sites of dislocations at the crystal surface, and the optical technique of stress-birefringence for revealing the distribution of strain.

The former technique depends on the energy of a crystal surface being higher at the point of emergence of a dislocation, because of the strain energy associated with the dislocation. It is thus possible to reveal the site of the dislocation by immersing the crystal in a solution in which the crystal is slightly soluble. With suitable solutions the region of the crystal very close to the dislocation is dissolved more rapidly than the remaining, more regular, surface. This leads to the production of dislocation etch pits on the surface; the size and shape of the pits depend upon the solution used, the time of etching, the type of crystal structure and the crystal plane which is etched.

The stress-birefringence technique relies on the fact that some transparent crystalline substances become doubly refracting when elastic strains are introduced. Under suitable illumination with polarized light, these strains are rendered visible as contrast effects. In this way, the slipped regions of a crystal, which are in a strained condition due to the elastic distortions associated with the dislocations may be detected. Both the above techniques are used in the present experiments and further details of their application to LiF are given in the following sections.

III. EXPERIMENTAL TECHNIQUES

(1) Crystal Material and Handling Precautions

The LiF is provided in the form of blocks (2.5 x 4 x 12 mm), cleaved from a large melt-grown single crystal so that all faces are \{100\} planes. The material contains a total impurity content of \(10^{-4}\) to \(10^{-5}\) mainly as polyvalent metal ions, such as Mg++. It is very easy to damage the specimens by careless handling. Specimens should be picked up near their ends using tweezers, and the faces to be examined must not be allowed to slide over hard surfaces, for example the microscope stage. Unless these precautions are followed numerous spurious dislocations will be introduced into the specimens.

(2) Microscopy

Two microscopical techniques are used to reveal the deformation features. The basic requirements are:

(a) A reflexion microscope, preferably with provision for some degree of oblique illumination, and if possible with a traversing stage for examination of surface features (magnification 50 – 500x). If a proper oblique illuminator is not available, suitable effects can sometimes be obtained by off-centring the light source, or for low power work a separate source can be directed on to the specimen from the side.

(b) A low power transmission polarizing microscope, preferably with a rotating stage, for examination of stress-birefringence (magnification ~ 30x).
(3) Etch Pits

Dislocation etch pits are reliably formed in LiF by etching in 20 vol.% hydrogen peroxide. Enough fresh solution to cover the specimen should be placed in a shallow dish, the specimen then being submerged for about 10 min. Specimens should be rinsed thoroughly after etching, in ethanol (and finally in ether). If the surface does not dry properly after a few seconds in the atmosphere, the rinsing procedure should be repeated. All reagents, especially the rinses, must be renewed after use with a few specimens.

![Plan view and sectional view of etch pits](image)

*Fig. 12. (a) Etch pit formed at freshly introduced dislocation. (b) Etch pit formed at 'annealed-in' dislocation. (c) Dislocation motion detected by double etching technique; dislocation at 'A' during first etch and at 'B' during second etch.*

Etch pits are best viewed in oblique reflected light at 100-500x magnification. Under these conditions contrast effects are produced between the facets of the pits and the surrounding surface. Etch pits formed at freshly introduced dislocations have the form of a square pyramid with the base edges in <100> directions and the apex of the pit sited at the line of the dislocation (Fig. 12(a)).

Dislocations that have been subjected to high temperature, for example those introduced during the growth of the crystal, have impurities precipitated on to them and this effect reduces the sensitivity of the etch. Pits formed at such dislocations tend to be somewhat rounded (Fig. 12(b)) and thus can be distinguished from those produced by plastic deformation.

Dislocation motion may be detected by a double etching technique. If a dislocation which has been located by means of etch pit is moved under the application of a stress, then during a second etch a new pit will be formed at the new site of the dislocation; meanwhile the original pit continues to develop laterally but not in depth (Fig. 12(c)).

Other pits which are shallow and rounded in form are produced at small impurity precipitates and these cause a general background attached of the crystal surface.

(4) Stress Birefringence

This effect is observed in transmitted plane polarized light. The polarizer and analyser are set in the extinction or crossed position with their polarizing axes at 90°. The specimen is sited between the polarizer and analyser with one of the cleaved faces perpendicular to the light beam. In the unstressed condition LiF is isotropic having
the same transmission characteristics for any orientation. The crystal thus appears dark for all orientations between crossed polarizers.

However, the optical characteristics are affected by stress and vary according to the stress distribution. As a result, with appropriate relative positions of polars and crystal, varying contrast is developed and the strained regions can be detected (Fig. 13).

In fact, two sets of stresses will normally be present in a crystal undergoing compression. First, there is an axial compressive stress (with associated birefringence) which fails to zero when the applied pressure is removed. Secondly, when the applied pressure is sufficient to produce plastic deformation, additional elastic strains are set up near the active slip planes. The principal stresses occur in the <110> directions, parallel and perpendicular to the active slip planes. Birefringence effects can now be observed around the slip planes when the crystal is viewed under suitable conditions.

It follows from detailed optical considerations that contrast effects associated with slip reach maximum contrast when the crystal is arranged with its edges parallel to the polaroid directions. Under these conditions birefringence effects due to any applied compressive stress are conveniently zero.

![Figure 13](image_url)

*Fig. 13. Stress birefringence due to slip in crystal compressed 2 %. x 30.*

*(Note: at lower strains, the pattern is much simpler).*
IV. EXPERIMENTS

(1) Examination of As-Received Material

Examine the \{100\} cleavage faces of a given specimen in reflected light (magnification, approximately 100x). A number of fine lines can be seen; these indicate cleavage steps which run continuously across the specimen in a direction roughly parallel to the direction of cleavage. The lines are small steps where the cleavage crack has changed from one (100) plane to another parallel plane. Examples may be seen in Figs. 15 and 20.

Etch the specimen and re-examine; there are two main new features:
(a) First, there are lines of closely spaced pits running in random directions which link together to form a network. These lines delineate sub-grain boundaries. The growth process is not quite perfect and the material comprises a number of slightly misoriented regions or sub-grains. The misorientation between subgrains (\(<< 1^\circ\)) is accommodated by a close array of dislocations which are revealed as a row of pits. The larger the misorientation between the sub-grains the greater is the density of pits in the boundary. In many of the boundaries in a given specimen it should be possible to resolve the individual pits. Other randomly distributed pits occur within the sub-grains at single grown-in dislocations. The total dislocation density is approximately $10^5$ per cm$^2$, this can be measured by counting the number of pits in a given area of an etched surface.
(b) The second feature of the etched specimen is the presence of a general background attack, caused by the higher solution rate of small impurity precipitates. This effect and that of the dislocation etch pits are illustrated in Fig. 14. In addition, etch pits occur along cleavage steps or at dislocations accidentally introduced during handling.

As a final stage in the examination of the as-received material, the crystal should be observed in transmitted polarized light in order to confirm that it is isotropic, indicated by the crystal appearing dark in all positions. It should be noted that a certain amount of strain is unavoidably introduced near the crystal surfaces during the cleavage operation. In consequence, edge regions may be of variable brightness. However, this effect should be ignored and attention concentrated on the general features in the centre of the crystal.

(2) Confirmation of the Slip Systems

Mount a specimen in the vice with slight pressure along the long axis and with the 2.5 x 12 mm side faces horizontal. Place the vice on the microscope stage for viewing in transmitted polarized light and focus halfway through the specimen. Increase the vice pressure slightly and rotate the specimen with respect to the crossed polaroids. The crystal appears dark (extinction occurs) when the polaroid axes lie parallel to the edges of the crystal. The crystal is now in the optimum position for viewing slip effects, those due directly to the applied stress are eliminated as previously indicated.
Leave the polaroids and crystal in the extinction position and slowly increase the stress on the crystal. When the yield stress is reached plastic deformation occurs and glide bands are produced; these are shown up as contrast effects by reason of birefringence (Fig. 13). Stop stressing when some 10-20 bands have been formed in the field of view and remove the crystal from the vice. Further examination in polarized light of one of the 2.5 x 12 mm faces and a perpendicular 4 x 12 mm face of the crystal should reveal that up to four slip systems have operated (Fig. 11), depending on the specific level of applied stress. In general, the two systems producing birefringence in the narrow faces of the crystal will predominate.

Slip steps will now be apparent in oblique reflected light. These lie in the <100> directions (cube edge directions) normal to the compression direction (Fig. 15). Check that no steps are visible in <110> directions (face diagonals).

Now etch the specimen and examine again in oblique reflected light. Rows of pits lying in both <100> and <110> directions are present, because although steps do not develop along the latter, dislocations emerge at the ends (corresponding to <110> directions) of the slip planes. In fact, if the amount of deformation has been small, it should be possible to trace the rows of pits round a corner of the specimen and thus delineate the {110} slip planes (body diagonal planes), as in Fig. 16, where the two slip systems mentioned above have predominated.
Fig. 15. Slip steps (vertical) in deformed crystal. Cleavage steps from top left to bottom right. X 200

Fig 16. Left: Etch pits at ends of two sets of intersecting slip planes on a (001)-face near a crystal edge. Right: Etch pits on the adjacent (010)-face. X 100
The facing view of Fig. 16 represents one crystal face, and the reverse side flap reveals an adjacent perpendicular face. By holding the two views at right angles, one has a highly magnified picture of a crystal edge with the slip planes marked out by their etch pit traces.

The observations thus far are summarized in Fig. 17. From this data the slip systems of LiF may be confirmed. The birefringence and etch pit observations reveal that the slip planes are \{110\}, and Fig. 17 illustrates slip on two sets of planes in this family, corresponding to those actually observed in Fig. 16.

As slip steps are seen only in \(<100>\) directions, and not in \(<110>\) directions, then the slip direction has no component in \(<100>\) directions and must therefore lie in one of the \(<110>\) directions. It should be appreciated that the foregoing statement refers broadly to groups of equivalent directions which are those parallel to cube edges or face diagonals respectively. The group indices are enclosed in acute brackets, whereas the specific indices (from each group) shown in the diagram are as by convention enclosed in square brackets.

Fig. 17. Summary of observations on deformed crystal.

Consider a quarter dislocation loop on a \{110\} slip plane with a \(<110>\) Burgers vector \(b\) (Fig. 18). Note that the part of the loop at A lies normal to the Burgers vector and is therefore of edge character, while that at C is parallel to the Burgers vector and must thus be of screw nature. Parts such as B are of mixed character. In general a glide band contains a system of such loops lying in several closely spaced parallel slip planes. Rows of etch pits found in \(<110>\) directions in the experimental crystal therefore correspond to edge dislocations and rows in \(<100>\) directions to screw dislocations. Under the indicated applied force, the loop expands and the step grows towards D; this accommodates the strain by making the crystal shorter.
Fig. 18. Quarter dislocation loop ABC on (101) slip plane—this plane is one of the {110} family.

Fig. 19. Examples of <100>, <110>, and <111> Burgers Vectors in LiF Structure.

The reason for the adoption of the <110> Burgers vector during slip may be appreciated from a consideration of the crystal structure of LiF (Fig. 19). The Burgers vector of slip dislocations for a particular crystal structure is usually the shortest vector that links equivalent ion sites. Vectors for specific examples of <100>, <110>, and <111> directions are shown in Fig. 19 and these have lengths of a, a/2, a/3, respectively, where a is the dimension of the elementary cell. As the energy of a dislocation is proportional to the square of its Burgers vector, the energies for dislocations in these directions are proportional to a², a²/2, and 3a² (note that the alternating positive and negative ions along the <100> and <111> directions make the vectors for these directions twice the distance between adjacent ions). The dislocations with the lowest energy are those with the 1/2<110> vector. These dislocations are therefore favoured for deformation, in agreement with the observed results.

The <110> directions are common to many crystal planes, for example {100}, {110}, and {211}. The Burgers vector for slip in many crystals lies in the planes of closest packing of atoms, which in LiF are the {100} planes, but observations show that the slip planes are the {110} series. The reasons for the adoption of the less
dense \{110\} planes are rather involved and cannot be fully explained here. However, it can be said that the main factor is associated with the repulsive forces between ions of like charge which are brought into close proximity as the dislocation moves. In LiF this effect is less for dislocations moving on \{110\} planes than for those moving on \{100\} planes.

(3) Interpretation of the Dislocation Pattern around Micro-Indentations

Make a series of approximately 10 micro-indentations along the centre line of one of the large faces of a new specimen. Use a microhardness indentor with a load of the order of 50g, or press the surface gently with a needle point. Afterwards etch the crystal, when typical -rosette- patterns of etch pits will develop around each indentation (Fig. 20). The rows of pits along the two \(<110\>\) directions correspond to edge dislocations and each direction is associated with just one slip system. The rows of pits in the \(<100\>\) directions are due to screw dislocations and in this case each direction could correspond to slip on either of two slip systems because this direction can lie in two equivalent slip planes (See Fig. 11). The nature of the dislocations in each case can be specified from the deduction given on p. 17, Fig 18.

![Etch pit distribution in a 'rosette' produced by micro-indentation. Crystal etched after indentation subjected to tensile force and re-etched. x 200.](image)

Now mount the specimen in the vice as before with the 2.5 x 12 mm side faces horizontal, and increase the stress until glide bands are nucleated along about one third of the total length of the crystal. Care must be taken not to apply too high a stress as the presence of too many glide bands will obscure further observations when the specimen is etched again. The purpose of the stressing is to move the dislocations in the rosettes and this occurs at a stress similar to that required to nucleate new glide bands.
Now mount the specimen in the vice as before with the 2.5 x 12 mm side faces horizontal, and increase the stress until glide bands are nucleated along about one third of the total length of the crystal. Care must be taken not to apply too high a stress as the presence of too many glide bands will obscure further observations when the specimen is etched again. The purpose of the stressing is to move the dislocations in the rosettes and this occurs at a stress similar to that required to nucleate new glide bands.

Re-etch the specimen and examine the indented face under oblique reflected light, selecting a rosette not obscured by the presence of the glide bands introduced by the compression. For reasons discussed below, little or no dislocation movement is expected for the screw dislocations corresponding to pits in $<100>$ rows parallel and normal to the stressing direction. However, the other dislocations are in a position to move, and any motion is indicated by the formation of new sharp pits, whereas the original pits become larger although remaining shallow under the second etching (Figs. 12(c) and 20(b)). Note that this movement is only detected in four of the eight rosette arms which contain edge dislocations, and reasons for this behaviour are given in the following analysis.

The micro-indentation has produced slip on all of the six possible slip planes in the crystal. Consideration of Figs. 11 and 20(a) will show that the $<110>$ branches of the rosette represent two of the slip planes. On the other hand, the $<100>$ branches each represent two equivalent but inclined slip planes.
A subsequent stress applied, as in the present case in a <100> direction, will not affect the planes represented by the <100> branches parallel to the stressing direction because no shearing action will be produced. In contrast, a shearing action will be exerted on the planes represented by the <100> branches perpendicular to the stressing direction. However, these branches contain screw dislocations, and it is known that under a given stress screws move approximately 50 times slower than edge dislocations. Hence little movement of the screws is to be expected.

It follows that only the more mobile edge dislocations will achieve significant movement. The edge dislocations involved will be those lying on planes undergoing a shearing action from the applied stress, such as those observed in the <110> branches of the rosette.

The direction of movement of the edge dislocations will be determined by their sign, that is the relative position of their half-planes. An edge dislocation of a given sign can in principle move in two directions normal to its half plane. In each case the resulting translation of the region containing the half-plane is in the direction of movement of the dislocation.

In the present case however, edge dislocations cannot readily move in a direction towards the centre of the indentation, especially at a relatively low stress. This is because the high concentration of dislocations existing around the indentation offers a definite obstacle. The general tendency will be for movement away from the indentation.

Thus under a given stress system edge dislocations of one sign will be able to move outwards, if this movement satisfies the deformation requirements of the applied stress. On the other hand, dislocations of the opposite sign will not be ready to move significantly because they could only achieve the desired response by moving towards the indentation.

Fig. 20 represents a crystal strained under tension, and in each case only one of each pair of <110> branches shows evidence of dislocation movement away from the indentation. The dislocations in these arms or branches are therefore of a sign permitting movement away from the indentation. As the dislocations in each of the other branch pairs do not move, they are of the opposite sign. If the stress system is reversed by applying compression, then the other branches in the pairs will show preferential movement of dislocations.

Fig. 21 analyses the stress system in one of the arms in which dislocations have moved. In this case an applied tensile force causes shearing which tends to move...
the material above the slip plane to the left and that below to the right (outwards). Positive dislocations, i.e. those with half planes above the slip plane would have to move inwards to achieve the induced shearing action. However, negative dislocations (with half planes below the slip plane) could produce the required shearing by moving outwards. It can thus be concluded that the dislocations that move under tension in arm A are negative, as illustrated in Fig. 22.

![Fig. 22. Interpretation of the dislocation movement revealed in Fig. 20, in terms of the positions of the half-planes of the edge dislocations.](image)

As the dislocations in the second arm B do not move, these are positive. When compressive stresses are applied to the crystal, as in the practical exercise, the shearing action is reversed, and the positive dislocations in the arms corresponding to B will move in preference to the negative dislocations in the arm represented by A.

![Fig. 23. Dislocation model for 'rosette' produced by micro-indentation (S = screw dislocations \(\{\{100\}\} \) directions or \(\{\{110\}\} \) directions, \(\{\{10\}\} \) directions or \(\{\{110\}\} \) directions).](image)

In experiment (3) it has been possible to analyze the deformation around an indentation in LiF. Branches of dislocations are revealed along certain crystallographic directions, and reference to the results of experiment (2) determines whether these are of screw or edge character. The application of a compressive stress then enables subsequent movement of some of the edge dislocations to be followed. Moreover, the selective tendency of this movement provides the basis for deducing the sign of the edge dislocations introduced by the indentation. The information obtained is summarized in Fig. 23.

(4) Recovery Behaviour

This experiment demonstrates the effect of temperature on dislocation distribution during so called recovery processes. Recovery involves the annihilation of many
dislocations, when dislocations of opposite sign cancel each other out on being brought to coincide. The remaining dislocations then arrange themselves into groupings of minimum energy, taking the form of sub-boundaries within the crystal.

Compress three specimens by 2%, in the direction of their longest axis with the 2.5 x 12 mm faces horizontal; approximately 2 complete turns of the vice screw will produce the desired amount of deformation. Keep one specimen for reference purposes and anneal the other two for 1/2 hour in air at 400°C and 600°C, respectively, allowing them to cool down in the furnace.

Comparison of the birefringence effects in each crystal (by means of transmitted polarized light with cross polars as previously specified) will show that the internal stresses produced by the deformation have been reduced by the anneal at 400°C and almost completely removed by the 600°C anneal. This is due to recovery and if the specimens are now etched it is obvious that recovery is accomplished by both a reduction in the dislocation density, and a rearrangement of dislocations. Note that etch pits will be rather indistinct after annealing because of precipitation of impurities on to the dislocations.

A compressive or tensile stress, of suitable magnitude, applied to a crystal generates approximately similar numbers of dislocations of each sign. Now, when two dislocations of opposite sign are brought into superposition, they exactly cancel or annihilate each other, and the lattice is restored to its original regular state.

The application of temperature allows dislocations to move together and to achieve this annihilation. Screws move by the process of slip on appropriate planes. This slip occurs under the action of the residual elastic stresses around the dislocations, when the dislocations become somewhat freed by the rise in temperature.

Edge dislocations move both by slip and climb, the latter being a diffusion controlled process. Climb involves the movement of vacancies to or from the ends of the half planes. Vacancies are unoccupied sites in the lattice; at elevated temperatures dislocations act as 'sources' or 'sinks' for vacancies and the half planes may thus climb up or down.

Fig. 24. Annihilation of two edge dislocations of opposite sign by a combination of slip and climb.
The mutual destruction of two edge dislocations of opposite sign by a combination of slip and climb is illustrated in Fig. 24. The negative dislocation at A has climbed 'down' a distance of $a'$ by the removal of material from the half plane, and slipped a distance $a''$ to reach the position A'. Correspondingly, the positive dislocation at B' has climbed 'up' a distance $b'$, and slipped a distance $b''$. A' and B' are coincident and the half planes are aligned so that the lattice is quite regular, the positive and negative dislocations disappearing.

![Diagram showing the destruction of two dislocations](image)

**Fig. 25. Formation of a low-angle tilt boundary during annealing. The edge dislocations originally in their slip planes (left hand view) have, by slip and climb, aligned themselves in the stable wall (right hand view).**

Generally, therefore the bulk of the dislocations cancel each other out during full recovery. There will, however, be some excess dislocations of a given sign which will form stable groupings of low energy. Edge dislocations do this by a combination of slip and climb, to form stable walls as in Fig. 25. The result, a set of edge dislocations aligned perpendicular to their Burgers vectors, constitutes what is known as a low-angle tilt boundary. Such boundaries and other more complex forms will be visible in the annealed specimens. It will be remembered that sub-boundaries are found in the as-received material, forming during crystallisation. However, those introduced during recovery are distinctly more numerous.