

PLASTIC PROPERTIES OF IONIC CRYSTALS

Thesis Submitted in Supplication

for the

Degree of Doctor of Philosophy

by

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August, 1962.

## SYNOPSIS

The plastic bending behaviour of NaCl, LiF and in particular MgO is investigated at room temperature, and that of MgO at temperatures up to 1550°C.

In all three materials the deformation is accomplished by the motion of dislocations, which, relative to the beam geometry are classified in two groups: two dimensional slip and three dimensional slip. Dislocations in the two dimensional classification all have Burgers vectors with a component in the beam cross-section normal to the neutral surface, while the Burgers vectors of those in the three dimensional classification have no component normal to the neutral surface

Beams which deform with slip in the three dimensional mode work harden much more rapidly than beams which deform in the two dimensional mode alone. A model for the geometry of three dimensional slip is postulated and the stresses in the cross-sections of beams arising from slip in this mode are examined photoelastically.

The factors effecting the formation and the predominance of slip in one mode or the other are discussed on the basis of the experimental observations. It is shown qualitatively that the stresses predicted on the basis of the model for three dimensional slip correspond to those observed photoelastically and that the greater rate of work hardening associated with this slip mode is the result of large elastic strains.

For the materials investigated it is concluded that the orientation of the slip is the most important single factor influencing the work hardening rate of single crystal beams.

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## INTRODUCTION

As the use of ceramic materials becomes more wide spread in many fields of present day technology, the need for a better understanding of their properties and the means by which these may be controlled assumes an ever increasing importance. Although it is unlikely that the use of large single crystals as mechanical components will ever become common practice, an understanding of the fundamental mechanisms which govern their plastic properties, and in particular their strength, will undoubtedly contribute significantly to the understanding of the more complex problems associated with the plasticity of polycrystalline materials. It was with this in mind that the investigations of the plastic properties of MgO reported here were embarked upon.

Some of the factors which influenced the selection of MgO as a suitable material for the proposed experiments were first of all the simplicity of its structure and slip systems, which are both the same as those of rock-salt, and its ductility at room temperature in spite of its very high melting point. Additionally it was hoped that some analogies might be drawn between its properties and those of other materials of the same structure on which extensive work had previously been done by numerous authors.

The first report of the plastic deformation of natural single crystals of MgO was made by Mügge(1920). He showed that under the combined influence of a hydrostatic confining pressure and a superposed compressive stress that MgO deformed by (110)  $[1\bar{1}0]$  slip. Although some work on natural crystals was reported in the intervening time by Bridgman (1937), it was not until 1954 that Wachtman and Maxwell reported that single crystals grown by the Norton Company could be deformed plastically. However, they found it necessary to raise the testing temperature to approximately 1100°C in order to obtain plastic flow in specimens without the added constraint of a high confining pressure.

Subsequently, Gorum, Parker and Pask (1958) showed that single crystals of the Norton material could be plastically strained

as much as 20% at room temperature, providing the condition of the specimen surface was properly controlled. Since then extensive investigations of the plasticity of MgO have been carried out by many authors, and much of what has been published has had a significant influence on the course of the work reported here.

Throughout the experimental work, bending was the method used to deform the specimens, and the tests were carried out at both room temperature and temperatures up to 1550°C. In addition, where it seemed advantageous, room temperature bending tests were performed on LiF and NaCl in order to clarify some of the phenomena observed.

To carry out the experimental work it was necessary to design and build a number of pieces of apparatus and it is with this aspect of the work that the thesis begins.

CHAPTER ONEAPPARATUS AND EXPERIMENTAL TECHNIQUES1.1 Introduction

In order to investigate the plastic behaviour of magnesium oxide deformed in bending it was necessary to prepare specimen beams from fairly large blocks of the material. These were obtained from both the Norton Grinding Wheel Co. in England and from Semi-Elements Inc. in the United States. All beams were cleaved to size, some were polished and etched and all were bent in standard bending jigs loaded in either room temperature compression machines or a high temperature furnace fitted with compression apparatus.

To clarify some of the phenomena observed in the bending of MgO it proved advantageous to perform similar bend tests on beams of lithium fluoride and sodium chloride. These beams were also prepared by cleavage, the LiF from blocks obtained from the Harshaw Chemical Co., and the NaCl from large crystals grown by Davidge in the apparatus described by Newey (1959). The NaCl crystals reported here were doped with Ca to give a resolved flow stress of  $200\text{g/mm}^2$ . Some of these beams were polished and etched and all were deformed in the same bending apparatus as the beams of MgO.

1.2 Cleaving Machine

NaCl, LiF and MgO can all be cleaved on the  $\{100\}$  planes by hand using a chisel and hammer, however, this method usually introduced significant plastic strain into the beams of NaCl and LiF and proved to be most uncertain and generally unsatisfactory when used with MgO. Since many authors; e.g. Stokes, Johnson and Li (1959 and 1961); Gorum, Parker and Pask, (1958); Stearns, Pack and Lad, (1960); and Gilman (1959a), have reported the critical dependence of the plastic behaviour of such materials on the distribution and orientation of slip sources at the onset of plastic flow, it was deemed necessary to at least standardise the

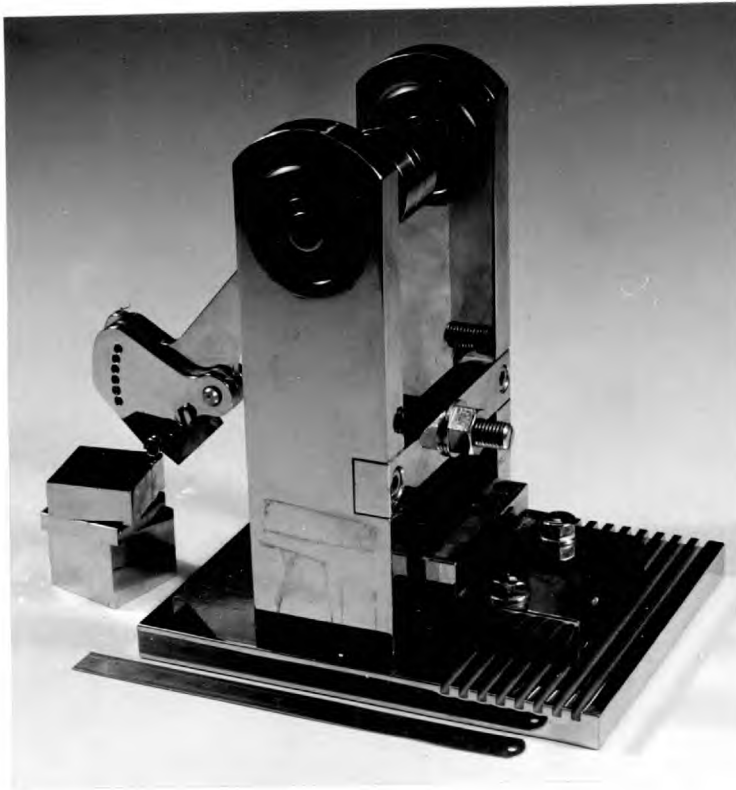


Fig 1. Pendulum-Type Cleaving Machine



Fig 2. The room temperature bending jigs shown with beams in position for loading; on the left the 3-point jig and on the right the 4-point jig.

cleaving process and in so doing to reduce to as low a level as possible the plastic deformation accompanying the cleavage. As a result of these considerations the pendulum type cleaving machine shown in Fig I. was designed and built. It is made of mild steel throughout except for the actual cleaving blade which is a tough tool steel hardened to about 62 Rockwell C and tempered.

As the angle at which the blade strikes the face or edge of the crystals is important the blade head was designed so that it could be set at  $2^{\circ}$  intervals over a total range of  $30^{\circ}$  by inserting a pin in the appropriately aligned holes. The stop against which the crystal is placed could be positioned at  $\frac{1}{4}$  inch intervals over a total travel of 2 inches. The screw bumper to arrest the pendulum arm was continuously adjustable and could be locked at any position with a nut, and the blade could be removed readily for sharpening or replacement by the removal of two screws. In practice it proved advantageous to place a cylindrical half-round between the crystal block and the back-stop with its radial plane in the plane of the knife edge when large blocks were being cleaved. This permitted the halves of the block to rotate freely away from the blade during cleavage and resulted in easier and cleaner cleavages.

It was found that a blade ground symmetrically to an included angle of  $30^{\circ}$  as suggested by Gilman (1959b) was most satisfactory for the cleaving of NaCl and LiF and for the final cleaving of the MgO beams, but a blade ground to  $40^{\circ}$  proved more satisfactory when first cleaving large blocks of MgO into smaller blocks and plates. With practice, very good cleavages could be made and the plastic deformation accompanying the cleavage could be limited to the immediate region of the blade impact. With the exception of the blades, all parts of the machine were chromium plated to prevent corrosion.

### 1.3 Room Temperature Bending Jigs

#### (a) Four-Point Bending Jig

The four-point bending jig used at room temperature, pictured in Fig 2., was machined out of stainless steel with the knife-edges ground

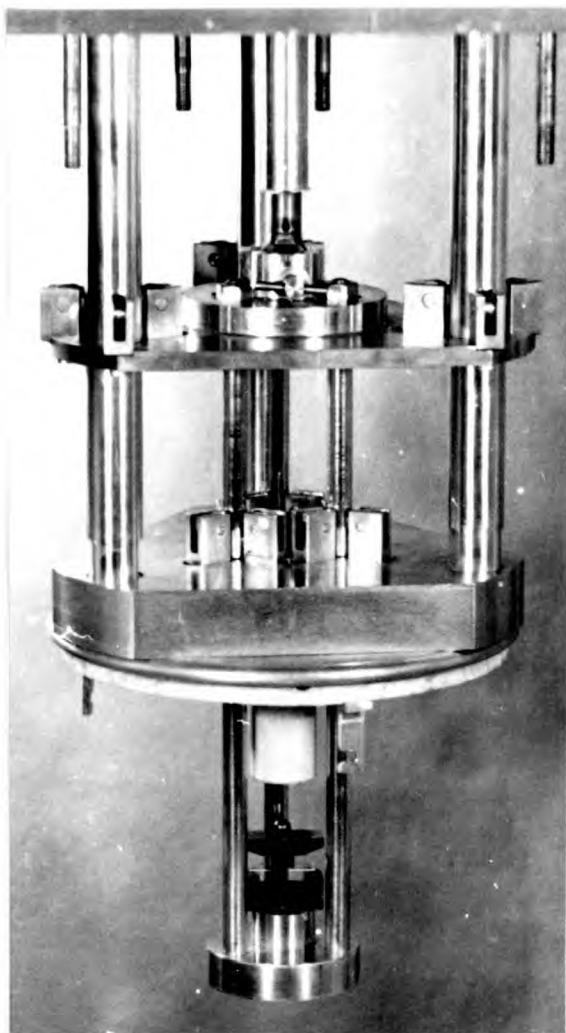


Fig 3 The modified Adams machine showing an overall view of the machine with associated recording and control apparatus, and a detailed view of the compression cage.

to  $90^{\circ}$ . The sharp corners of the knife-edges were broken slightly with very fine emery paper and were polished with  $\frac{1}{2}\mu$  alumina polishing compound. The inner knife-edges were guided onto the specimen with posts set into the block of the outer knife-edges. A good sliding fit was attained by grinding the posts in with the  $\frac{1}{2}\mu$  alumina and then lubricating with graphite applied with a soft pencil. This provided a sliding fit with extremely little play and virtually no friction.

The knife-edges could be driven together by any machine with two reasonably parallel plates capable of applying a compressive load. In actual testing the block of the outer knife-edges was placed flat on one compression plate. The other plate applied the load to a small flat, ground on a  $\frac{1}{4}$  inch steel ball, which in turn fitted into a graphite lubricated recess ground into the top centre of the inner knife-edge block.

#### (b) Three-Point Bending Jig

The three-point bending jig pictured in Fig 2. differed from the four-point jig only in that it had a single central knife-edge which was ground to an included angle of  $60^{\circ}$ . Its preparation and use were identical to that described above for the four-point jig.

### 1.4 Room Temperature Testing Machines

All the bend tests carried out at room temperature were performed in either a commercially available table model Instron, or in a hollow lead screw machine obtained from U.K.A.E.A. Harwell. The Instron is mechanically and electrically comparable to the larger machine described by Hindman and Burr (1949) and was fitted with the compression jig originally designed by Pratt (1951) and modified by Newey (1959). The hollow lead-screw machine was originally conceived by Adams and shall hereafter be called the Adams machine. Substantial modifications to this machine proved necessary before it could be used and suitable results obtained.

The modifications to the Adams machine consisted of removing

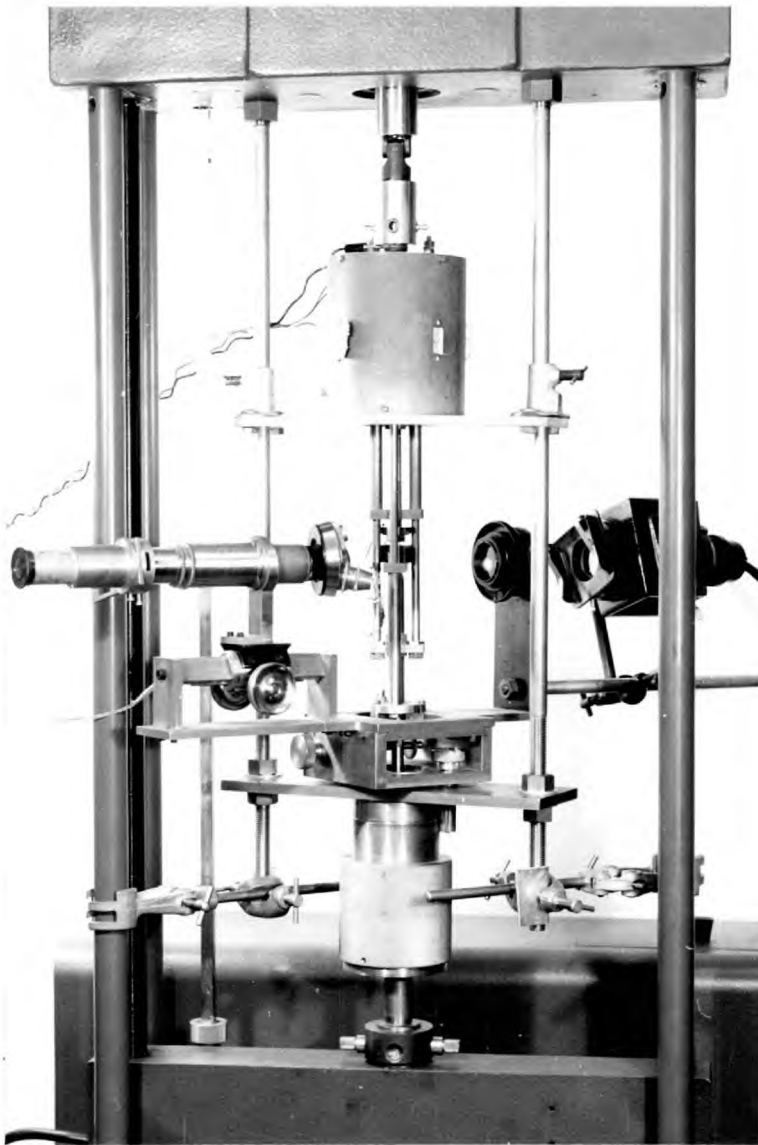


Fig 4. The bending jig mounted in the compression cage in the Instron with the polariscope in position. The polariscope may be raised or lowered and rotated about the jig by approximately  $100^{\circ}$ .

the load cell, the tensile testing jig, and the driving plate, and replacing all of these with completely redesigned apparatus. It was considered advantageous to reconstruct the machine as an open-ended compression machine so that cooling or heating apparatus could be readily fitted and removed. A general view of the entire modified machine with a detailed view of the loading cage is provided in Fig 3.

A Langham Thompson type U.F.2. dynamometer of 100 pound total capacity was used in place of the one ton proving ring originally in the machine, the dynamometer being hung on a universal coupling inside the closed end cylinder at the top of the machine. The dynamometer supported a rod which was coupled to the top cage plate by another universal. The top cage plate was positioned by three adjustable ball bearings running on each of the three loading rods passing through it. In turn the top cage plate supported the three cage rods which passed through holes in the main loading plate. Each of these rods was positioned relative to the loading plate by three more adjustable ball bearings, and finally supported the bottom cage plate which rested on shoulders turned on the end of each of the rods.

The main loading plate could be driven down at any rate from about 0.001 to 0.100 in/min. The speed of the driving motor could be varied continuously over a useful range of a factor of ten. The additional factor of ten variation was accomplished with a change in the driving gear ratio.

The input voltage to the dynamometer was adjusted so that when it was switched to four preset values, an output of 5mv at loads of 5, 10, 25, and 50Kg was obtained. This output was applied to a Honeywell recorder with a full scale deflection at 5mv, through a Cambridge potentiometer. At zero applied load the recorder scale could be accurately adjusted to zero with the potentiometer, and by using it to back off known increments of the dynamometer output the potentiometer could be used to broaden the recorder scale without changing its calibration.

Towards the end of the experimental work reported, a polariscope designed by Davidge was fitted to the Instron permitting birefringence

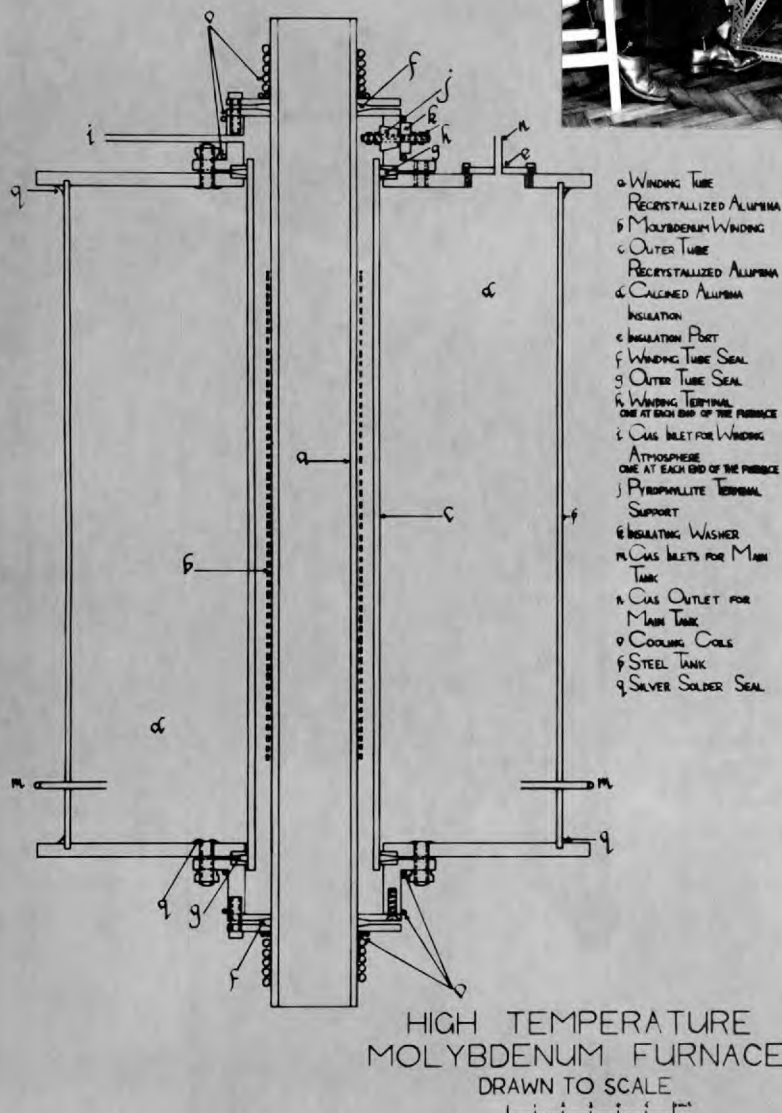


Fig 5. Overall view of the high temperature testing facility with a detailed section of the furnace.

observations to be made during the tests. It can be seen in Fig. 4, which in addition shows the bending jig mounted in the compression cage. It proved possible to use this polariscope on the Adams machine as well with very little modification.

## 1.5 High Temperature Testing Machine

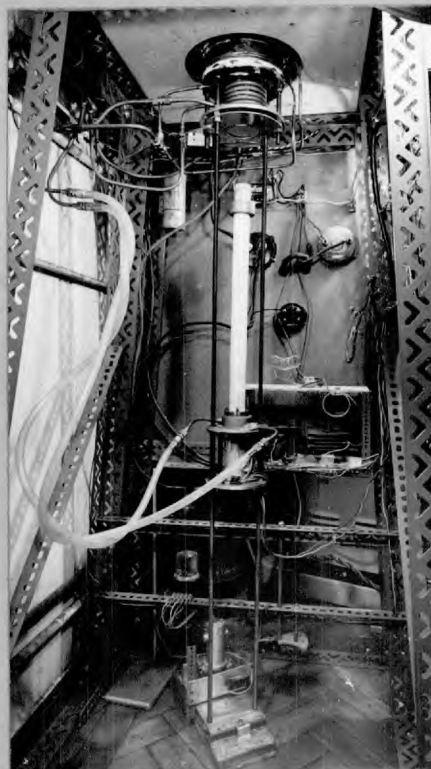
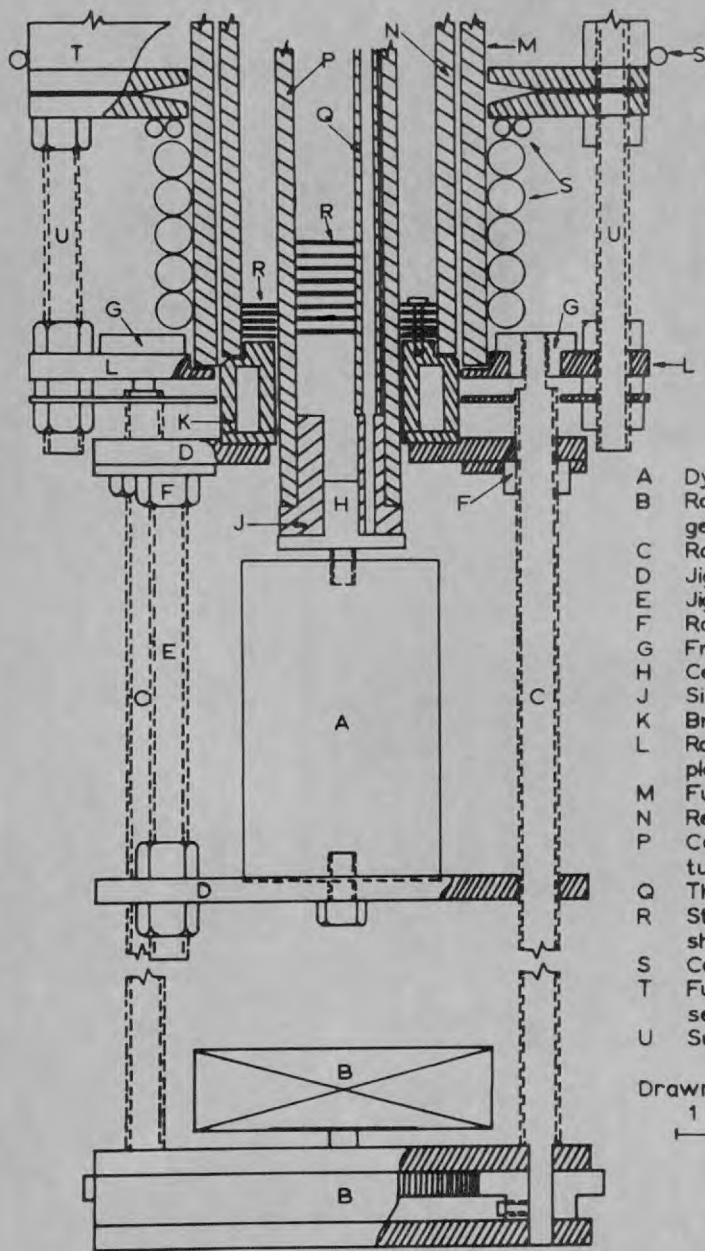
### (a) High Temperature Furnace

To provide a facility for the testing of MgO at high temperatures a large molybdenum wound furnace was built. It is illustrated in Fig 5. along with an overall view of the complete apparatus. Its principal feature of interest was that the recrystallized alumina tube on which the molybdenum tape was wound was separated from the calcined alumina insulation by a second larger diameter alumina tube. This larger tube completely sealed off the main insulation tank rendering it necessary to control the atmosphere only between the two tubes to prevent oxidation of the winding. The winding tube was supported and sealed at both ends of the furnace by two flanges belted to the end bells. The material used in the seals was asbestos cord impregnated with graphite and silicon grease.

Though it was not necessary to control the atmosphere within the main insulation tank at low temperatures, it was purged with 25%  $H_2$  + 75%  $N_2$  gas so that at higher temperatures, when the outer tube might well become permeable to the gasses in the tank, oxidation of the winding would not occur.

The primary reason for using this concentric tube construction was to simplify the removal of the heating tube for rewinding by obviating the moving of the entire tank and the removal of all the insulation. This advantage was considered important in the light of all the auxiliary equipment aligned with and fastened to the furnace and proved a worthwhile feature when the winding had to be replaced after open circuiting during a power failure.

Cooling coils were wound on the ends of the winding tube and were soldered to the end bells and sealing flanges to prevent the gas



- A Dynamometer.
- B Raising motor and gear box.
- C Raising lead screws.
- D Jig plates.
- E Jig rods.
- F Raising nuts.
- G Friction thrust bearings.
- H Cell support stud.
- J Silicon nitride plug.
- K Brass cooling collar.
- L Raising jig supporting plate.
- M Furnace winding tube.
- N Reaction tube.
- P Compression cell support tube.
- Q Thermocouple tube.
- R Stainless steel heat shields.
- S Copper cooling coils.
- T Furnace end bell and sealing plates.
- U Supporting studs.

Drawn to scale.  
1 inch.

Fig 6. The mechanical apparatus for raising and supporting the compression cell at the bottom end of the furnace.

seals from becoming too hot and breaking down. The ends of the winding tube were closed with hollow brass caps to which were affixed stainless steel radiation shields and through which water was circulated. These were of prime importance in preventing the auxiliary equipment at both the top and bottom of the furnace from overheating.

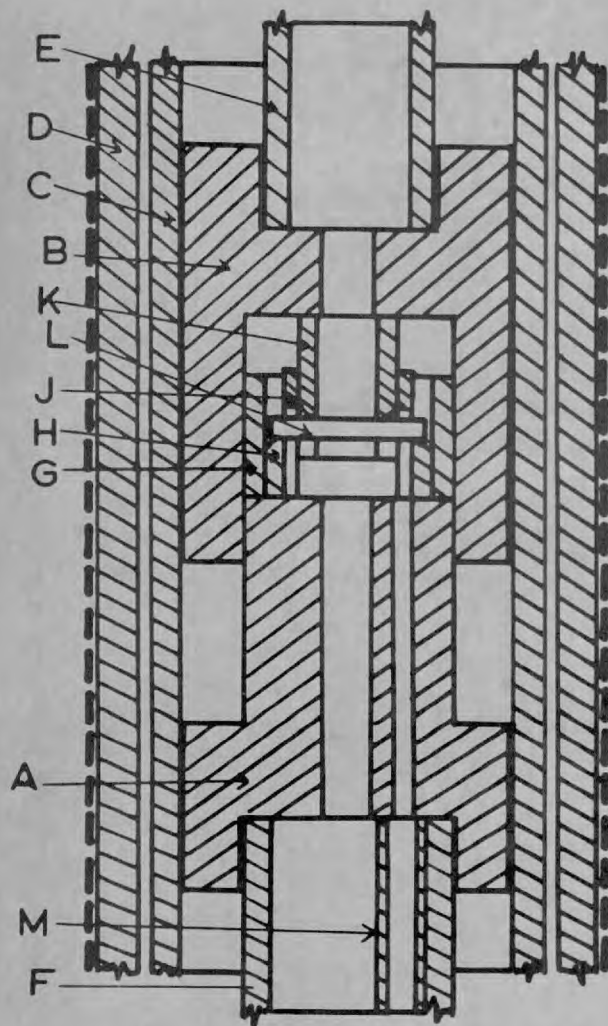
The power was supplied by two 20 amp Variacs in parallel, and the temperature controlled by an Ether proportional temperature controller calibrated for operation with a 20% Rh 80% Pt -- 5% Rh 95% Pt thermocouple and used to switch a small load in and out of the primary side of the Variacs. Both the current and voltage were metered on the secondary side of the Variacs and at equilibrium only 2.5Kw was needed to maintain the temperature at 1550°C.

#### (b) Mechanical Apparatus

To raise the specimens and test cell into the furnace a double lead screw racking mechanism was hung from friction bearings in a plate supported by four studs in the bottom end bell. The lead screws were rotated by a reversible motor driving them through a gear box which can be seen at their bottom end in Fig 6. The cage, which supported the dynamometer and hence the compression cell, rested on nuts threaded onto the lead screws. The nuts fitted into hexagonal holes cut in a brass plate bolted to the under side of the upper cage plate. If these nuts were engaged in the holes, the cage was raised or lowered by them depending on the direction of rotation of the lead screws.

The lead screws passed through close tolerance holes in the bottom cage plate, thereby keeping the cage in line with the centreline of the furnace with which the lead-screws were aligned. The dynamometer was bolted to the bottom cage plate and a steel stud with a wide shoulder turned on it screwed into its top. A silicon nitride tube support fitted over this stud, resting on its collar, and supported the recrystallized alumina tube on which the silicon nitride compression cell rested.

A thermocouple, for measuring temperatures at the beam inside the compression cell, passed through the steel collar, the silicon nitride



- A Piston.
  - B Cylinder.
  - C Reaction tube.
  - D Furnace tube with winding.
  - E Driving tube.
  - F Support tube.
  - G Spacer cylinder.
  - H Outer knife edge cylinder.
  - J Spacer cylinder.
  - K Inner knife edge cylinder.
  - L Specimen beam.
  - M Thermocouple tube.
- Drawn to Scale.

1 inch.



Silicon Nitride.



Recrystallized Alumina.

Fig 7. The silicon nitride compression cell is shown at top right, and the alumina bending jig is shown dismantled on its left. The cell is shown completely assembled inside the furnace in the detailed drawing.

tube support, up a small bore alumina tube within the larger supporting tube and through the bottom of the compression cell. The small bore tube in addition served as a support for stainless steel radiation shields attached near its lower end.

The brass cooling ring with its radiation shields attached was clamped to the top of the upper cage plate so that when the cage was raised, and the compression cell fully racked into the furnace, the cooling ring fitted into and essentially sealed off the end of the winding tube. It is illustrated in this position in Fig 6. The recrystallized alumina tube supporting the compression cell passed freely through the centre of the cooling ring. When a reaction tube was used inside the winding tube, as in all the tests reported here, it rested on the cooling ring during the test, but was not raised into and lowered out of the furnace with every specimen. Between actual tests it rested on a sliding aluminium plate used to close off the end of the tube when the cell was withdrawn.

The compression cell, shown in Fig 7., consisted of a piston and cylinder of silicon nitride machined to close tolerances by B.S.A. Its outside diameter was just small enough to allow free sliding inside the alumina reaction tube at room temperature. The reaction tube kept the parts of the cylinder axially aligned, thereby reducing friction between the two parts to as low a level as possible and ensuring that the load was axially applied. The actual bending of the beams was accomplished in a small four-point bending jig made of concentric alumina cylinders with bevelled slots cut in their walls. These cylinders were ground to diameters giving as good a fit as possible yet allowing free slip.

To perform a test, the alumina cylinders were assembled with the beam in position, and placed on top of the silicon nitride piston. The cylinder was placed over this entire assembly and the complete unit jacked into the furnace. The limits of travel of the racking mechanism were controlled with adjustable microswitches in the motor circuit.

At the top of the furnace a steel plate standing on four studs in the end bell supported the complex cantilever beam on a ball bearing mounted shaft as illustrated in Fig 8. The beam which acted as the loading lever was driven down at its long end and applied the load to the

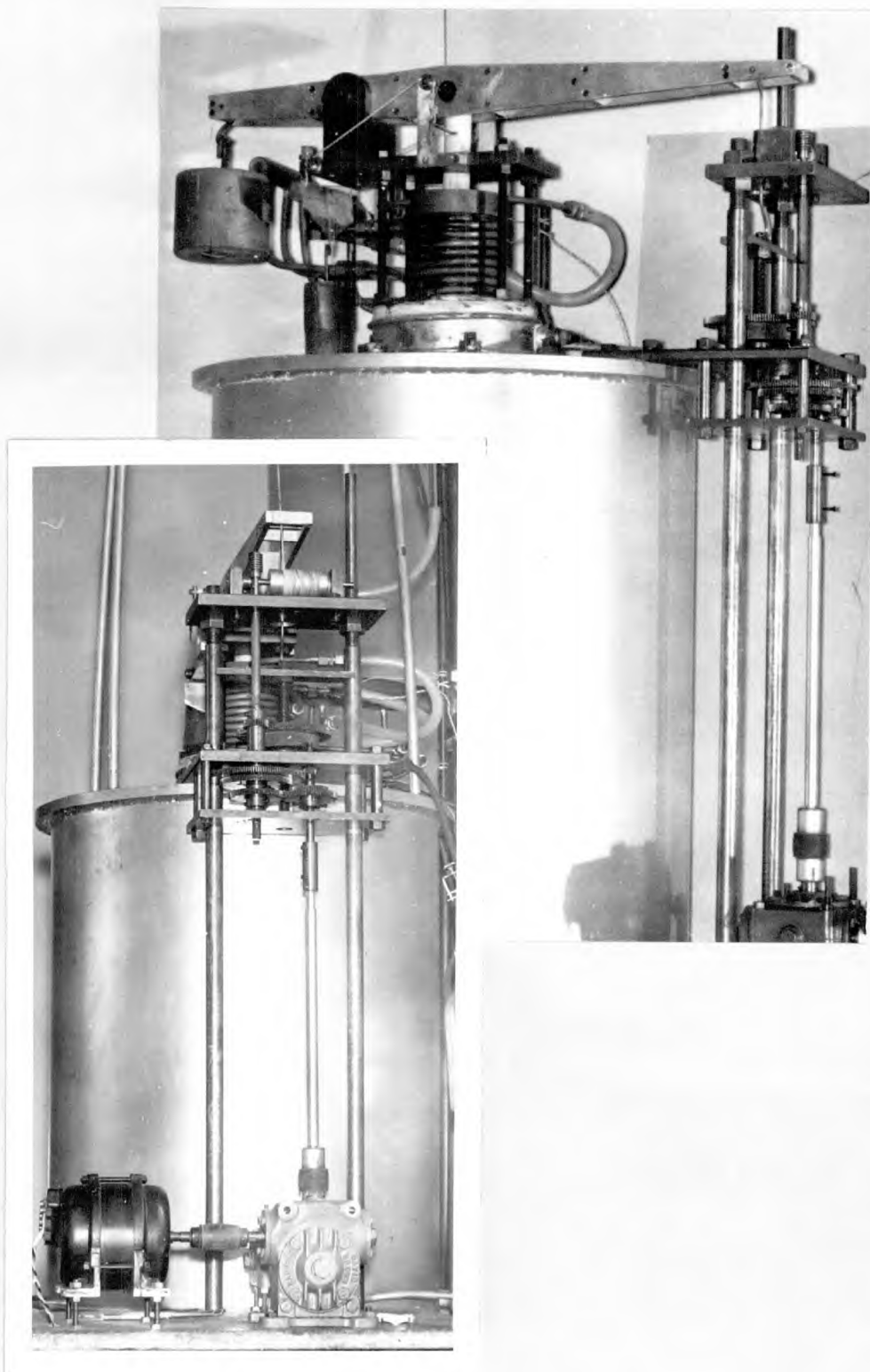


Fig 8 The loading mechanism and drive showing details of the top of the furnace.

loading tube with a mechanical advantage of four to one. The load was actually applied by a  $\frac{3}{4}$  inch steel shaft ground flat on one side to a diametral plane and supported by ball bearings mounted in the sides of the beam. This flat rested on a silicon nitride plug which fitted into the end of the alumina loading tube which in turn passed through a hole in the brass cooling collar. The bottom end of the loading tube engaged in the recess turned in the top of the compression cell cylinder. Hence when the end of the loading beam was drawn down the knife-edges were driven together at  $\frac{1}{4}$  the driving rate applied to the beam end. Both the beam and the loading tube were counterbalanced.

A gear box with reduction ratios of 1:1, 1:2, and 1:8 was built to drive the final reduction gear which rotated a threaded collar, fixed in a thrust bearing, about the lead screw thereby driving the lead screw up or down. The lead screw was coupled to the end of the beam with a rod. This gearbox was driven by a reversible synchronous motor operating through two more fixed ratio gear boxes which gave a net reduction of 1:1500. It was possible with this arrangement to obtain strain rates roughly the same as those used in the room temperature testing machines.

The machine could be converted to a soft machine simply by substituting a spring of an appropriate force constant for the rod connecting the lead screw to the end of the beam. In this modification strain could be measured with a sensing rod passing through the central hole of the compression cell and driving the core of a differential transformer substituted for the dynamometer. However, this modification was not used for any of the tests reported here.

The power supply for the dynamometer and the recording of its output were exactly the same as described for the Adams machine, but in addition the temperature within the compression cell was continuously recorded throughout each test on a Honeywell recorder. This provided a record of the heating and cooling rates during the introduction and removal of each specimen as well as a record of the temperature during the actual bending.

## 1.6 Microscopes

Both a table model Reichert and a Reichert Zetopan microscope were used for after-test examination of the deformed beams. Both could be used with either transmitted or reflected illumination, but most of the birefringence observations were made on the Zetopan as it was fitted with a tint plate, and it proved much easier to add  $\frac{1}{4}$  wave plates to its optical system than to that of the table model Reichert.

## 1.7 Polishing and Etching Techniques

### (a) MgO

To remove any damage to the surface of the cleaved MgO the beams were polished in an 80% aqueous solution of orthophosphoric acid at 150°C. Two to three minutes were normally sufficient to remove enough surface to eliminate fresh dislocations and any microcracks at the beam edges introduced during cleavage.

The etchant used to reveal dislocations was a solution of five parts saturated ammonium chloride, aqueous solution, five parts concentrated sulphuric acid and two parts distilled water with an extremely small addition of ferric chloride, sufficient to give the etch a faint yellow tinge. During etching the solution was gently stirred on a rotating sloping platform. An etch of four minutes proved sufficient to produce well defined pits at fresh dislocations and shallow diffuse pits at grown-in dislocations.

### (b) LiF

A solution of one part HCl to three parts glacial acetic acid was used to polish beams of LiF, and etching was carried out in hydrogen peroxide in which a small quantity of ferric chloride had been dissolved. The addition of  $\text{FeCl}_3$  increased the etching rate substantially, but appeared to have little other effect.

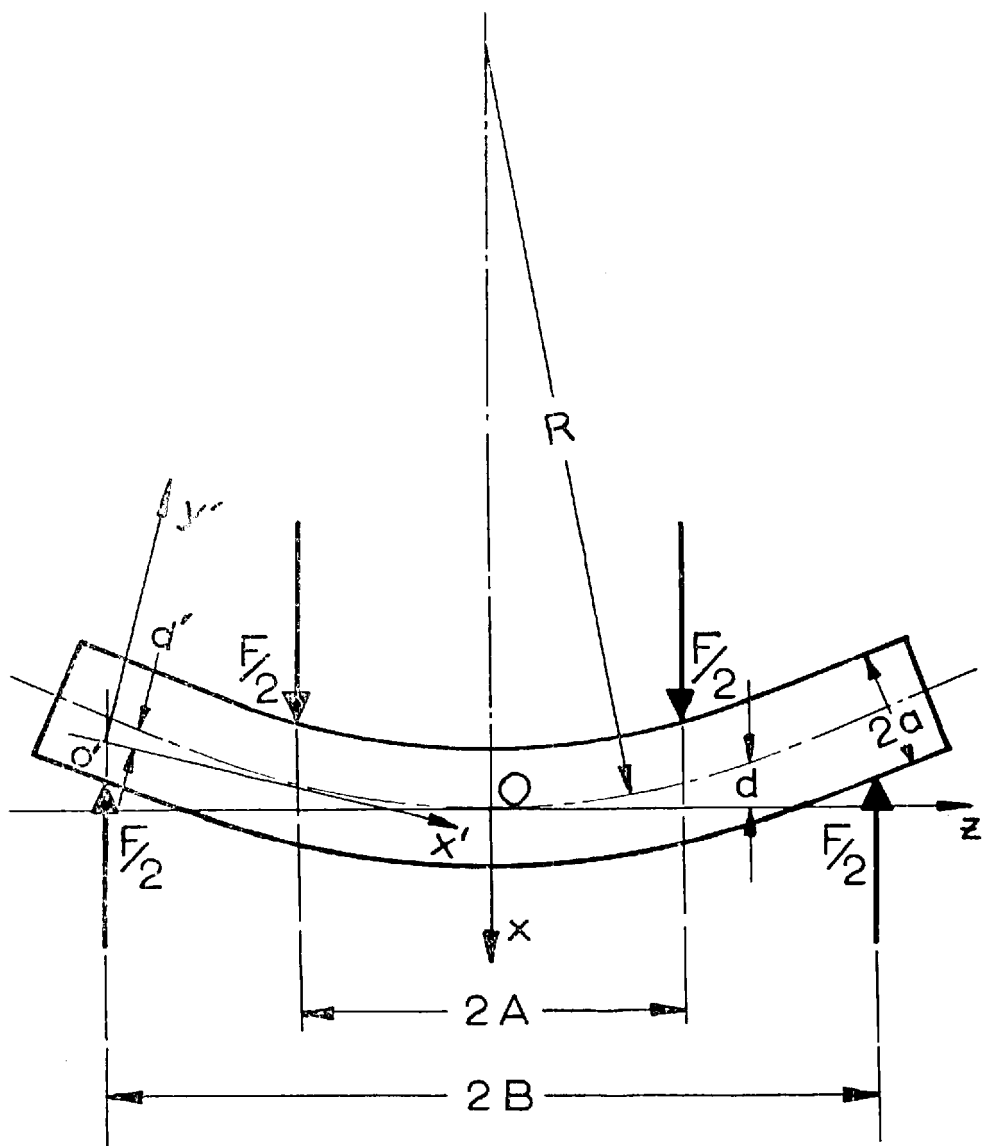


Fig.9 Schematic diagram of a beam undergoing four-point bending.

(c) NaCl

The NaCl beams were polished in a solution of one part methanol in three parts ethanol, and were etched in a solution of one part methanol in three parts glacial acetic acid.

1.8 Calculation of Strain

The record produced of a bend test by all three of the loading machines described above was a graph of the load applied vs. the displacement of the inner knife-edges. A calculation of the stress in the beam made on the basis of simple beam equations, is not valid once the beam has started to deform plastically. Read (1957) has shown that the stress at any point in the beam becomes a more complicated function of both the load, and the stress necessary to nucleate and to move dislocations. Consequently, the stress was not used in the plots of the characteristic bending curves; the load was simply reduced to the ratio, the load upon the load applied at the macroscopic flow stress. However, reasonably accurate calculations of the outer fibre strain can be made up to strains of approximately 5% by applying the simple beam equations of Timoshenko (1934). Strictly, these equations apply only to elastically deformed beams, but the error involved in their application to beams deformed plastically in four-point bending up to about 5%, proved to be insignificant.

To make a calculation of beam strains for four-point bending, consider the beam located in the four-point bending jig with its long axis perpendicular to the knife-edges; see Fig 9. The two systems of rectangular co-ordinate axes were located so that the origin O was in the neutral axis of the beam midway between the inner knife-edges, while the O - x axis was superimposed on the long axis of the beam and remained tangential to the neutral axis at the origin O, when the beam became bent. The O' - x' axis was made tangential to the neutral axis at the vertical plane of the inner knife-edge and perpendicular to the knife-edges, while the origin O' was located in the vertical plane of the outer knife-edge.

Let the displacement of the neutral axis relative to the O' - x' axis be  $d'_c$  at  $x' = c$  for values  $0 < x' \leq (B-A)$  and let the total displacement

of the neutral axis from the  $O - z$  axis be  $d_o$  at  $z = c$  for values  $-B \ll z \ll B$  where  $2B$  is span of outer knife-edges and  $2A$  is span of inner-knife edges.

Then the displacement of the inner knife-edges relative to the outer knife-edges is given by :

$$D = d_B - d_A \quad (1)$$

Now the central portion of the beam  $-A \ll z \ll A$  experiences pure bending with a moment,  $M$ , given by

$$M = F/2 (B-A) \quad (2)$$

where  $F/2$  is the force exerted on each knife-edge. This moment induces a curvature in this region of radius

$$R = EI/M \quad (3)$$

where  $E$  is Young's modulus in  $[100]$  and  $I$  is the moment of inertia of the beam cross-section about the neutral axis.

At  $z = A$ , the slope of the neutral axis is  $A/R$ . Hence the slope of the  $O' - x'$  axis relative to the  $O - z$  axis is  $-A/R$  and the displacement of the origin  $O'$  from the  $O - z$  axis is  $(B-A)A/R + d_A$ . Therefore

$$d_o = (B-A)A/R + d_A + d_o' \quad (4)$$

It follows from the expression for the displacement of a simple cantilever (Timoshenko 1934) that :

$$d_{o'} = \frac{F(x')^3}{12EI} - \frac{F(B-A)^2 x'}{4EI} + \frac{F(B-A)^3}{6EI} + \frac{F(2a)(B-A-x')}{16GI} \quad (5)$$

where  $2a$  is the depth of the beam and  $G$  is the shear modulus in  $[100]$  and is approximately  $\frac{1}{2}E$  in  $[100]$ .

Therefore at  $x' = 0$

$$d_{o'} = \frac{F(B-A)^3}{12EI} + \frac{F(2a)^2(B-A)}{16GI} = \frac{1}{R} \left( \frac{(B-A)^2}{3} + \frac{4a^2}{3} \right) \quad (6)$$

by substitution of equations (2) and (3).

Substituting (6) in (4) and the resulting expression in (1) gives :

$$D = \frac{1}{R} (A(B-A) + \frac{(B-A)^2}{3} + \frac{4a^2}{3})$$

which gives the expression for the radius of curvature of the central portion of the beam :

$$R = \frac{1}{D} (A(B-A) + \frac{(B-A)^2}{3} + \frac{4a^2}{3}) \quad (7)$$

Consequently, knowing the jig dimensions, the beam depth and the displacement of the inner knife-edges relative to the outer knife-edges, one can compute the outer fibre strain,  $\epsilon$ , from the expression :

$$\epsilon = a/R$$

By substituting equations (2) and (7) in equation (3) it was possible to calculate values of Young's modulus in  $[100]$ . The values for  $E$  obtained experimentally were in good agreement with other reported values. Making this calculation provided a convenient check to determine whether the beam had initially been in contact with all four knife-edges. If the beam were initially in contact with only one of the inner knife-edges owing to surface irregularities of the crystal, the calculated value was very much smaller than the expected value. Acceptable values of  $E$  were often obtained in such cases when calculated on the basis of three-point bending for the jig. With the fitting of the polariscope to the testing machines this check was rendered unnecessary.

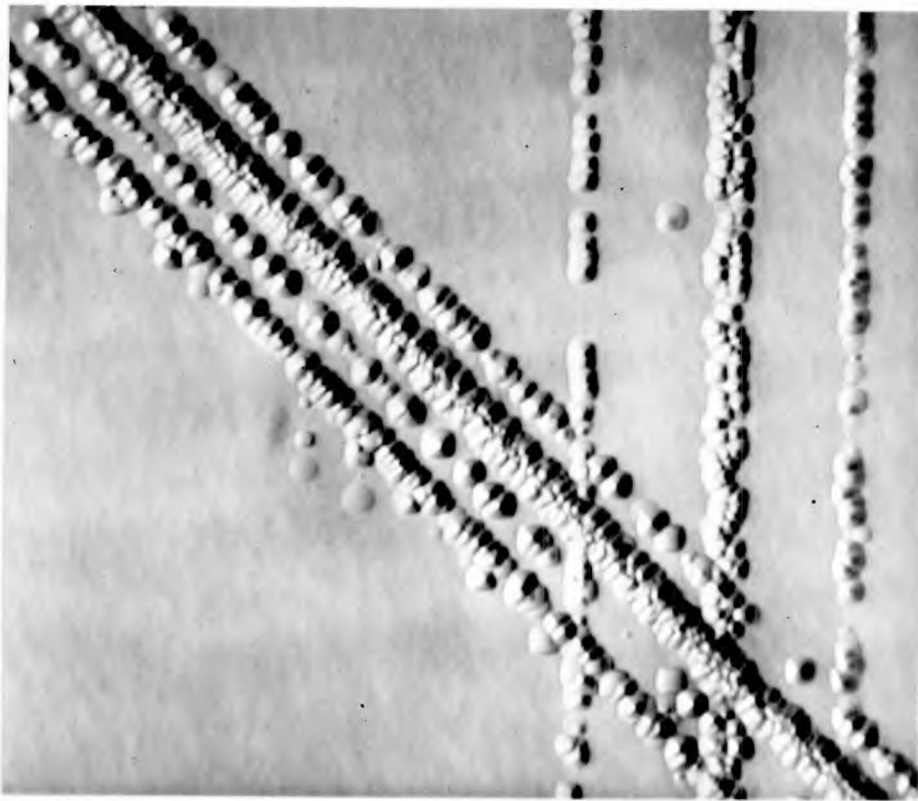


Fig 10. Etched bands of edge dislocations lying diagonally across figure and screw dislocations parallel with the figure side. Note asymmetry of screw pits. x850

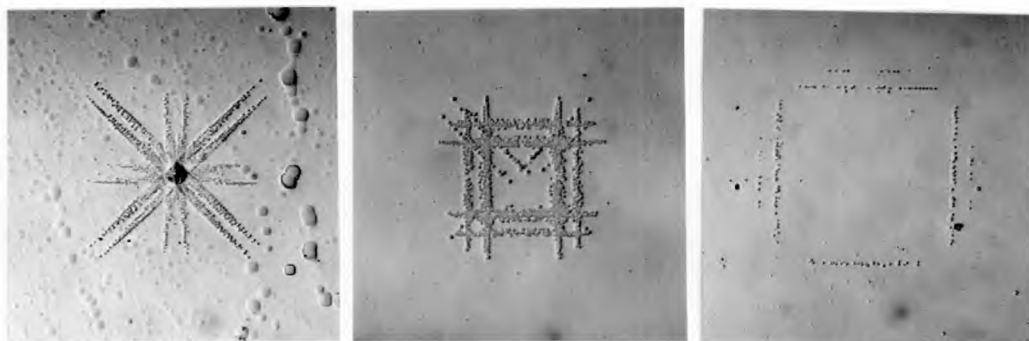


Fig 11. Successive stages in the polishing of a microhardness indentation. x 400.

CHAPTER TWODISLOCATION MOBILITIES AND BENDING MODESII.I Introduction

The slip systems that operate in MgO are the same as those that normally operate in both LiF and NaCl at room temperature, that is, slip occurs on all six planes of the class  $\{110\}$  in the directions  $\langle 110 \rangle$  or  $\langle \bar{1}\bar{1}0 \rangle$ , a total of twelve possible slip directions. These materials all cleave on their  $\{100\}$  planes giving simple orthorhombic specimens. Therefore when slip occurs in a cleaved specimen the traces of a slip plane are normal to the crystal edges on the two opposite faces and at  $45^\circ$  to the crystal edges on the other two faces. There will be slip steps along the traces normal to the crystal edges, whereas no change in the crystal surface should occur along the traces inclined at  $45^\circ$  to the edges.

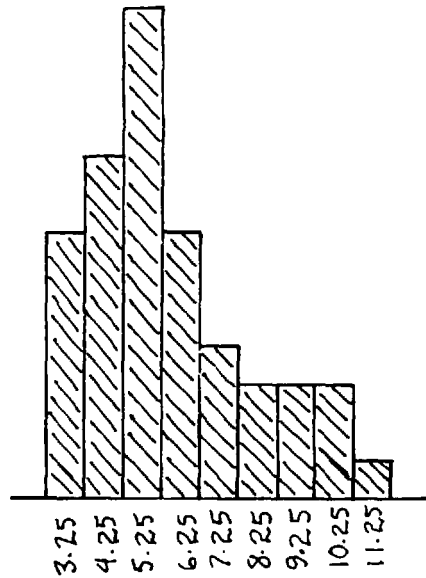
Etching reveals the ends of screw dislocations along the normal slip plane traces and the ends of edge dislocations along the  $45^\circ$  traces. Hence by observing the orientation of the etched slip traces it is very easy to distinguish between edge and screw dislocations. It often proved possible to distinguish between individual edge and screw dislocations as the etch pits at screw dislocations were usually ~~asymmetrical~~. This asymmetry is a result of the obliqueness at which the screw dislocations meet the surface. The edge dislocations, generally being normal to the surface, produce perfectly symmetrical pits. This difference in symmetry can be seen in Fig 10. There was very little evidence in MgO of the screw dislocations cross gliding out of slip bands to positions normal to the surface, thereby producing the dispersed glide bands observed by Gilman and Johnston (1957) in LiF.

When a beam is deformed in pure bending, before any dislocation motion occurs the maximum longitudinal stress is reached in the outer fibres at the tension and compression faces, this stress generally being a simple uniaxial stress. Both the edge and screw dislocations at the tension and compression faces have Burgers vectors with a component  $b/\sqrt{2}$  in the stress direction, and hence, both should be subject to the same stress

Test no. N1:8(e)

Resolved outer fibre stress: 4870 g/mm<sup>2</sup>

Duration of stress application: 15 sec.



Histogram of velocity distribution

| Distance Moved in 5 μ units | Number of Dislocations h | Mean Distance for group in 5 μ units d |
|-----------------------------|--------------------------|----------------------------------------|
| 2.5                         | 0                        |                                        |
| 3.0                         | 6                        | 3.25                                   |
| 3.5                         | 1                        |                                        |
| 4.0                         | 5                        |                                        |
| 4.5                         | 4                        | 4.25                                   |
| 5.0                         | 8                        |                                        |
| 5.5                         | 5                        | 5.25                                   |
| 6.0                         | 5                        |                                        |
| 6.5                         | 2                        | 6.25                                   |
| 7.0                         | 3                        |                                        |
| 7.5                         | 1                        | 7.25                                   |
| 8.0                         | 1                        |                                        |
| 8.5                         | 2                        | 8.25                                   |
| 9.0                         | 2                        |                                        |
| 9.5                         | 1                        | 9.25                                   |
| 10.0                        | 3                        |                                        |
| 10.5                        | 0                        | 10.25                                  |
| 11.0                        | 1                        |                                        |
| 11.5                        | 0                        | 11.25                                  |

Total Population,  $n = 49$

Mean for Histogram distribution

$$M = \frac{\sum_{d=3.25}^{11.25} h d}{n} = 5.82$$

$$N = \sum_d (h d^2) = 1854.0$$

$$\nu = n - 1 = 48$$

$$S^2 = \frac{1}{\nu} (N) - M^2 = 4.8$$

$$s^2 = \frac{n}{\nu} S^2 = 4.9$$

$$\therefore s = 2.21 \quad \text{and} \quad \frac{st}{\sqrt{n}} = \frac{(2.21)(2.01)}{7} = .64$$

where 2.01 is value of  $t$  with probability of 0.05 of being exceeded.

∴ .95 probability that mean lies between  $5.82 \pm 0.64$

∴ .95 probability that mean velocity is in the range  $1.94 \pm .21 \mu / \text{sec.}$

and move when a sufficient stress magnitude is reached. The velocity with which these two types of dislocations move will influence directly the amount of plastic strain accommodated by each, assuming there are many random sources of both orientations present.

In order to assess the probable slip pattern in beams undergoing pure bending deformation, experiments of the etch-load-etch variety, employed by Johnston and Gilman (1959) on LiF, and by Stein and Low (1960) on silicon iron, were carried out to determine the relative velocities of edge and screw dislocations in MgO.

## II.2 Dislocation Velocities in MgO.

Beams of MgO were cleaved to dimensions typically 1mm. deep by 1.5mm. wide by 20mm. long, and polished to remove any cleavage steps and surface damage. If an attempt was to be made to measure the velocity of screw dislocations, a number of very small indentations were made on one face of the beam. These were made with the micro-hardness tester on the Reichert microscope, or by sprinkling 45 mesh carborundum onto the surface from a height of about three inches as described by Stokes, Johnston and Li (1959(b)). The beams were polished again until etching revealed fresh dislocation loops only in the screw orientation in characteristic square patterns illustrated in Fig 11. At this stage polishing was continued carefully until the sides of many of these squares were reduced to single screw dislocation loops. The beam was then loaded into the small four-point bending jig so that the loops would lie in the tension surface, since they were predominantly in the proper orientation to expand under the application of a tensile stress. A weight, sufficient to give the desired resolved stress, was then lowered onto the jig for the required time. The beam was removed from the jig, re-etched, and the distance each branch of each single loop had moved was measured. From these measurements the mean velocity was computed and 95% confidence limits applied to the results as in the accompanying calculation.

Since the edge components of the dislocations move more readily than do the screw components, the loops which met the surface as screw

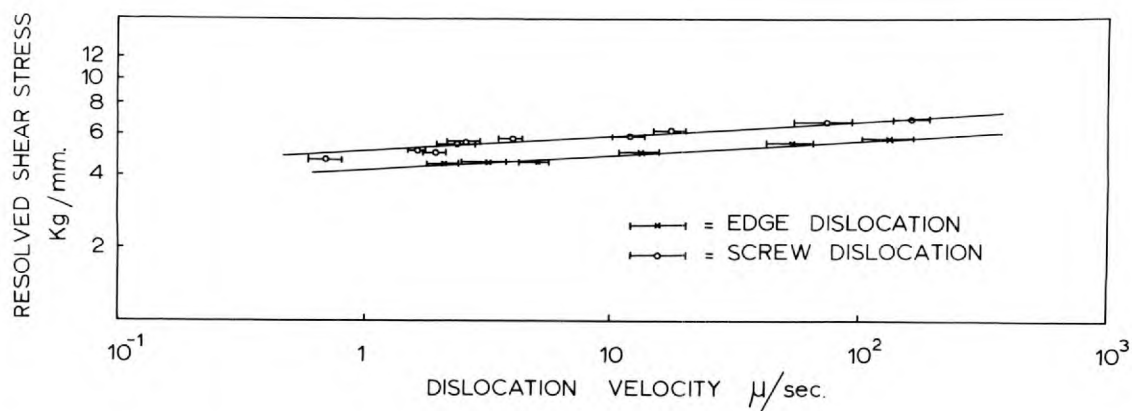


Fig 12. Plot of velocity vs. stress for edge and screw dislocations in MgO at room temperature.



Fig 13. An expanded dislocation loop showing multiplication and new dislocations within the original loop. x 475.



Fig 14. The expansion of many randomly orientated dislocation loops in MgO. x45.

dislocations, and were inclined to the surface at  $45^\circ$ , expanded more deeply into the crystal than did the loops whose screw components moved towards the neutral axis. This rendered it almost impossible to remove all the surface loops in the screw orientation and still leave single loops in the edge orientation for velocity measurements. Consequently, for the measurement of edge dislocation velocity, the beams were sprinkled and then polished until etching revealed rosettes with all but one or two edge loops removed. These specimens were loaded, as described above, and the same calculations applied to the results, care being taken only to measure the distance moved by the edge dislocations whose motion was not interfered with by other expanding dislocation loops.

The results obtained for velocities up to about  $100\mu/\text{sec}$  are plotted in Fig 12. It is evident that the edge dislocations move significantly faster than do the screws at the same applied stress as suggested by the work of Stokes, Johnston and Li (1959b) on the effect of surface conditions in three-point bending tests.

At velocities in excess of about  $5\mu/\text{sec}$  extensive cross slip and multiplication occurred. In addition new dislocations appeared within the original loop, these results corresponding very closely to the observations reported by Gilman and Johnston (1957) for LiF. Such an expanded loop in the screw orientation is shown in Fig 13. The multiplication probably had some effect on the stress driving the outermost loop, but since this type of multiplication occurs under normal testing conditions, results obtained for velocities in excess of  $5\mu/\text{sec}$  were plotted, the velocity of only the leading dislocation being calculated.

Some tests were performed at high stresses by impacting the jig, but the results obtained had a great deal of scatter. These results, though not giving a good velocity measure, gave for any single test an indication of the ratio of the velocities of edge and screw dislocations.

The results all indicated that at room temperature, over the range of velocities studied, edge dislocations move at a velocity fully an order of magnitude greater than that of the screws. This difference is evident in Fig 14 which shows a beam with many random sources after it had been loaded for a short period of time.

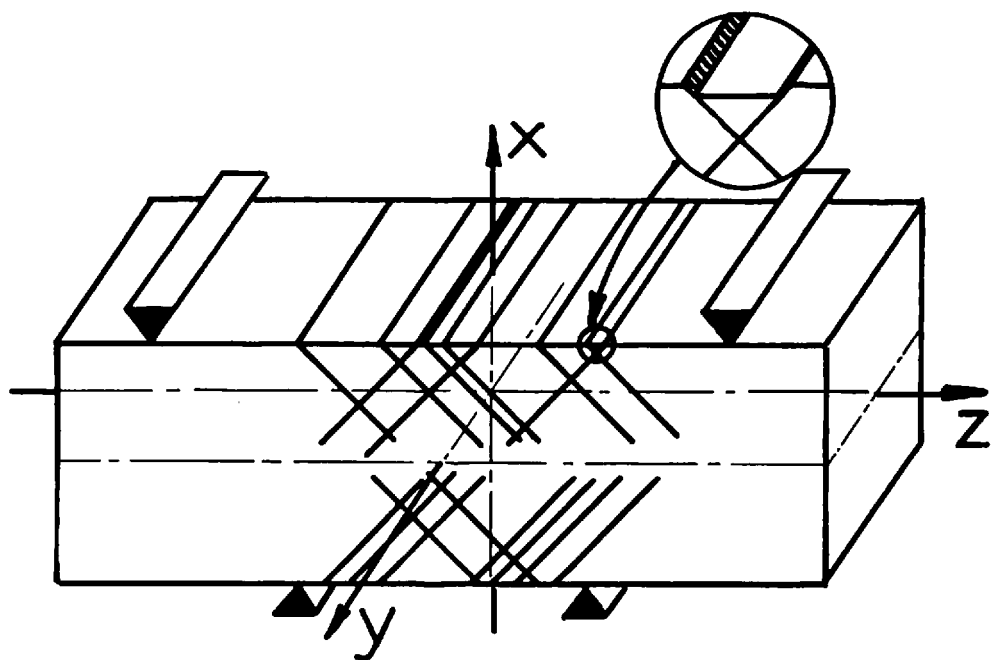


Fig. 15(a) Schematic diagram of 2D bending slip.

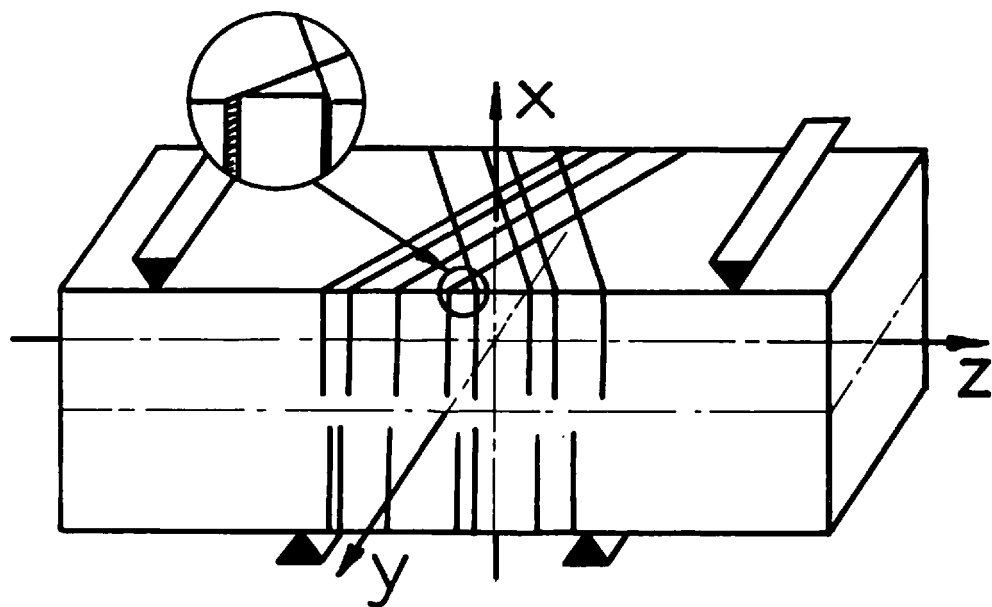


Fig. 15(b) Schematic diagram of 3D bending slip.

In LiF, Johnston and Gilman (1959) report that edges move at approximately 50X the velocity of screws in the velocity range reported above for MgO. Experiments by Davidge (1961) indicate that the edges are more mobile than the screws in the calcium doped NaCl, although no absolute measure of the velocity was obtained.

Consequently, one would expect that, were there many randomly orientated sources in the tension and compression faces of a beam of any of these materials, a significant proportion of the slip should take place in bands crossing the tension and compression faces at  $45^\circ$ . This indeed occurred, as is shown in Fig 14, and the slip in this orientation was found to have a profound effect on the mechanical properties of the beams in which it occurred. In the light of these observations it proved important to differentiate between the two modes of slip, and to define each more carefully relative to the beam geometry.

### II.3 Two Dimensional Slip

Assuming the beam orientated in a system of axes as illustrated in Fig 15(a), the slip that is normally assumed to produce the deformation in the beam is in the planes (101) and (10 $\bar{1}$ ). The Burgers vectors of the dislocations lie in the directions [ $\bar{1}$ 01] and [ $\bar{1}$ 0 $\bar{1}$ ], on the tension side, and [10 $\bar{1}$ ] and [101] on the compression side of the beam. Ignoring small variations in the strain across the beam width, the slip in this orientation can be entirely defined in any plane in the beam parallel with the plane  $y=0$ , since the bands of slip are all normal to such a plane, and the Burgers vectors of the dislocations all lie in the plane. Consequently all slip so orientated shall be defined as two dimensional slip after Nye (1953), and shall hereafter be referred to as 2D slip.

### II.4 Three Dimensional Slip

Assuming again that the beam is orientated in the co-ordinate axes as illustrated in Fig 15(b), the slip produced by edge dislocations moving at the tension and compression faces, lies in the planes (011)

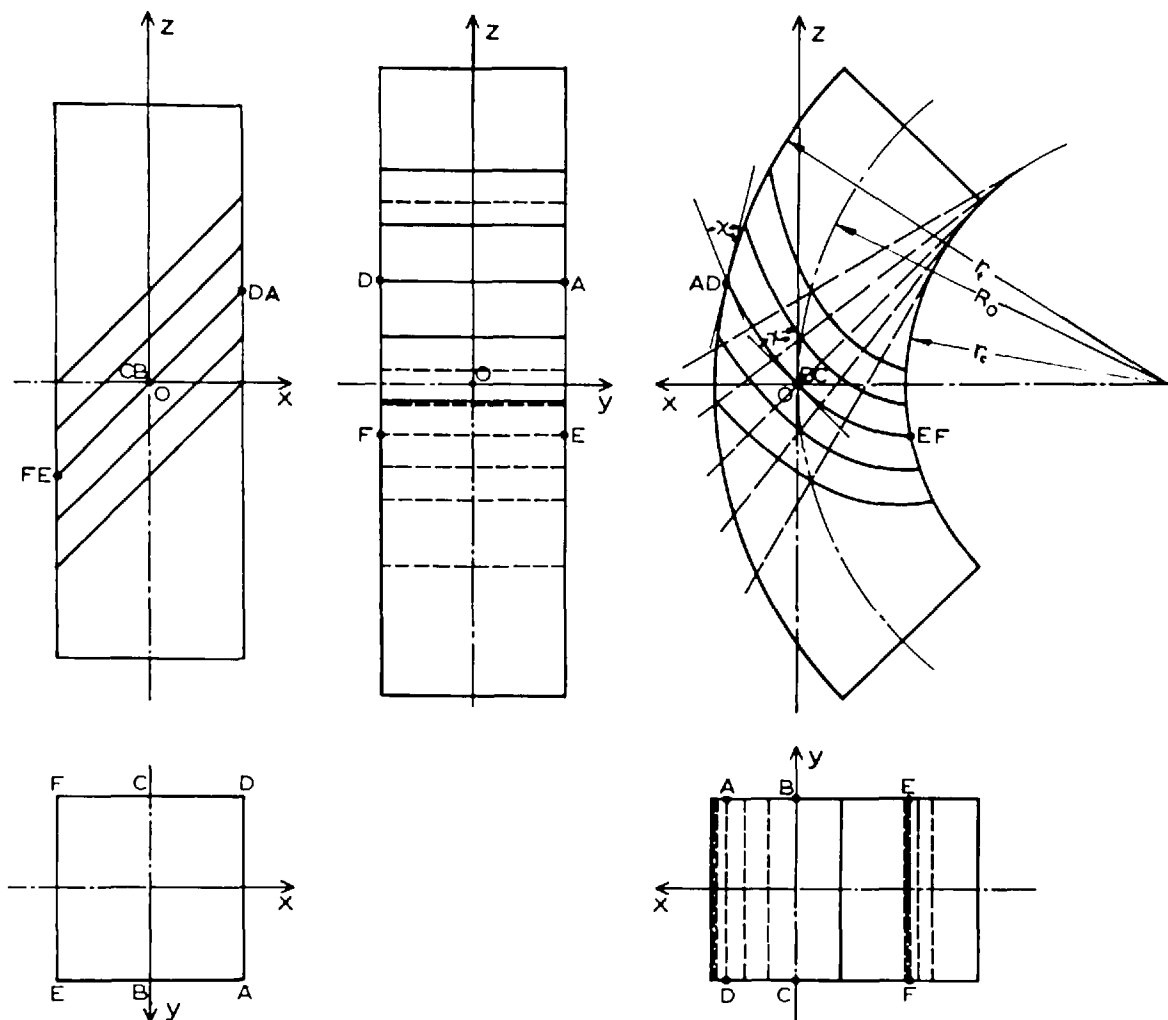


Fig 16. A schematic diagram of a beam severely strained in pure bending with all the deformation produced by homogeneous 2D slip. On the left a slip plane in the undeformed beam is shown and on the right the same plane, DAEF, is shown after deformation. For the strain illustrated the slip surface has become an involute of a circular cylinder of radius  $r_c$ .

and  $(0\bar{1}1)$ . The Burgers vectors lie in the directions  $[0\bar{1}1]$  and  $[011]$  at the tension surface, and  $[0\bar{1}1]$  and  $[011]$  at the compression surface. The dislocation density in each slip band decreases as the neutral axis is approached from either the tension or compression side. In addition the slip bands are not normal to the sides of the beam, and are both parallel with and normal to each other. Consequently, this slip cannot be defined in a single plane, but must be defined relative to all three dimensions of the beam. Hence slip so orientated shall be defined as three dimensional slip, again after Nye (1953), and shall be referred to subsequently as 3D slip.

## II.5 Lattice Geometry Associated with 2D Slip.

When slip occurs in the 2D orientation under the influence of a pure bending stress it is likely that the vast majority of the dislocations in the glide bands are of such a sign that under the influence of the stress they move into the beam towards the neutral axis. When this is the case the dislocations not only permit the beam to bend about the major axis of curvature by producing the necessary extension and contraction in the beam, but in addition create a curvature in the lattice. If it is assumed that in any specific part of the beam all the slip has occurred in a single direction, there being no slip on the orthogonal planes, the conditions described by Nye (1953) for the case of single glide in the uniform plane bending of a beam are met. Nye showed for this case that the glide planes become involutes of a circle and that the glide plane normals remain straight lines. This type of deformation is illustrated in Fig 16 for the particular case in which the slip takes place on  $(10\bar{1})$  in an orthorhombic specimen bounded by  $\{100\}$ . A B E F C D is a slip surface shown both before and after deformation.

In the case of a beam with a rectangular cross-section of depth  $2a$  and width  $2b$  and whose slip system is that described above, the radius of the generating circle of the involutes is  $2\sqrt{2}a$ . This radius is the theoretical minimum radius to which the compression surface can be bent,

and corresponds to a strain sufficient to turn the glide surfaces to a position normal to the beam face at the compression surface. Assuming that the volume change due to elastic deformation is negligible, then when the compression surface is bent to this radius,  $r_c$ , the neutral axis will be bent to a radius  $R_o$ , and the tension surface to a radius  $r_t$  where

$$\frac{1}{2\pi R_o} \int_{r_c}^{R_o} 4b\pi r dr = 4lab = \frac{1}{2\pi R_o} \int_{R_o}^{r_t} 4b\pi r dr.$$

$$\text{Therefore, } \frac{1b}{R_o} (R_o^2 - r_c^2) = 4lab = \frac{1b}{R_o} (r_t^2 - R_o^2).$$

Since  $r_c = 2\sqrt{2}a$ ,  $R_o = 4a$  and  $r_t = 2\sqrt{6}a$ .

Consequently, the maximum strain that can be accommodated in a beam undergoing pure bending without moving the neutral axis corresponds to a radius of curvature of twice the beam depth, or on the basis of the most rudimentary calculation, an outer fibre strain of 25%. At this strain the depth of the beam on the tension side of the neutral axis will have been reduced from  $a$  to  $2\sqrt{6}a - 4a$ , or approximately by  $0.05a$ , while on the compression side the beam depth will have increased from  $a$  to  $4a - 2\sqrt{2}a$ , an increase of  $0.17a$ . The resultant total depth of the beam will be  $2.12a$ .

The angle between the slip plane and the surface  $r = \text{constant}$  at any point in the beam will be given by  $\chi = \cos^{-1} \left( \frac{r^2 - 8a^2}{r^2} \right)^{\frac{1}{2}}$ . At the tension surface  $\chi$  will be approximately  $35^\circ$ , at the neutral axis  $45^\circ$ , and at the compression surface  $90^\circ$ , for the strain considered. Therefore, the resolved shear stress in the slip planes at the compression surface will decrease to zero as this strain is approached regardless of the applied load, and this strain must be the limit to which beams can be deformed in bending if the neutral axis is not allowed to move.

However, a practical limit is reached at strains significantly below this strain. The stress on the tension side must be greater than the stress on the compression side if equal portions of the bending load are to be borne on each side of the neutral axis, a condition of equilibrium in the bending of beams. Furthermore, the relative magnitude of the stress

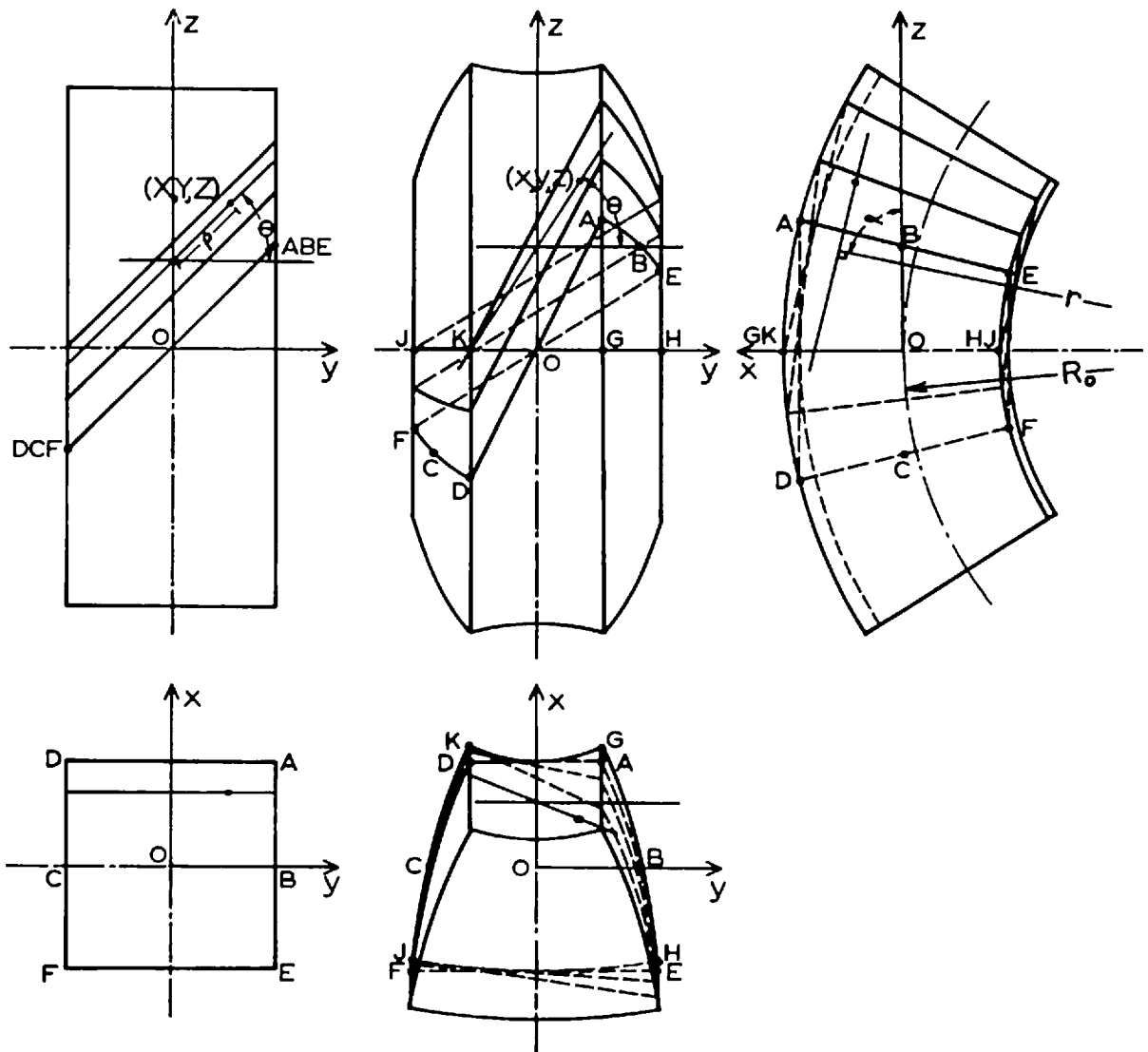


Fig 17. A schematic diagram of a beam severely strained in pure bending with all the deformation produced by homogeneous 3D slip. On the left a slip plane ABEFCD and a random point  $(X, Y, Z)$  are shown in the undeformed beam. The line drawn through the point is parallel with the Burgers vectors of the dislocations passing through the point. The beam cross-section GHKJ is bounded by the ellipse

$$\frac{(X+R)^2}{2R^2} + \frac{Y^2}{2b^2} = 1$$

for the sides GH and JK, and the hyperbolas

$$\frac{X^2}{(R_0 \pm a)^2} - \frac{Y^2}{(R_0 \pm a)^2 \tan^2 \theta} = 1$$

for the top and bottom, GK and HJ respectively.

resolvable in the direction of slip is always greater at the tension surface than at the compression surface. Consequently, in beams deformed plastically in this mode the stress driving the dislocations is always greater on the tension side of the neutral axis than on the compression side. This leads to the inward displacement of the neutral axis at strains significantly below the limiting case considered here. These arguments, added to those of Read (1957) demonstrate further the complexity of the stresses that must be present in beams plastically bent to large strains even when the conditions of slip are the simplest possible.

## II.6 Lattice Geometry Associated with 3D Slip.

To determine the distortion of a beam loaded in pure bending and deformed exclusively with slip in the 3D orientation all in a single direction, consider first an  $(0\bar{1}1)$  plane in an undeformed beam such as the plane AEFD in Fig 17. Assuming that the elongation of the tension side of the beam and the contraction of the compression side are produced by the rotation of the lattice, then every line in the plane parallel with the AD or EF edges of the plane will undergo a rotation, but will remain a straight line of the same length. If  $\theta$  is defined as the angle between any such line and the plane  $z=0$ , after deformation  $\theta$  will have increased to values greater than  $45^\circ$  on the tension side of the beam whereas on the compression side of the beam  $\theta$  will have decreased to values less than  $45^\circ$ . The extreme maximum and minimum values will occur at the tension face and the compression face respectively, while at the neutral axis  $\theta$  will remain  $45^\circ$ .

Consider the line passing through any point  $(X,Y,Z)$  in the undeformed beam in the direction  $[011]$ . After deformation, the normal to this line passing through the centre of curvature of the beam will be inclined to the  $z$ -axis at an angle  $\alpha$  and the line itself will be inclined to the  $y$ -direction at the angle  $\theta$ . Let  $\rho$  be the distance from the centre point of the line to the point  $(X,Y,Z)$  and let  $r$  be the distance from the line to the centre of curvature of the beam. Then if the co-ordinates of the point after deformation are  $(x,y,z)$  we can write,

$$x = r \sin \alpha - \rho \sin \theta \cos \alpha \quad \text{-----} \quad (1)$$

$$y = \rho \cos \theta \quad \text{-----} \quad (2)$$

$$z = r \cos \alpha + \rho \sin \theta \sin \alpha \quad \text{-----} \quad (3)$$

Therefore

$$x^2 = r^2 \sin^2 \alpha + \rho^2 \sin^2 \theta \cos^2 \alpha - 2r\rho \sin \alpha \sin \theta \cos \alpha$$

$$y^2 = \rho^2 \cos^2 \theta$$

$$z^2 = r^2 \cos^2 \alpha + \rho^2 \sin^2 \theta \sin^2 \alpha + 2r\rho \sin \alpha \sin \theta \cos \alpha$$

$$\text{and } x^2 + z^2 = r^2(\sin^2 \alpha + \cos^2 \alpha) + \rho^2 \sin^2 \theta (\sin^2 \alpha + \cos^2 \alpha) \\ = r^2 + \rho^2 \sin^2 \theta$$

and substituting equation (2)

$$x^2 + z^2 = r^2 + y^2 \tan^2 \theta$$

giving

$$\frac{x^2}{r^2} - \frac{y^2}{r^2/\tan^2 \theta} + \frac{z^2}{r^2} = 1 \quad \text{-----} (4)$$

This is the equation of a hyperboloid of one sheet.

If R is redefined more exactly as the radius of curvature after deformation of any line originally lying parallel to the z-axis in the plane  $X = 0$  of the undeformed beam, and  $R_0$  the radius of curvature of the particular line originally lying along the z - axis, then r is equal to  $X + R_0$ . Hence any plane originally  $x = X$  becomes the hyperboloid of one sheet,

$$\frac{x^2}{(R_0 + X)^2} - \frac{y^2}{(R_0 + X)^2/\tan^2 \theta} + \frac{z^2}{(R_0 + X)^2} = 1 \quad \text{-----} (5)$$

and intersects the plane of cross section of the beam in the hyperbola

$$\frac{x^2}{(R_0 + X)^2} - \frac{y^2}{(R_0 + X)^2/\tan^2 \theta} = 1 \quad \text{-----} (6)$$

It follows that the tension and the compression faces of the beam, originally  $X = \pm a$  respectively, intersect the plane  $z = 0$  along the hyperbolas,

$$\frac{x^2}{(R_0 \pm a)^2} - \frac{y^2}{(R_0 \pm a)^2/\tan^2 \theta} = 1$$

Making use of the above definition of  $R$  we can express the longitudinal strain in the beam at any point as  $(r-R_0)/R_0$  which is  $X/R_0$ . Hence we can write,

$$\sin \theta = (1 + X/R_0)/\sqrt{2}$$

$$\sin^2 \theta = (1 + X/R_0)^2/2 \quad \text{-----}(7)$$

$$\cos^2 \theta = (1 - 2X/R_0 - X^2/R_0^2)/2 \quad \text{-----}(8)$$

$$\tan^2 \theta = (1 + X/R_0)^2 / (1 - 2X/R_0 - X^2/R_0^2) \quad \text{-----}(9)$$

If  $X > 0$ ,  $\tan^2 \theta > 1$ ; if  $X < 0$ ,  $\tan^2 \theta < 1$ ; and at  $X = 0$ ,  $\tan^2 \theta = 1$ .

Hence, the equation of the surface originally the plane  $X = 0$  and defined as the neutral surface in the deformed beam is

$$\frac{x^2}{R_0^2} - \frac{y^2}{R_0^2} + \frac{z^2}{R_0^2} = 1,$$

a rectangular hyperboloid of one sheet, which intersects the plane  $z = 0$  along the rectangular hyperbola,

$$\frac{x^2}{R_0^2} - \frac{y^2}{R_0^2} = 1$$

Returning to equation (2) and substituting values for  $\cos \theta$  from equation (8) we can write

$$y^2 = \rho^2(1 - 2X/R_0 - X^2/R_0^2)/2$$

and since

$$\rho^2 = 2Y^2$$

$$y^2/Y^2 = 1 - 2X/R_0 - X^2/R_0^2$$

$$\frac{(X + R_0)^2}{2R_0^2} + \frac{y^2}{2Y^2} = 1$$

But  $\frac{X + R_0}{R_0}$  in the plane of cross-section,  $z = 0$ , can be very closely approximated by  $\frac{x + R}{R}$ .

Therefore we can write that any planes  $y = \frac{1}{2} Y$  in the undeformed beam, after deformation become parts of the surface of the oblate spheroid

$$\frac{(x + R)^2}{2R^2} + \frac{y^2}{2Y^2} + \frac{z^2}{2R^2} = 1 \quad \text{-----(10)}$$

and the sides of the beam intersect the cross section  $z = 0$  along the ellipse

$$\frac{(x + R)^2}{2R^2} + \frac{y^2}{2b^2} = 1 \quad \text{-----(11)}$$

It is interesting to note that the family of hyperbolas of equation (6) with the parameter  $X$  and the family of ellipses of equation (10) with the parameter  $Y$  defined in the plane of cross-section of the beam are not confocal and consequently not orthogonal.

## CHAPTER THREE      CHARACTERISTICS OF BEAMS DEFORMED IN PURE BENDING

### III.1 Introduction

Either three-point loading or four-point loading can be employed to deform beams in bending tests. The stress at any point in a beam loaded at three points is the sum of a constant simple shear stress, and a bending stress which decreases linearly from the inner loading point out to the outer loading points. Consequently, there is no region of constant stress, and the maximum stress and strain are reached in the immediate region of the inner loading edge. The contact of the loading edge with the beam undoubtedly influences the nucleation and propagation of slip, and as a result influences the apparent plastic behaviour of the material.

In a four-point loading test these complications are minimised. Between the inner loading points the stress does not vary with displacement along the beam and is the result of a constant simple bending moment only. Between the inner and outer loading points the same conditions exist as in three point loading, but these portions of the beam may be ignored if necessary.

Since dislocation velocity is extremely sensitive to small differences in stress, as indicated in II.2, it seems of prime importance that the stress driving the dislocation in a particular test be as nearly constant as possible. In three point loading, apart from the inherent longitudinal variation in the bending moment, the additional shear stress can be resolved in the direction of 2D slip and not in the direction of 3D slip. The component resolvable in the direction of 2D slip aids the slip in one direction on each side of the neutral axis and inhibits the slip in the orthogonal direction on each side. Consequently, the use of three point bending biases the occurrence of slip in favour of 2D slip on both the tension and compression sides of the beam.

With these considerations in mind, it was decided to consider only the results of tests performed under four-point loading conditions, and further, to limit the investigation of bending



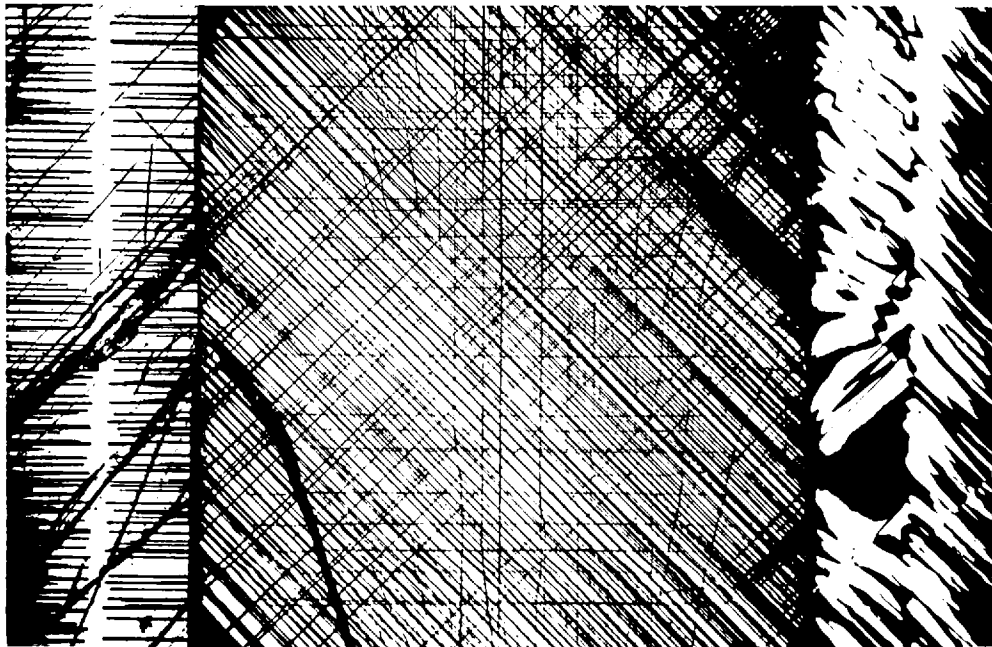


Fig 20. Composite photomicrograph of the etched tension surface, one side, and the birefringence pattern observed from the other side for a beam deformed in the as-cleaved state. x 32

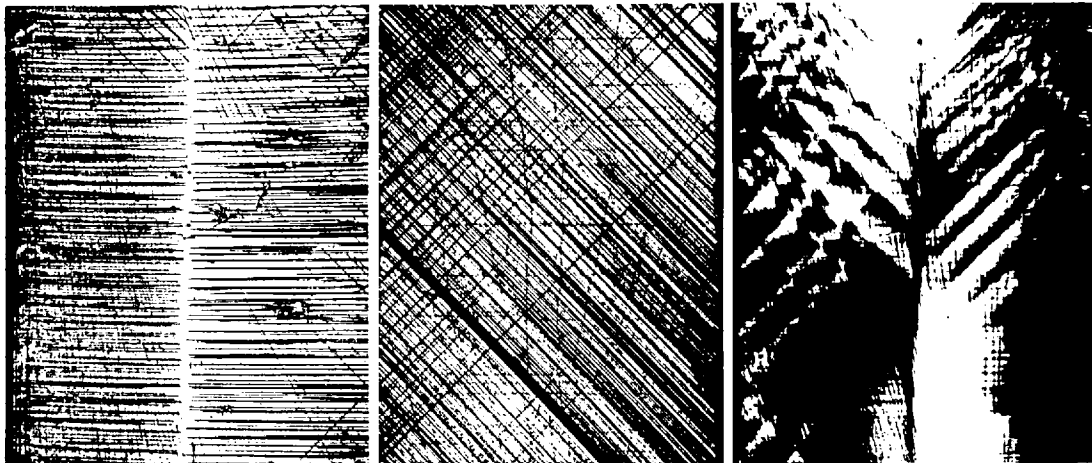


Fig 21. Composite photomicrograph of the same views as those in Fig 20 but for a much deeper beam deformed in the as-cleaved state. x 26

phenomena to the region between the inner knife-edges. On some occasions it proved necessary to consider the behaviour of the beam between the inner and outer knife-edges, in order to explain the observed phenomena, but these occasions were generally rare.

### III.2 As-Cleaved Beams

When as-cleaved beams of MgO were bent in the four-point bending jig at room temperature, they almost always fractured at very low strains, only rarely exceeding 0.1%. The fracture usually originated at a microcrack at an edge of the tension surface, and propagated through the cross-section as a cleavage crack on the (001) plane. This type of fracture, originating at a point source at an edge, was reported by Parker (1959) and Clarke and Sambell (1960) in tension specimens, and by Johnston, Stokes and Li (1959), and Clarke, Sambell and Tattersal (1962), in bending specimens. A picture of fracture originating in this way is reproduced in Fig.18. Clarke (1962) has recently established that the microcrack at which the fracture originates need not be a result of the cleaving, but may be formed at a point where a 3D slip band reaches the edge of the tension face.

Typical plots of load/load at flow, vs. strain, are given in Fig.19. Most of the beams fractured at a load roughly 1.5x load at flow showing a very steep rate of work hardening up to this point. Usually a great deal of the slip in the beam was in the 3D orientation and in those beams in which this was the predominant mode of slip, what slip there was in the 2D orientation appeared to originate near the centre of the tension and compression faces and not to propagate completely out to the edges. A beam of this description is shown in Fig.20 after it had been etched. The birefringence pattern shows, that although the 2D slip did not propagate out to the edges, it did propagate deeply into the beam to the neutral axis. Fig.21 is a composite photomicrograph of a beam that behaved very much the same as the beam shown in Fig.20 even though their dimensions were completely different. A feature common to all beams that deformed predominantly in the 3D mode was that the neutral axis

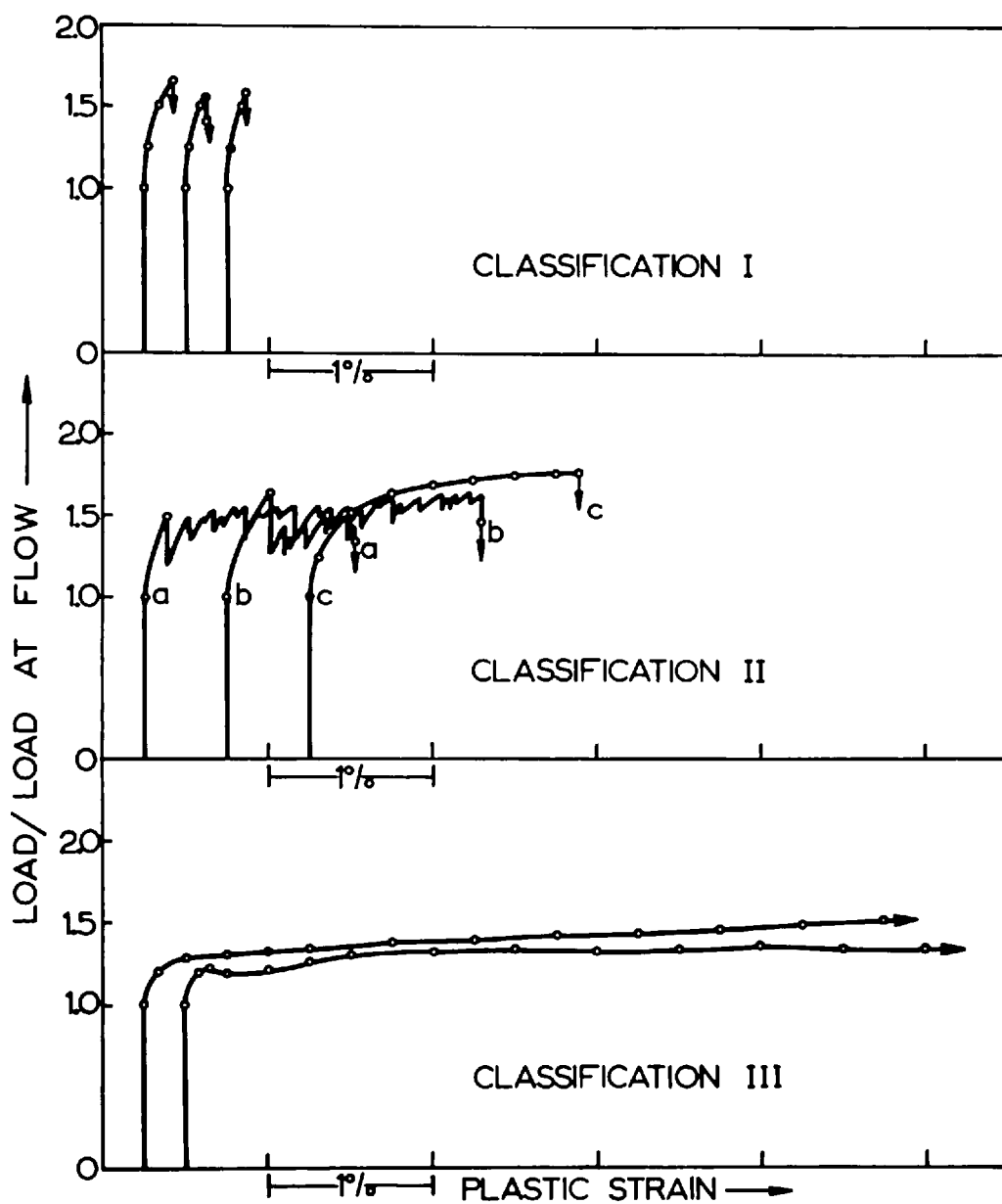


Fig 22. MgO load-strain curves for beams bent at room temperature after polishing.

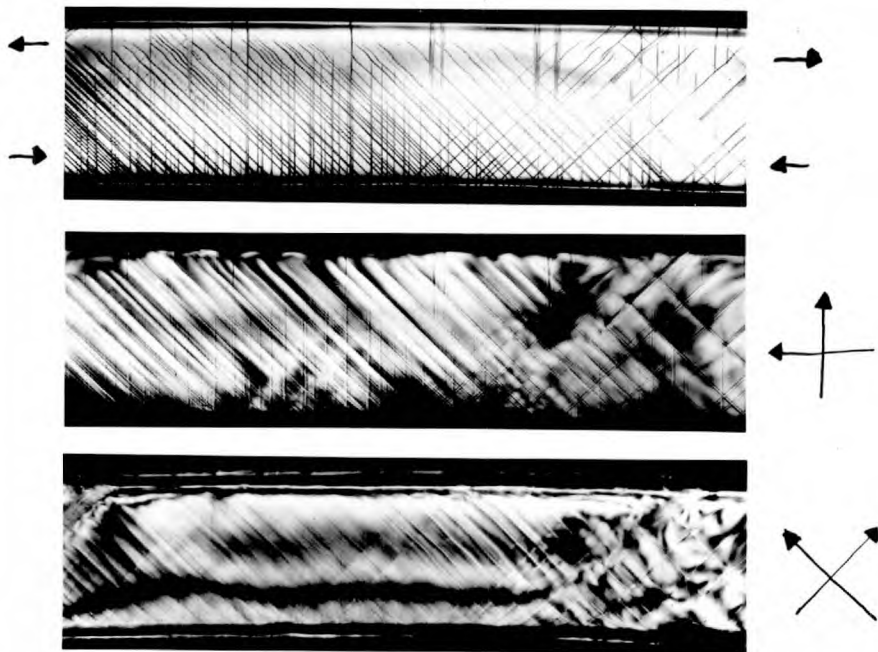


Fig 23. A lightly deformed, polished beam in which the neutral axis was distorted and displaced. Top: etched side of beam. Middle and lower: Birefringence in same view with the nicols in the directions indicated. x 28

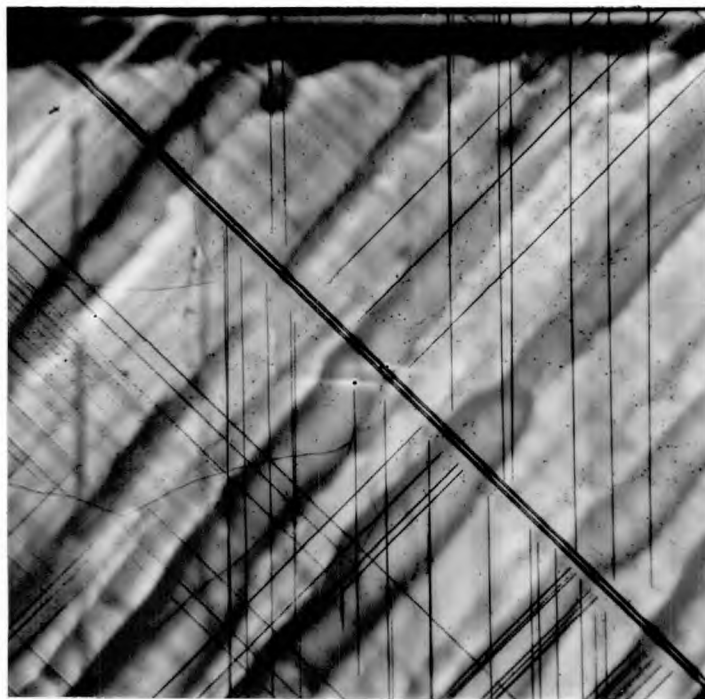


Fig 24. Etched tension surface of a beam slightly bent in the polished condition showing two edge-on three dimensional slip bands blocking the propagation of other slip. Tension direction horizontal. x 30



Fig 25. Complex fracture  
originating from a (101) slit. x 85.

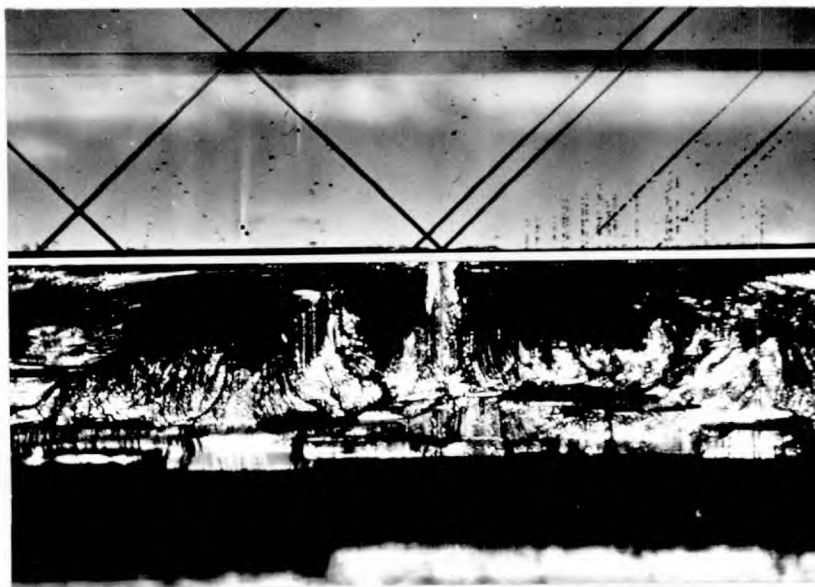


Fig 26. Composite photomicrograph showing the intersection  
of two 3D slip bands on the tension surface at which the  
fracture originated. The fracture surface is in the beam  
cross-section and the tension direction vertical. x 105.

remained clearly defined as is evident in both Figs.20 and 21.

### III.3 As-Polished Beams

Beams of MgO, polished before bending until all etchable fresh dislocation loops had been removed, displayed a wide variation in behaviour. They could be classified in three groups according to the strain achieved before fracture.

#### (a) Classification I

Some of the polished beams fractured at a very early stage in the deformation, at strains from 0.1% to 0.5%. Representative load-strain curves for such beams are plotted in Fig.22. It can be seen from the curves that the work-hardening rate varied from specimen to specimen but generally was quite high and that most of the beams fractured at loads roughly 1.5 x load at flow. The slip bands were normally fewer in number, but much heavier than those in the as-cleaved beams and a significant proportion of the slip occurred in both the 2D and the 3D orientation. In many beams the slip pattern was extremely irregular with no recognizable neutral axis evident, either on the etched surface, or in the interior birefringence pattern. A beam that deformed in this fashion is shown in Fig.23. The interference of established 3D slip bands with bands formed at a later stage in the deformation was frequently observed and is clearly portrayed in Fig.24.

In almost all the beams in this category, the fracture originated at the intersection of two orthogonal slip bands. If the slip bands were in the 2D orientation, the fracture normally grew from a line source along the intersection of the slip bands, leaving the characteristic (101) step on the fracture surface. Sometimes this step did not completely traverse the cross-section, possibly because of the interference of slip in the other orientation, this behaviour giving rise to complicated fracture surfaces of the type illustrated in Fig.25. Line sources of this description were reported by Stokes, Johnston and Li (1959(a)) in bending specimens, and by Stokes et al. (1958) and by Johnston et al. (1959) in tension and compression specimens.

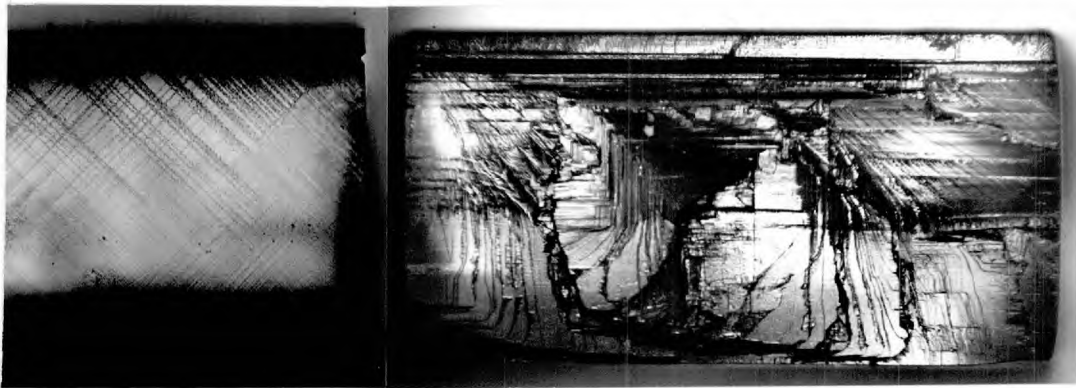


Fig 27. A typical fracture that originated at a line source along the intersection of two 2D slip bands. x 28.

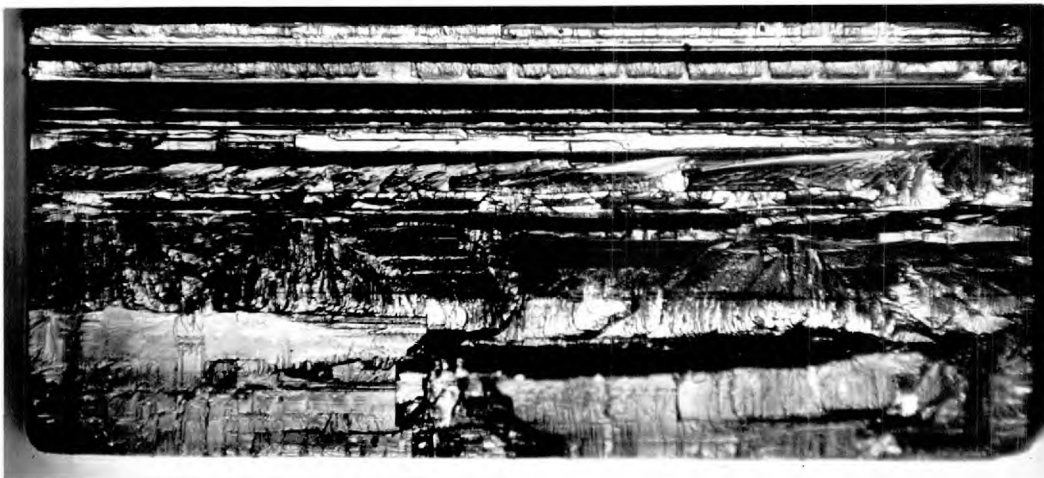


Fig 28. A typical fracture surface crossed by a number of parallel (101) steps. x 40.

In a significant number of the beams the fracture originated at the intersection of two 3D slip bands at the tension surface, as illustrated in Fig.26. The mechanism responsible for the crack nucleation is probably the same as that operating at the intersection of 2D bands, but since there is a strain gradient along the 3D intersection the characteristic long slit and resultant step is not formed. In both cases, the mechanism creating the initial crack at the slip band intersection is probably that assumed by Stroh (1955) in which edge dislocations, forced against a barrier, coalesce to form the crack nucleus. It is evident in Fig.26 that the slip bands need not be very heavy to provide a sufficiently strong barrier for a pile-up. It might even be suggested, the slip lines are so fine in the illustrated case, that the fracture originated at a microcrack already in the surface. However, were this the case, one would expect the radiating slip bands to be much heavier, since such a crack would act as a source of slip from the earliest stages of deformation.

#### (b) Classification II

The great majority of the beams, deformed in the polished state, accommodated between 1% and 2% plastic strain before fracture. The load-strain curves for these beams, reproduced in Fig.22, varied from very smooth curves with a relatively low rate of work-hardening to extremely irregular curves with sharp load drops which sometimes amounted to 20% of the applied load.

The beams for which the load-strain curves were smooth, deformed predominantly in the 2D mode in many intersecting slip bands. Usually they fractured from a line source along the intersection of two of these bands as described earlier. A typical fracture is illustrated in Fig.27.

On the other hand, the beams for which the load-strain curves were discontinuous deformed with a much larger proportion of slip in the 3D mode. When observed under polarised light during deformation, it could be seen that the load drops coincided with the formation of at least one heavy band of 2D slip, and in addition, were accompanied by an audible "squeak" or "click". Discontinuities of a similar nature in the load-strain behaviour of MgO were reported by Johnson and Cornet (1961) and Stokes (1962). Classen-Nekludova (1929) reported

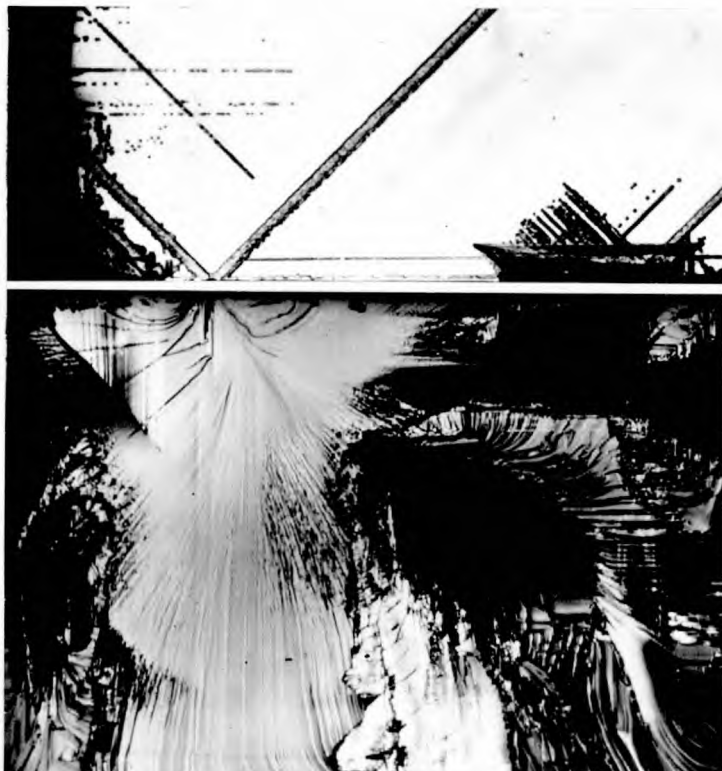


Fig 29.  
 Composite photomicrograph  
 showing a fracture that  
 originated at the inter-  
 section of two 3D slip  
 bands on the tension sur-  
 face.  
 x 110.

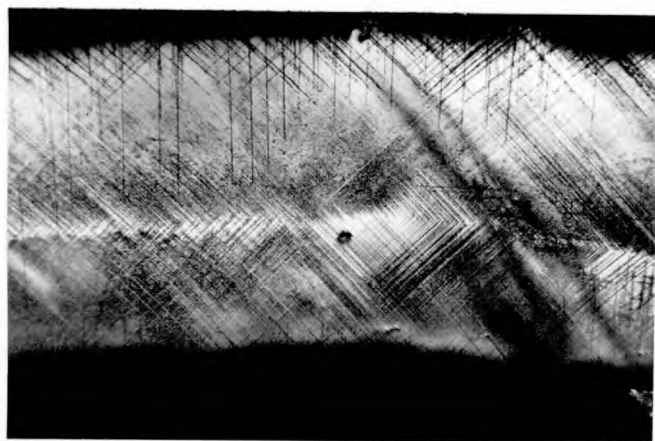


Fig 30.  
 The etched side of a beam  
 that deformed heavily,  
 with slip almost exclus-  
 ively in the 2 Dimensional  
 orientation and almost in  
 a single direction in any  
 given area.  
 x 35

apparently similar "clicking" during jerky flow at elevated temperatures in NaCl. Between the load drops the rate of work-hardening was usually significantly greater than that at the same strain for beams which deformed smoothly.

The fracture in nearly all the beams in this category grew from a line source formed at the intersection of two reasonably heavy bands of 2D slip. Very often the propagation of the fracture was forced out of the (001) plane into a (101) plane for a short distance by the other bands of 2D slip before returning to the (001) cleavage plane. This resulted in a fracture surface crossed with a number of parallel (101) steps in addition to the one at which the fracture actually originated. This type of fracture surface is shown in Fig.28.

Occasionally the fracture originated at the intersection of two 3D slip bands as shown in Fig.29, but this was seldom the case in beams deformed much more than 1%.

### (c) Classification III

A small number of the polished beams deformed almost exclusively in two dimensional slip, which, in any part of the beam was almost entirely in a single direction. When this was the case, the load-strain curves were smooth and displayed very low work-hardening rates as is evident in Fig.22. Beams deformed thus were seldom strained to fracture though strains as high as 7 to 8% were reached. Part of a beam which deformed in this manner is shown in Fig.30.

### III.4 Beams with Sources Introduced

The results of the bend tests on as-cleaved and as-polished beams seemed to indicate significant differences in the bending behaviour of beams deformed in 2D slip as opposed to 3D slip. The polished beams of classification III provided some indication of the behaviour at fairly large strains of beams deformed exclusively in 2D slip. However, all the beams that deformed almost exclusively in 3D slip were bent in the as-cleaved state and fractured at very low strains. Therefore, if a comparison were to be made between the two modes of slip, at significant strains, a method had to be found to reach much higher strains with only 3D slip operating. This meant

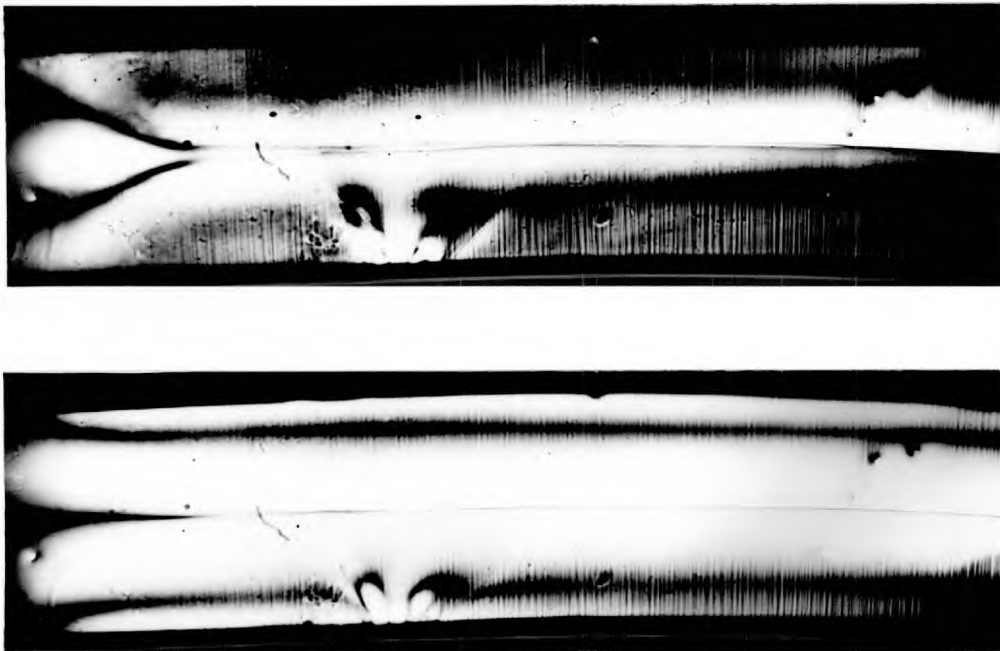


Fig 31. Two birefringence pictures taken from the side of a beam deformed entirely in 3D slip save at point of contact of the inner knife-edge.  
Top: nicols in  $\{100\}$ ; lower: nicols in  $\{110\}$ . x 21.

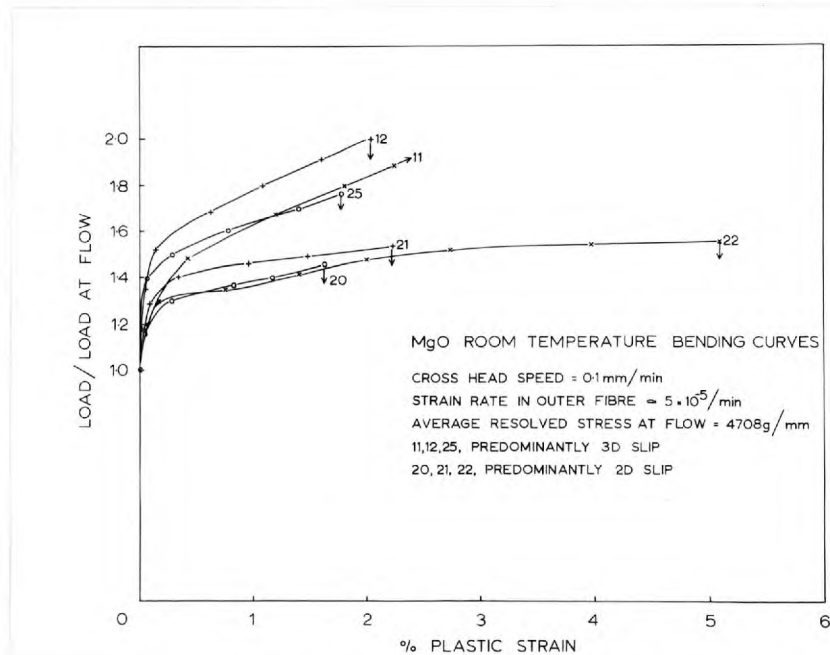


Fig 32. Load-strain plots for beams of MgO deformed at room temperature.

that beams had to be prepared with slip sources exclusively in the 3D orientation and without any microcracks that might initiate early fracture.

This was accomplished by first polishing beams until there were no etchable fresh dislocation sources in that part of the beam that would lie between the inner knife-edges. These beams were then sprinkled on their sides with 220 mesh silicon carbide from a height of three inches. As previously shown by Stokes, Johnson and Li (1959(b)), the impact of the silicon carbide under these conditions was just sufficient to produce small single dislocation loops lying in planes at  $45^\circ$  to the surface, and no loops in the planes normal to the surface. Great care was taken to prevent any damage whatever being done to the tension and compression faces during this treatment.

One half of the loops introduced in this way were in the right orientation, and some of these were of the right sign to expand when the bending stress was applied. The expanded loops all lay in (011) or (0 $\bar{1}\bar{1}$ ) planes and created bands of 3D slip on both the tension and compression sides of the beam. Beams prepared in this way deformed up to about 2% before fracture. Some of them had no more than two or three bands of 2D slip, and these bands usually originated at the loading edges; such a beam is pictured in Fig.31. Load-strain plots of some beams prepared in this way are given in Fig.32.

Those beams that deformed up to about 2% usually reached an applied load approximately twice the load at flow, and their work-hardening rate was substantially greater than that of the beams deformed in 2D slip over comparable strains. A strain of 2% proved insufficient to allow any accurate checking of the distortion of the beam cross-section predicted in II. 6. In fact, it proved very difficult to obtain any sections of the beams for examination that did not contain numerous cracks. Beams strained to this extent in 3D slip, though displaying no cracks when removed from the bending jig, shattered into many pieces if any attempt was made to cleave or saw them. In some cases, apparently flawless beams shattered during etching, and many others cracked while being handled in spite of the

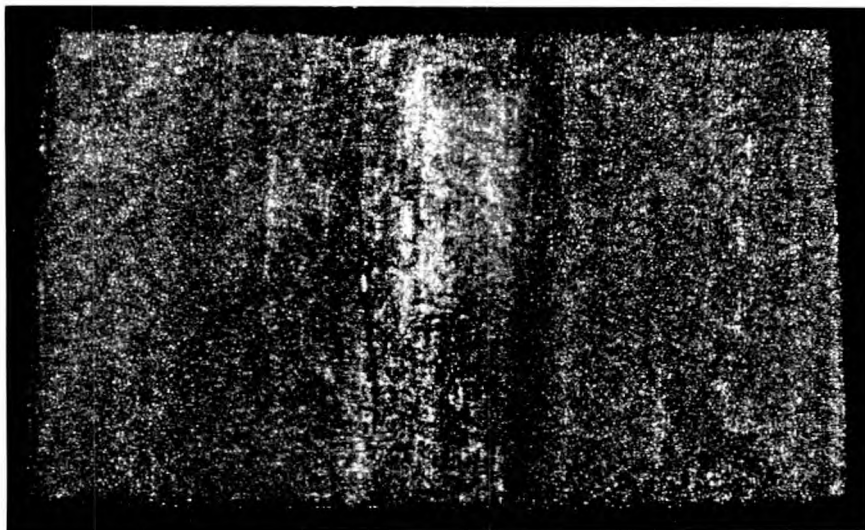


Fig 33. The sawn cross-section of a beam of MgO deformed approximately 2% with slip exclusively in 3D orientation. x 65.

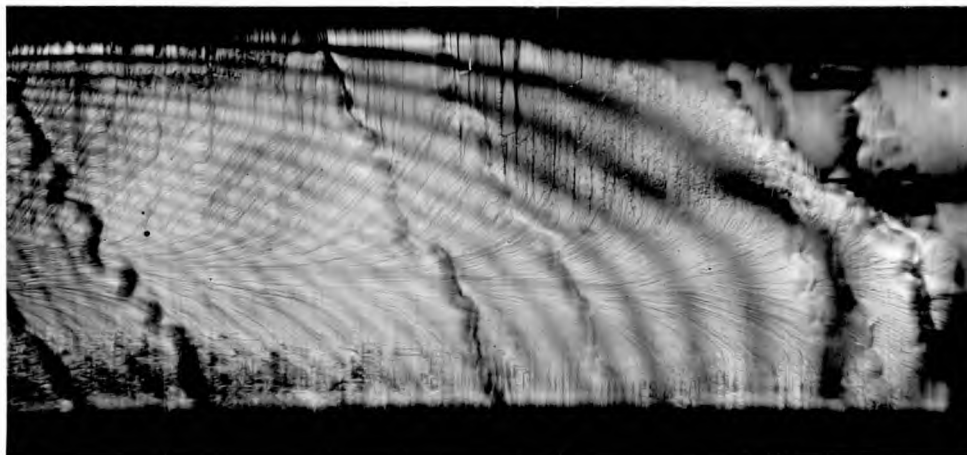


Fig 34. A typical longitudinal crack that propagated along the beam on the compression side of the neutral axis. x 38.

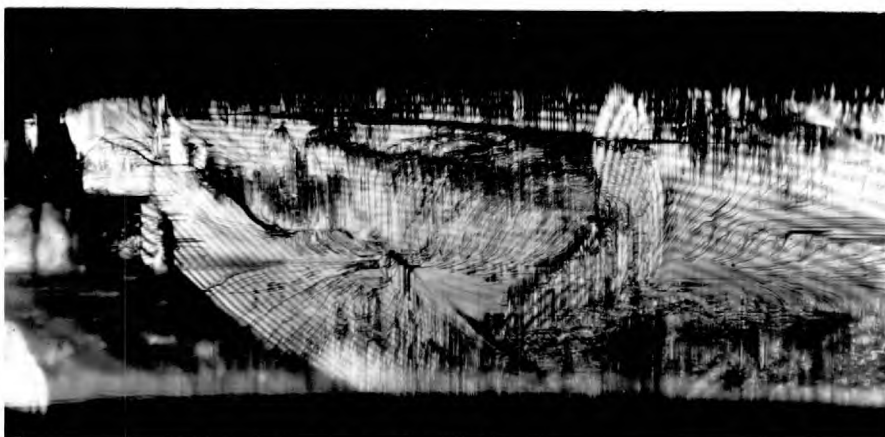


Fig 35. The region of the crack origin in a beam deformed in 3D slip showing cracking on many planes including the longitudinal (010) and (101) diagonal planes. x 35.

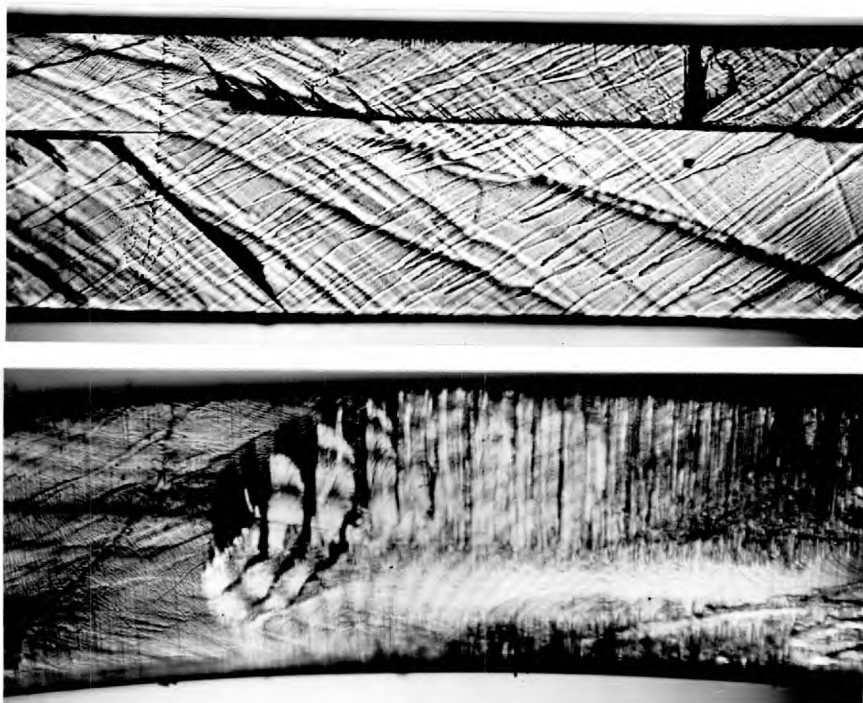


Fig 36. The leading edge of a longitudinal crack showing feathery off-shoots turning into (011) on the tension side of the neutral axis. x 28.

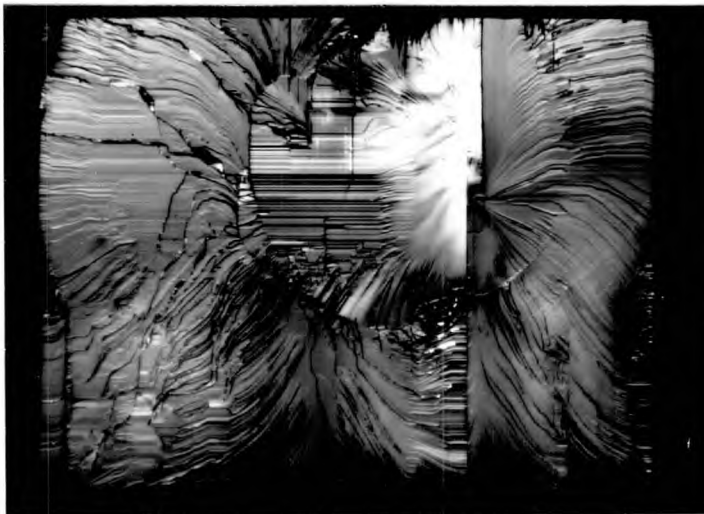
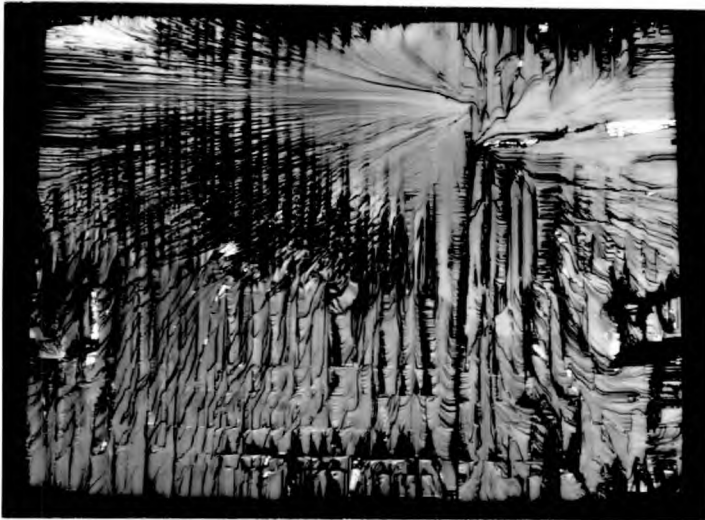


Fig 37. Transverse cleavage fractures that originated at a longitudinal crack on the tension side of the neutral axis. In the lower picture note the nearly circular hesitation line in the fracture pattern. Both x 45.

great care exercised; and, as might be expected, many of the beams shattered while still in the bending jig during the removal of the load. However, some suitable sections of the beams were obtained for examination, and one of these is shown in Fig.33. Although it is only slight, the distortion of the cross-section of the beam can be seen, and seems to conform, at least in sign, with that predicted earlier.

An interesting feature of the fracture that occurred during and after the removal of the load was that it often originated on the compression side of the beam. In some cases it appeared to have actually originated in an (010) plane and propagated as a cleavage crack longitudinally down the compression side of the beam. A photograph of such a fracture is reproduced in Fig.34. Often, the same type of longitudinal crack was formed, but the point at which it originated corresponded with the point at which a crack in a (101) plane originated as is illustrated in Fig.35. Consequently it was not possible to determine which crack had in fact occurred first. It can be seen clearly in Fig.34 that the longitudinal crack propagated along the compression side of the beam and turned through  $90^\circ$  to propagate out to the tension surface. As the longitudinal crack slowed down, the off-shoots towards the tension surface tended to twist out of the (010) plane into (011) planes as can be seen near the leading edge of the fracture in Fig.36.

The cleavage fractures through the cross-section of the beams very often originated at the longitudinal crack. In Fig.37 this type of cross-section cleavage fracture is clearly illustrated and in addition shows the nearly circular pattern of the crack front at one hesitation stage in its propagation.

In some beams, otherwise deforming completely in the 3D mode, there appeared at strains around 2% and above, what seemed to be faint bands of 2D slip near the neutral axis. They did not seem to propagate from either the tension or compression surface, but originated, presumably, at the sides of the beam near the neutral axis. Upon examination after unloading, it was evident that in fact they did not propagate through the beam, but were limited to a

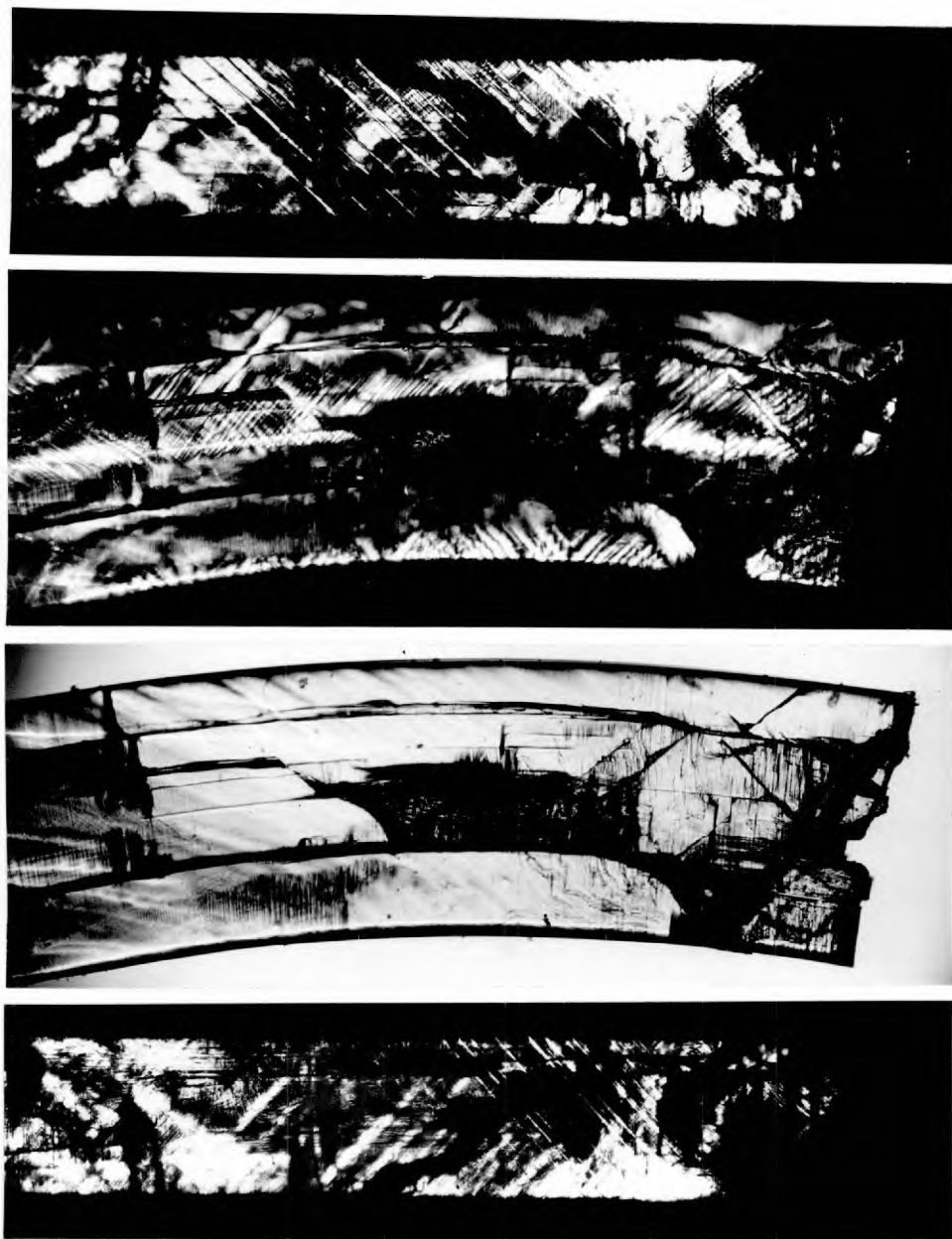


Fig 38. Showing a part of a deep beam deformed to 5% strain in 3D bending. Top: birefringence seen at tension surface showing lattice rotation. Bottom: birefringence on the compression side. Middle 2: Polarised and ordinary transmitted illumination from the side. One of the inner knife edges was in contact with the beam near the right end. x 17.

narrow region at each side. One beam in which bands of this nature were very clearly defined was a beam which did not fracture during loading, although a strain in excess of 5% was reached. Unfortunately, upon unloading, this beam shattered, and is shown in this condition in Fig.38. Although the slip pattern was undoubtedly modified by the numerous cracks there is visible evidence in the birefringence picture from the side that the 2D slip near the neutral axis existed before any cracking occurred. The 2D slip that can be seen at the compression surface formed during unloading. The rotation of the lattice can be seen in the birefringence pictures taken from the tension side and the compression side of the beam. The magnification of all the reproductions in Fig.38 is identical and consequently, the observable difference in width at the tension and compression surfaces is a real difference indicating the distortion of the cross section. The angle  $\theta$ , defined in II.6, is measureable in these pictures and corresponds within experimental error to the values that might be predicted on the basis of the calculated strain, being approximately  $48^\circ$  on the tension side and  $42.1^\circ$  on the compression side.

The beams that failed under load usually shattered into several pieces. However, some of these broke into only two or three pieces. The breaks in these beams very often roughly followed a (101) plane outwards from the inner knife-edges. Longitudinal cracks were invariably present in the remaining pieces, but because the beams were so full of cracks, it was almost impossible to determine the point at which the fracture had originated, and the planes in which it propagated initially. Occasionally, when a nucleation point could be found, it was common to find two fracture surfaces which often appeared to have formed simultaneously. However, it was interesting to note that in almost every beam that failed under load, the fracture originated in the neighbourhood of, or just outside the inner knife-edges, in the region where there was the additional simple shear stress. There frequently was some slip in the 2D mode in this region, but whether the fracture actually originated at the intersection of slip in this mode with slip in the 3D mode, as reported by Keh, Li and Chou (1959), and Washburn, Gorum and Parker (1959), was not determined.

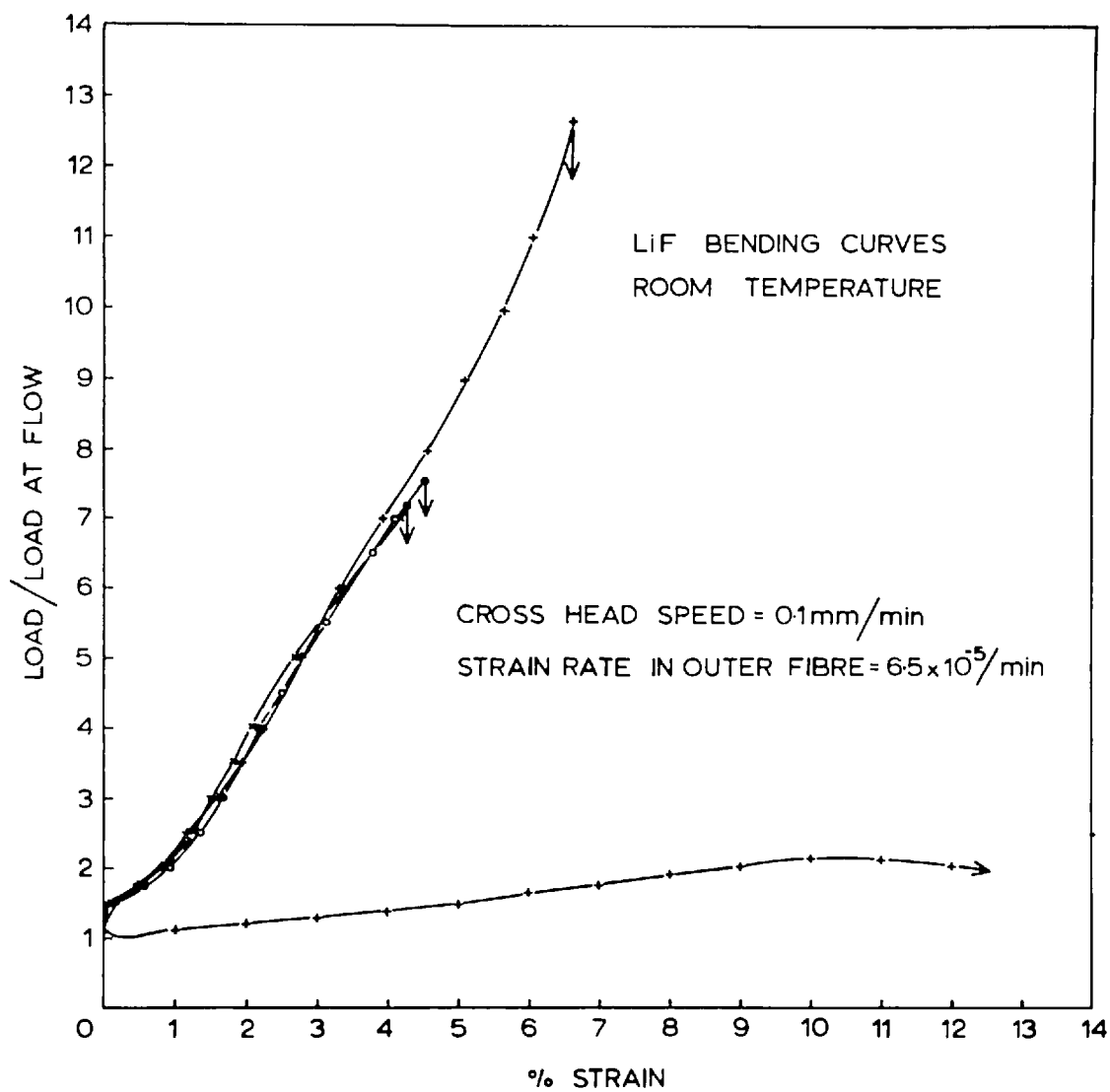


Fig 39. Load-strain plots for beams of LiF.



Fig 40.  
Sawn cross-section of a  
beam of LiF strained 13%  
with slip entirely of the  
2D mode. x 30.

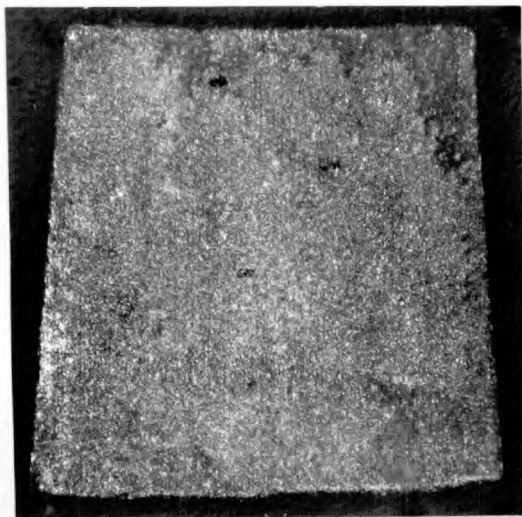


Fig 41.  
Sawn cross-section of a  
beam of LiF strained 7%  
with slip entirely of the  
3D mode. x 30.

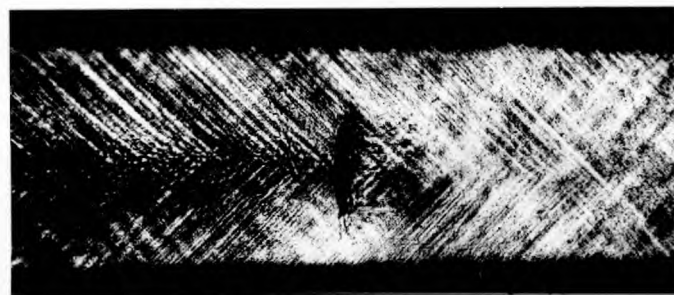
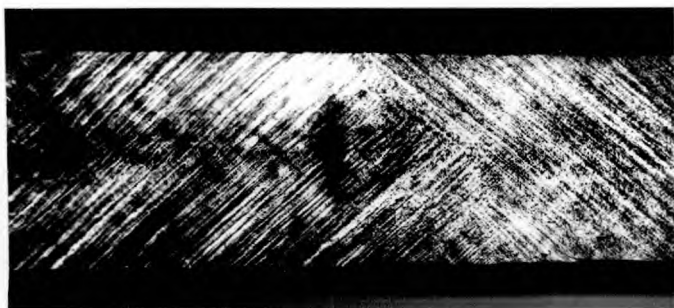


Fig 42.  
The birefringence observed  
at the tension surface  
(upper) and compression sur-  
face (lower) of the same  
beam pictured in Fig 41.  
Note the lattice rotation  
and change in width.

### III.5 Bending of LiF and NaCl

Since it proved impossible to strain beams of MgO sufficiently to permit the checking of the predicted distortion of the cross-section, other materials with the same slip systems were turned to. Two materials that were readily available and proved suitable were LiF and NaCl.

Beams of both LiF and NaCl were cleaved and polished until etching revealed very few if any fresh dislocation sources in the beam surfaces. No further preparation was carried out before bending.

#### (a) LiF

The beams of LiF were all bent in the four-point bending jig at the same strain rate as were the beams of MgO. Up to about 0.5% strain, slip in both the 2D and 3D modes was operative, but at approximately this stage in the deformation, one mode of slip virtually ceased operating and the other became dominant. The resulting load-strain plots are reproduced in Fig.39. The very much higher rate of work hardening for beams deformed with 3D slip, compared to that of beams deformed with 2D slip, is immediately obvious. The linearity of the plots for beams deformed in 3D bending and their very close agreement is striking.

Some of the beams were sawn through after photographs of sufficient of their lengths had been taken to permit accurate estimates to be made of the curvatures, and hence the strain, anywhere along beam. The sawn cross-sections of the beams were photographed; one of a beam deformed in 2D slip is reproduced in Fig.40, and one of a beam deformed in 3D slip in Fig.41. The extreme distortion of the cross-section of the beam deformed with 3D slip contrasts markedly with the still rectangular form of the cross-section of the beam deformed to a much higher strain with 2D slip. The lattice rotation is evident in the birefringence pictures taken of the tension and compression sides of the same beam as that pictured in Fig.40, and reproduced in Fig.42. The measured radius of curvature of the neutral axis of the beams for the region in which the pictures in Fig.42 were taken was 12.84mm and the beam depth was 1.74mm. On the basis of the model of II.6, the angle  $\theta$  at the tension and compression surfaces

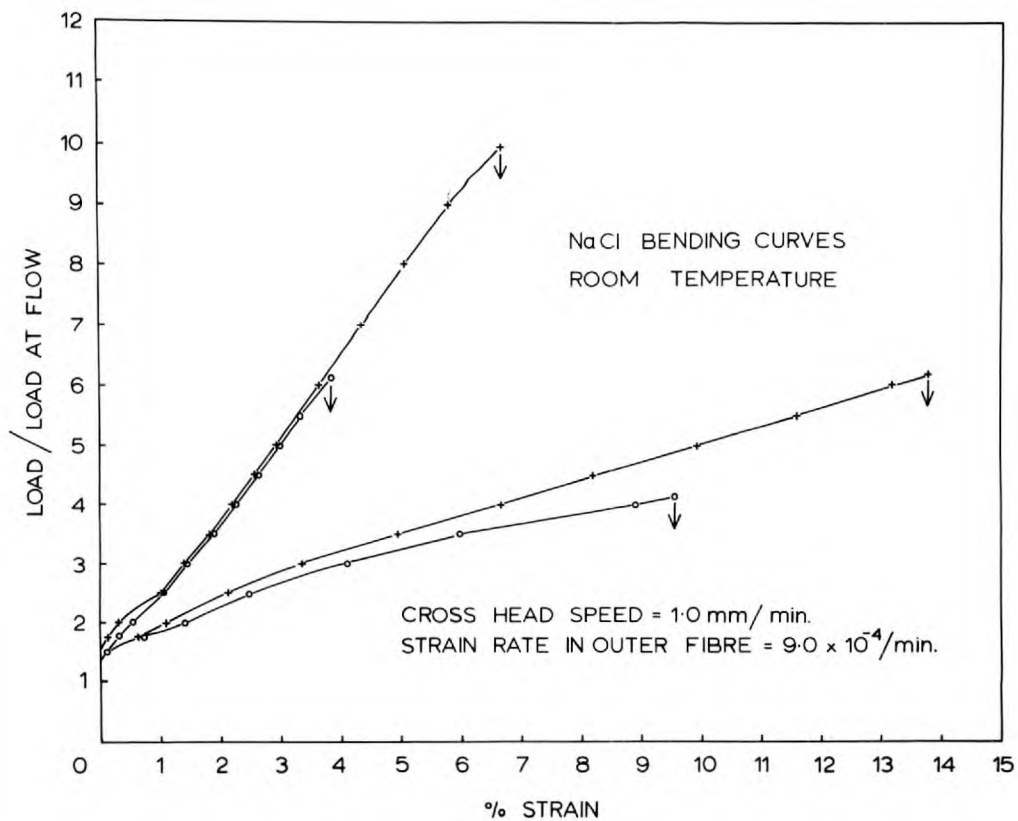


Fig 43. Load-strain plots for NaCl.

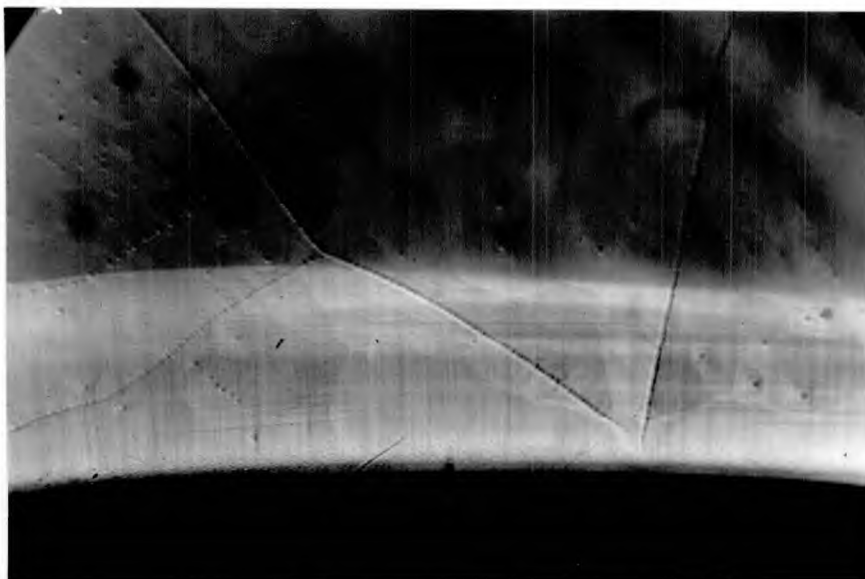


Fig 44. A kink along a compression edge of a beam of NaCl.  $\times 140$ .

should be  $49.3^\circ$  and  $41.1^\circ$  respectively. The measured values of these angles are approximately  $49.2^\circ$  and  $41.4^\circ$ . These measurements were taken from birefringence observations and as a result should not indicate quite as large a change in  $\theta$  as is predicted by the theory. This is a result of the birefringence picture being an integration of the stress pattern over a finite depth of the beam, and since the rotation of the lattice decreases as the neutral axis is approached the integrated photoelastic effect would indicate a smaller change in  $\theta$  than in fact occurred at the surface.

#### (b) NaCl

The beams of NaCl, when strained at the same rate as were the beams of MgO and LiF, deformed with a combination of 2D and 3D slip at the outset of plastic flow. At approximately 0.5% strain the 3D slip invariably stopped forming, save along the edges in some beams, permitting the 2D slip in every case to become the dominant mode of deformation. However, an increase in the strain rate of a factor of ten changed this behaviour. The beams then displayed the same characteristics as LiF with either 2D or 3D slip becoming the completely dominant mode after about 0.5% strain. The load strain curves obtained are plotted in Fig.43 and show the same marked difference between beams deformed in 2D slip and beams deformed in 3D slip. The distortion of the cross-section of beams deformed in 3D slip was obvious, as in the beams of LiF.

Some of the beams of NaCl were deformed at strain rates another factor of ten higher. At these strain rates kinks of the type described by Stokes, Johnston and Li (1962) were very often formed along both the tension and compression edges of the beams. Upon examination these usually proved to occur only in orientations that modified the glide planes to anticlastic surfaces. A picture of a kink formed along the compression edge of a beam is reproduced in Fig.44. Although it was difficult to make detailed observations at the strain rates employed, it appeared that the kinks did not form suddenly, but simply developed throughout the deformation. Normally, 3D slip became the dominant mode of deformation in the kink, while in the body of the beam, 2D slip became the dominant mode. The boundary between the two slip modes, formed in this way, is identical to the

boundaries formed in squat NaCl compression specimens, reported originally by Brilliantow and Obreimow (1937), and later by Taylor and Pratt (1958). The same block slipping and resultant boundaries were observed in squat compression specimens of KCl by Vaughan, Leivo and Smoluchowski (1958). Stokes, Johnston and Li (1961) and (1962) also observed kink boundaries in bent beams of NaCl, LiF and MgO, but report seeing these only on the tension side of the beams.

### III.6 High Temperature Bending of MgO

#### (a) Factors Effecting Dislocation Motion in MgO

May and Kronberg (1960) found that the critical resolved shear stress of MgO measured in 3-point bending at temperatures below 800°C could be significantly reduced by air quenching the specimens from 1250°C before testing. The critical resolved shear stress of crystals prepared in this way decreased rapidly with testing temperature to a minimum at about 500°C, but then increased until at about 800 to 900°C it was the same as that for crystals tested in the as-received or slowly-cooled state. The authors suggested that the stress reduction brought about by quenching was the result of the impurities being frozen in solution, and that as the impurities precipitated out again at temperatures above 500°C the effect of quenching was nullified. The local stress minimum reached in their tests was not the same for all specimens, but appeared to be dependant on impurity content. However, the results of their tests on specimens in the as-received state indicated that the critical resolved shear stress decreased almost linearly with temperature up to the limit of their tests, about 900°C. Both Hulse and Pask (1960), and Phillips (1960) reported a similar reduction in the yield stress up to about 1000°C for compression tests on as-received specimens and at higher temperatures a levelling off of the stress to a relatively constant value. Thompson (1960) observed much the same stress behaviour in bending tests, but in addition found a significant difference in the absolute value of the stress recorded for specimens with different impurity contents even at temperatures above 1000°C.

The important effect of even a relatively small impurity content was demonstrated by Gorum, Luhman and Pask (1960) who reported nearly a four-fold reduction in the critical shear stress at room temperature when the Fe impurity level was reduced from 30 p.p.m. to 10 p.p.m. Furthermore they observed significantly different effects for specimens annealed at 2000°C before testing from those observed for specimens annealed at 1000°C. Stokes (1962) found specifically that annealing at temperatures as high as 2000°C was necessary to dissolve some of the precipitate particles in MgO that might otherwise act as dislocation sources at high stresses.

Extensive studies of the factors effecting dislocation motion in LiF were carried out by Gilman and Johnston (1957,58,59(a), 60) and Johnston and Gilman (1959,60). Johnston (1962), summarising the observed effects of heat-treating on impure LiF compared these results to the data available for MgO and predicted critical shear stresses as low as 200 to 250g/mm<sup>2</sup> in pure MgO.

All these results indicate the importance of the part played by impurities in the deformation of MgO, Johnston's prediction amounting to a fifty-fold reduction of the critical resolved shear stress observed for most of the material used in the work reported herein. Thus it may be assumed that the room temperature behaviour of MgO differed from that of LiF and NaCl, at least in part, because of the influence of the impurities. Since no very pure MgO was available it was decided as an alternative method of reducing the effect of the impurities to perform the bending tests at as high a temperature as was practically possible. Furthermore, this would have the added advantage of increasing the ratio of test temperature upon melting temperature to a value higher, but, according to the observations of Johnston, Stokes and Li (1959(b)), more closely comparable to the homologous temperatures at which the LiF and NaCl were deformed.

To provide a high temperature testing facility the molybdenum wound furnace and bending apparatus described earlier were built.

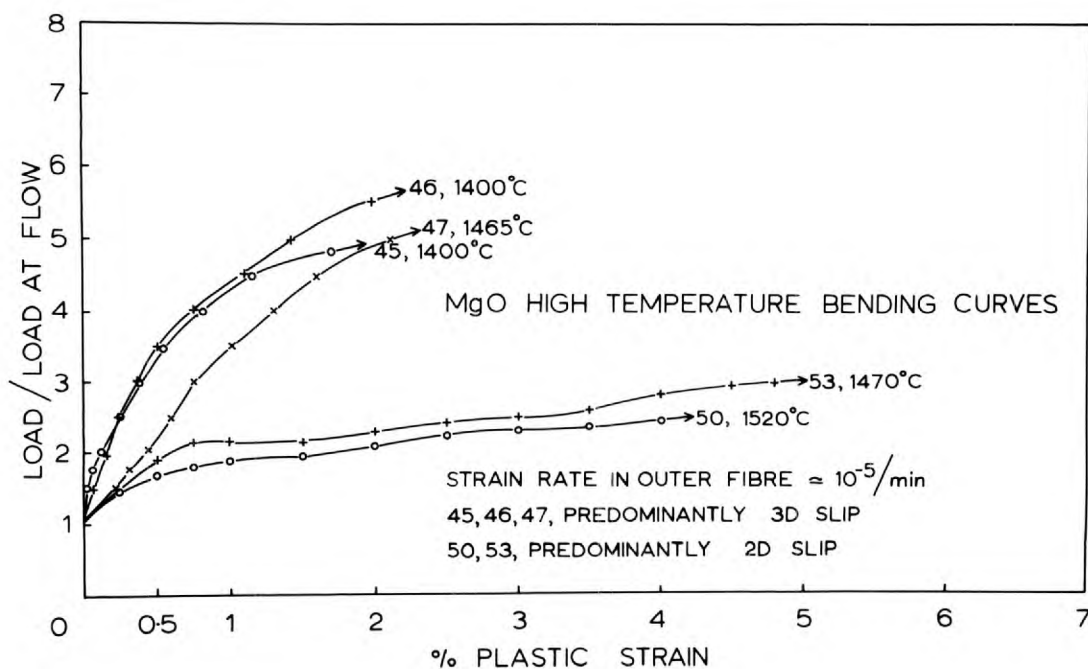


Fig 45. Load-strain plots for beams of MgO bent at the temperatures indicated.



Fig 46. A double kink in a beam of MgO deformed at  $1500^{\circ}\text{C}$  at a strain rate of approximately  $10^{-5}/\text{min}$ . The slip in the kinks is in the 3D mode.  $\times 200$ . (The tension surface)

### III.6 (b) Results of High Temperature Bending Tests

Several bending tests were carried out at temperatures from about  $1350^{\circ}\text{C}$  to  $1550^{\circ}\text{C}$ . A few tests were performed at temperatures up to  $1690^{\circ}\text{C}$ , but it was found at temperatures much over  $1550^{\circ}\text{C}$ , that the rate of erosion of the silicon nitride cylinder became significant. In addition, where it was in contact with alumina, the silicon nitride tended to react, a thin glaze being produced on the surface. Furthermore, the alumina knife-edges reacted slightly with the MgO beams at the highest temperatures, and undoubtedly deformed significantly under the applied load. As a consequence, the machine could not be assumed to be hard at these very high temperatures, and it was necessary to correct the strains computed from the output chart, to agree with the measured curvature of the deformed beam. However, the strains calculated for beams tested at temperatures up to about  $1550^{\circ}\text{C}$  agreed fairly well with the observations made after the beam had been removed from the machine.

The beams were polished, loaded into the machine, and heated to the testing temperature over a period of about one and one-half hours. The knife-edges were then driven together at a rate giving the same strain rate at the outer fibres of the specimen as was used in the room temperature tests. When the test was completed the cell was racked out of the furnace and the beam removed. None of the beams were deformed to fracture.

The load-strain plots of some of the beams are reproduced in Fig.45. These curves show that there is a significantly greater difference in the work hardening rates associated with the two slip modes at this temperature, than at room temperature. Furthermore, the beams deformed almost exclusively in one mode or the other, there being no evidence of the generally mixed slip that normally occurred in polished beams at room temperature. In these respects the high temperature behaviour of MgO corresponded much more closely with that of LiF and NaCl deformed at room temperature.

Kinking was frequently observed along both the tension edges and compression edges of these beams in spite of the very low strain



Fig 47. A kink along the tension edge of a beam of MgO deformed at 1500°C at a strain rate of approximately  $5 \times 10^{-5}$ /min. The slip in the kink is in the 2D mode: Upper, from the tension surface, and lower from the side. x 40.

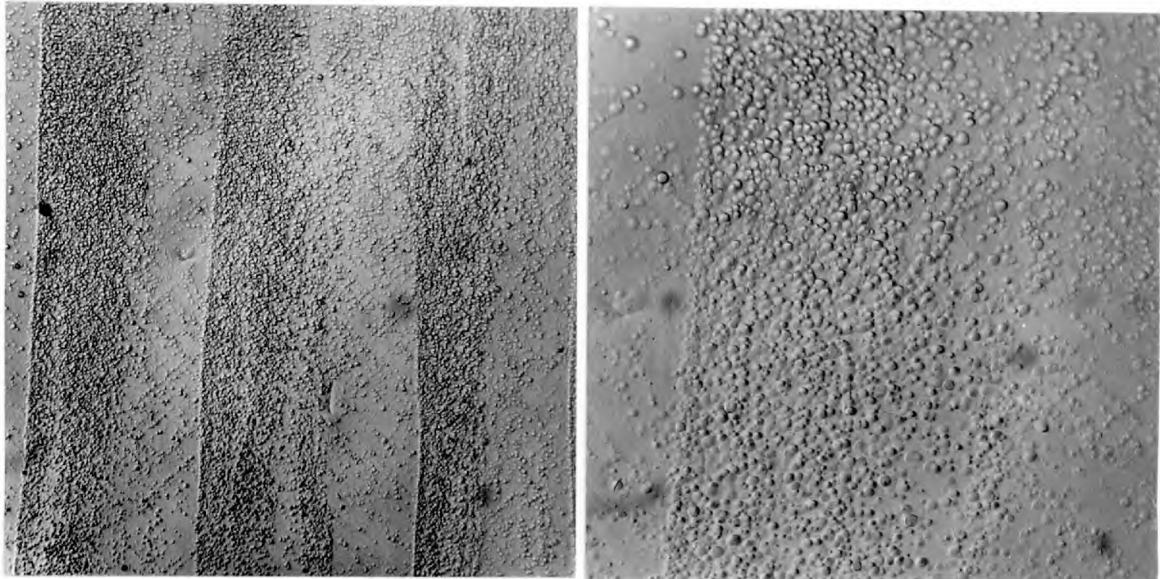


Fig 48. The etched side of a beam deformed at about 1500°C; on the left x 140 and on the right x 385.

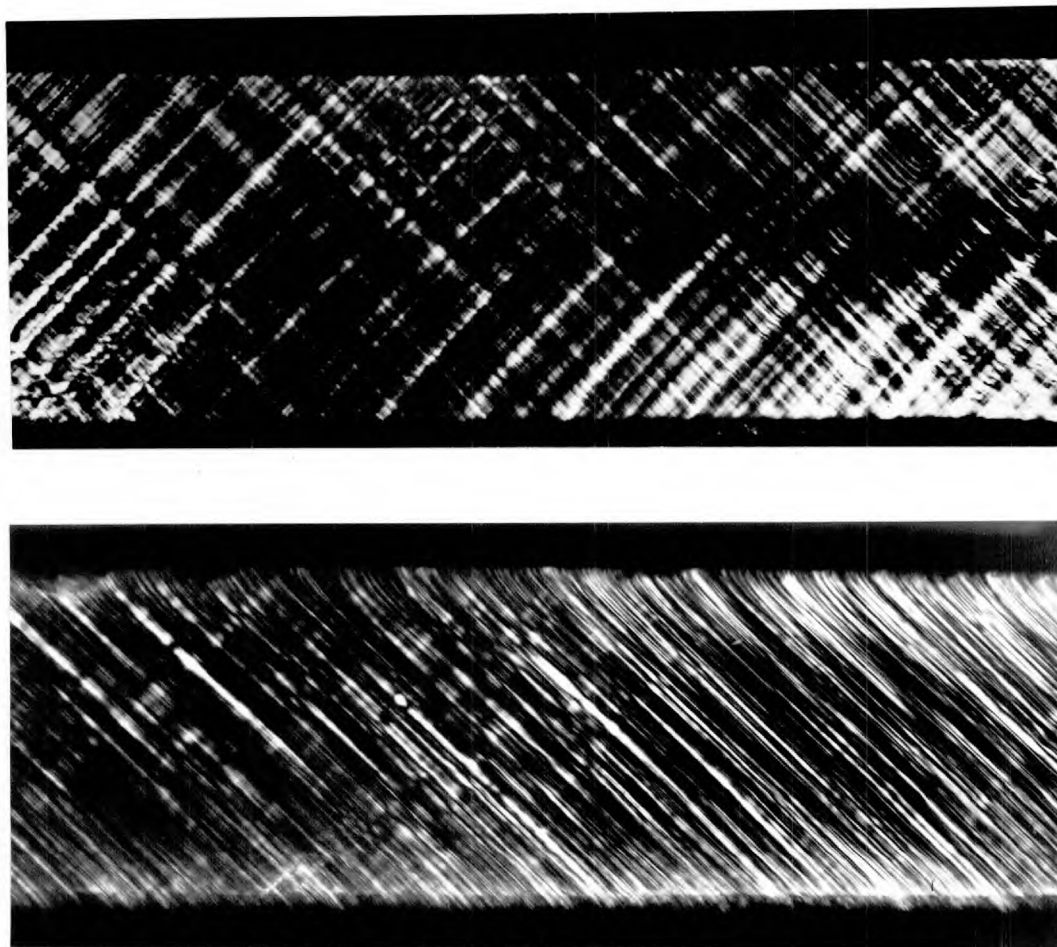


Fig 49. Birefringence in beams strained to the same extent: Upper, the compression surface of a beam strained at room temp., x32: Lower, the tension surface of a beam strained at 1450°C, x28. Note the lattice rotation in both.

rates, approximately  $10^{-5}$ . Normally the slip in the kinked portion was in the 3D mode while the slip in the main portion of the beam was entirely in the 2D mode as can be seen in Fig.46. However, on one occasion quite a large kink occurred in a beam deformed entirely in the 3D mode save for the kinked material which deformed in the 2D mode. A photograph of this kink is reproduced in Fig.47. Neither of the kinks shown in Figs.46 and 47 were simple straight kinks, but in the case of Fig.46 there were two parallel kinked regions while in Fig.47 a portion of the kink appears to have branched away from the main part.

After being deformed at these high temperatures the beams no longer etched properly. The pits produced at dislocations varied greatly in size and shape, but were generally shallow and diffuse, typical more of those observed in as-received material. A comparable change in the etching characteristics of material after heating was reported by Gorum, Luhman and Pask (1960) and by Stokes (1962).

In the resulting etch-pit patterns the density of the pits coupled with their shallowness and irregularity of shape made it difficult to establish the traces of any specific slip band. However, in beams not strained too heavily it was possible to see specific slip bands as in Fig.48. Very often there was evidence of cross gliding of screw dislocations (see Fig.48) at the surface as observed by Gilman and Johnston (1957) in LiF, indicating a substantial reduction in the hardening effect of the impurities at these elevated temperatures.

The birefringence pattern observed in the deformed beams was a great deal more diffuse than that observed in the MgO specimens deformed at room-temperature. This difference can be readily seen by comparing the two pictures in Fig.49.

From the observations made on most of the high temperature specimens it might be concluded generally that not only was virtually all the slip in a single mode, but in addition it occurred in a single direction. Furthermore, the slip bands formed at these temperatures were no longer as well defined as those formed at room

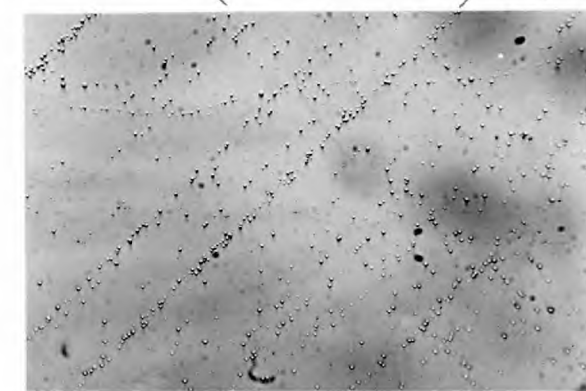
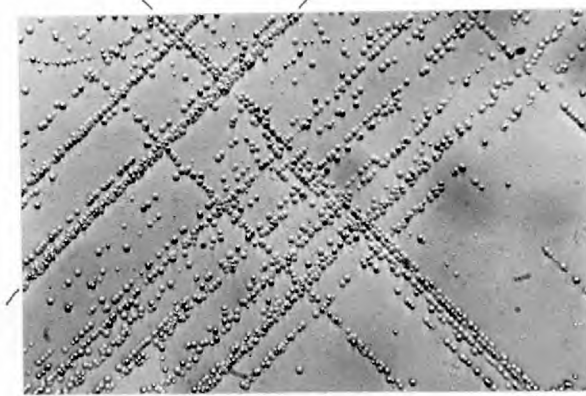
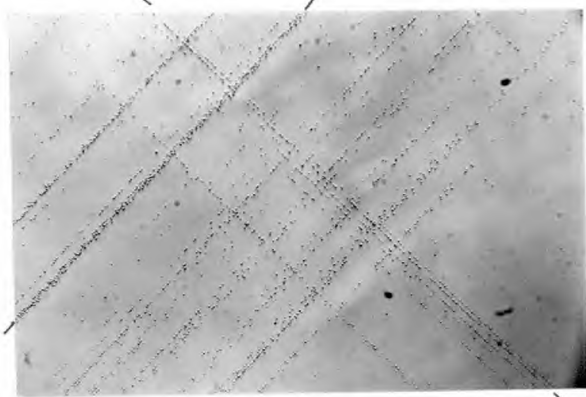


Fig 50. A beam lightly deformed at room temperature.  
 (a) etched immediately after deformation.  
 (b) the etching continued after annealing at  $1150^{\circ}\text{C}$   
 for one hour. (The same location as (a))  
 (c) polished after (b) and then re-etched.  
 (The same locations as (a) and (b)).x385.

temperature. The dislocation density within the bands, as far as could be discerned from the etch-pit patterns, was significantly lower, and the general appearance of the bands resembled more closely the broader and less dense slip bands observed in relatively pure NaCl and LiF deformed at room temperature. However, this may not indicate a genuine difference in dislocation density since simply heating a beam of MgO that had been slightly deformed at room temperature to a temperature little in excess of  $1000^{\circ}\text{C}$  greatly reduces the number of etch pits in the slip lines. In Fig.50 the etch pit pattern on a beam slightly deformed at room temperature is compared with the results of a continued etch in the same area after annealing the specimen at  $1150^{\circ}\text{C}$  and finally with the etch pit pattern in the same area after a polish and re-etch. In the final picture it can be seen that all the pits which had become flat-bottomed after the anneal were no longer present. It might be argued that the final picture indicates the density of slip dislocations and all the other pits in the original picture were simply debris pits of the sort described by Johnston and Gilman (1960) in LiF, and by Davidge and Whitworth (1961) in NaCl.

### III.7 Summary

At room temperature, beams of LiF and NaCl bent with slip almost exclusively in the 2D or 3D mode after a brief period of mixed slip at the outset of plastic flow. The work hardening rate for the beams deformed in the 3D mode was very much higher than that for the beams in the 2D mode and was very nearly linear. For MgO there was generally mixed slip in the beams deformed at room temperature. Occasionally beams deformed almost exclusively in the 2D mode, but to obtain beams significantly strained with slip exclusively in the 3D mode it was necessary to introduce appropriate slip sources. The work hardening rate of the beams deformed in the 3D mode was greater than that of beams deformed in the 2D mode, the difference being accounted for in part by significant elastic strain energy which was sometimes partially released with explosive multiple fractures.

On the other hand, the beams of MgO deformed at high temperatures behaved very much more like those of LiF and NaCl at room temperature, one slip mode usually completely dominating, and all the slip often in a single direction. The difference in the work hardening rates associated with the two slip modes was substantially greater than that observed at room temperature.

In all the bend tests significantly more work was necessary to achieve the same strain if the bending slip was in the 3D mode as opposed to the 2D mode.

## CHAPTER FOUR    A PHOTOELASTIC EXAMINATION OF CROSS-SECTION STRESSES

### IV.1 Introduction

An investigation of the stresses in a beam deformed plastically with 3D slip was obviously important if the marked differences in the behaviour of beams deformed by the two modes of slip were to be understood. Since MgO is optically active when stressed, a photoelastic examination of the stresses proved possible and was carried out.

### IV.2 The Photoelastic Method of Investigation

The first observation of birefringence produced by the application of stress to an isotropic transparent solid was made by Brewster in 1816. In 1841 Neumann investigated the phenomenon further and developed a theory on the basis of strains, and in 1852, Maxwell, apparently unaware of Neumann's work, developed a theory of precisely the same form on the basis of stresses. However, the photoelasticity of stressed transparent crystals was used but little in the investigation of the mechanical properties of such crystals until Obreimow and Schubnikoff (1927), and later Brilliantow and Obreimow (1933) employed it in their studies of the deformation of NaCl. The most comprehensive work directly investigating the relation between the observed birefringence and the state of stress in a plastically deformed solid is that of Nye (1949a,b) on AgCl.

In recent years the investigation of the propagation and orientation of slip in transparent crystals using stress birefringence observations has become a standard technique. It has proven especially suited to the crystals of the rocksalt structure that cleave on  $\{100\}$  planes and slip on  $\{110\}$  in  $\langle 1\bar{1}0 \rangle$ , since observations can be made through cleaved faces, normal to the Burgers vector, for slip in any of the twelve systems. Its sensitivity as a qualitative tool in the observation of slip was indicated by Mendelson (1961), who reported observable birefringence only a few dislocations back from the leading edge of glide bands in NaCl.

The use of stress birefringence as a quantitative tool in the determination of the direction and magnitude of stress or strain in plastically deformed crystals is a good deal more complicated and consequently has not been as widely employed. Stepanow (1934) made quantitative measurements of the stress in AgCl using photoelastic techniques as did Brilliantow and Obreimow (1933, 1934, 1937) in NaCl. Nye (1949a,b) contributed greatly to the quantitative use of stress birefringence in plastically deformed materials with his analysis of AgCl, and subsequently, he with others, Nye, Spence and Sprackling (1957), Sprackling (1959), and Stowell (1962), have profitably applied the technique in their investigations of the longitudinal stresses in bent beams, and the stresses and strains in kinked crystals.

#### IV.3 Photoelasticity in Bent Beams

Before any analysis of the stresses in a three dimensional stress system can be carried out using photoelastic techniques, two important assumptions must be made. If the directions of the principal stresses are to be calculated, it must first be assumed that at all points on the path of any ray of light passing through the specimen, the directions of the principal axes of the indicatrix remain the same, and that the direction of observation corresponds with one of the axes. It must further be assumed that the magnitudes of the principal stresses normal to the direction of observation be very nearly uniform along the path of any ray of light, if the difference in the magnitudes of the principal stresses is to be calculated with any accuracy.

Now, if the stresses in the cross-section of a beam are to be examined by longitudinal birefringence observations, the first assumption implies that one of the three principal stresses,  $P$ ,  $Q$ , or  $R$ , must, at every point along the beam, correspond with the  $z$ -axis, the direction of observation. The second assumption implies that none of the principal stresses may vary from point to point along any line parallel with the  $z$ -axis. These assumptions immediately impose the restrictions that the beam be deformed very little so that the curvature remain small, and

that the part of the beam under observation have a nearly homogeneous distribution of slip along its length. Fortunately, slip in the 3D mode tended to be far more evenly distributed than slip in the 2D mode, permitting both assumptions to be made.

The greatest experimental difficulty arose in attempting to cut out suitable sections from deformed beams for observation. Beams strained as little as 0.2% with 3D slip tended to crack on longitudinal (010) planes and diagonal (101) planes during sawing, some completely shattering when simply touched by the wire. Thus it proved necessary to use specimen beams that had fractured cleanly on an (001) plane during testing, and to attempt to estimate the optical path length for which the above conditions were met in each case.

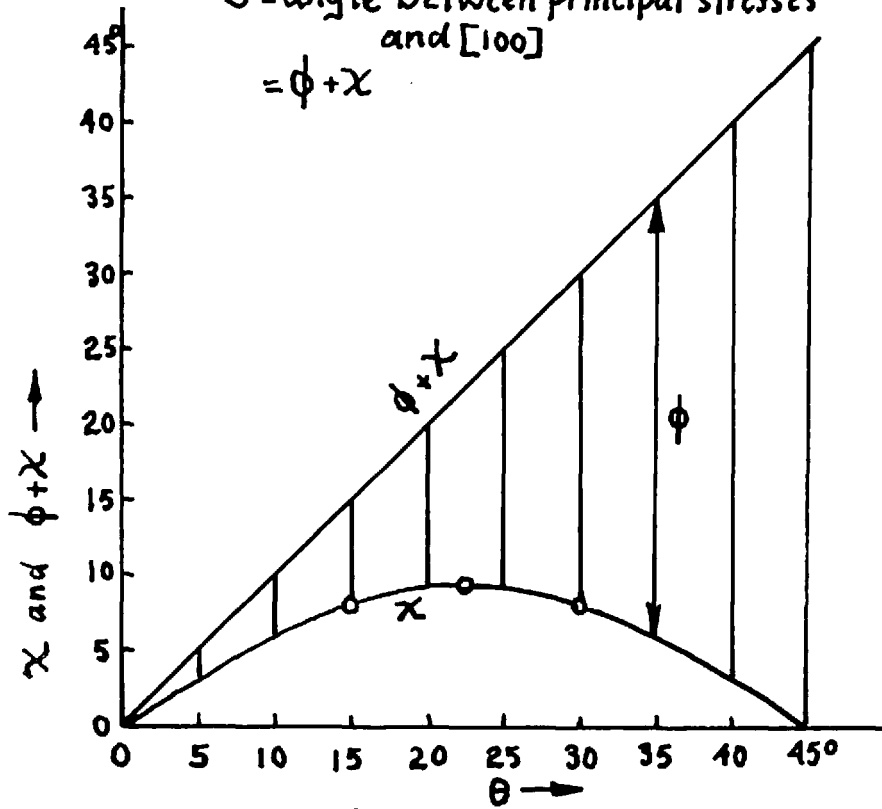
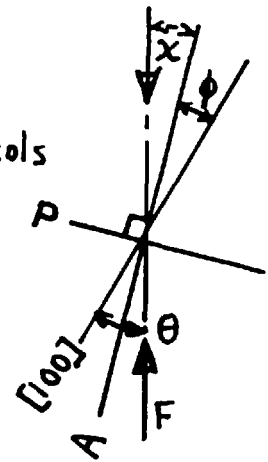
#### IV.4. Principal Stress Directions

When a perfectly isotropic solid is stressed, and examined with transmitted polarised light, the directions of the principal stresses in the plane normal to the direction of observation are the same as the directions of the analyser and polariser at every point of extinction, unless the principal stresses at the point are equal, or zero. However, in stressed MgO the directions of the principal stresses at any point in an isoclinic fringe do not generally correspond with the direction of the analyser and polariser. They differ from this direction by an angle  $\chi$  for all stress directions, save those of elastic symmetry in the plane considered.

In Fig.52 a plot of the variation of Young's Modulus with direction in a (001) plane, calculated with the equations of Sokolnikoff (1956), is given for one quadrant. From simple crystal symmetry it is readily shown that the only axes of elastic symmetry in (001) are  $[100]$  and  $[110]$ .

Holister (1962) measured  $\chi$  for the simple case of compressive uniaxial loading applied in a direction inclined to the  $[001]$  direction in the (100) plane, at an angle  $\theta$ . The specimens were prepared by sawing bars from a cleaved plate so that the test pieces were bounded by

$\phi$  = angle between  $[100]$  and nicols  
 $\chi$  = angle between principal stresses and nicols  
 $\theta$  = angle between principal stresses and  $[100]$   
 $\theta = \phi + \chi$



Plot of  $\chi$  vs.  $\theta$  assuming  $\chi$  varies sinusoidally with  $\theta$ .

Using Holister's data,  $\chi = 9.25 \sin 4\theta$

Plot of  $\chi$  vs.  $\phi$

$$\phi = \theta - \chi$$

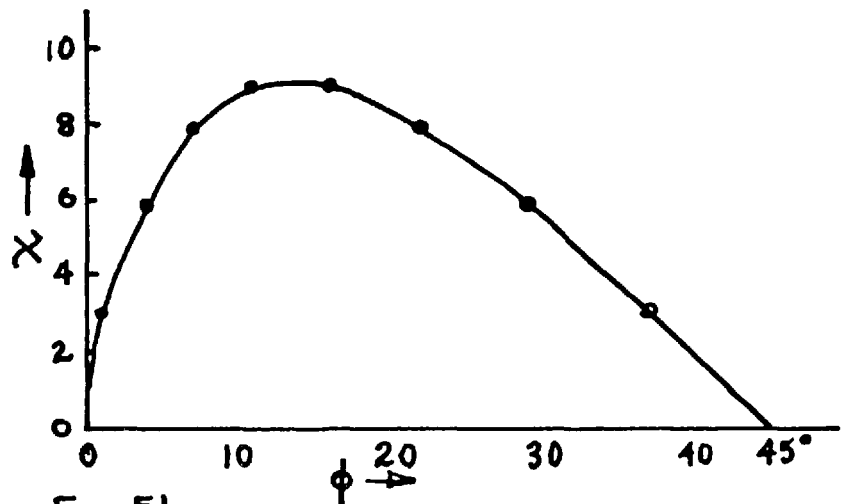


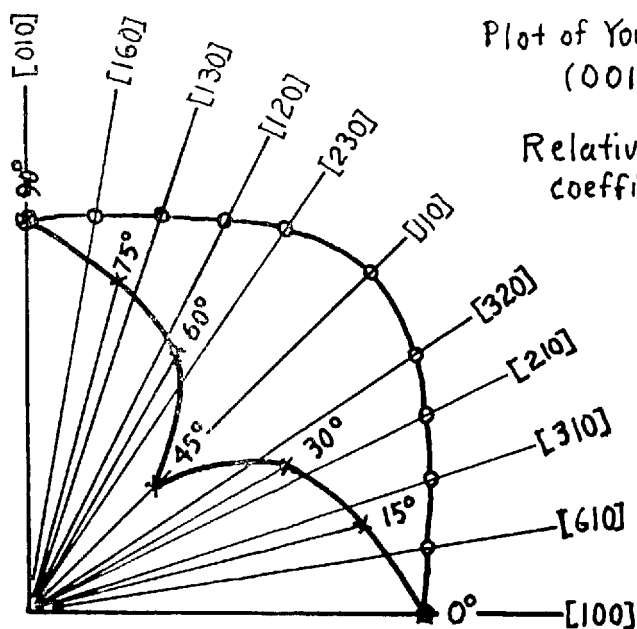
Fig. 51

two cleaved surfaces, two surfaces sawn parallel to the direction in which the load was to be applied, and ends sawn and ground normal to the loading axis. The observations were made in the  $[100]$  direction normal to the cleaved surfaces for  $\theta = 0^\circ, 15^\circ, 30^\circ$  and  $45^\circ$ . As expected,  $\chi$  was 0 at  $\theta = 0^\circ$  and  $45^\circ$ , and was  $8^\circ \pm 1^\circ$  at  $\theta = 15^\circ$  and  $30^\circ$ . It was assumed that  $\chi$  varied smoothly with  $\theta$ ; the approximation that the variation is sinusoidal giving the values graphically represented Fig. 51. The values of  $\chi$  obtained in this way were added to  $\phi$ , the angle between  $[010]$  and the direction of the crossed nicols, to give the direction of the principal stresses along the isoclinics observed for all values of  $\phi$ .

Although, in the problem considered, this simple case of uniaxial loading did not apply, it was assumed that the values of  $\chi$  remained the same for the same values of  $\theta$ , where  $\theta$  is redefined as the smallest angle between the direction of the principal stresses and either of the cube axes in the plane considered. This assumption is based on the fact that at any point both of the principal stresses are inclined to a cube axis at the same angle, and hence, since the same value of  $\chi$  should apply to each stress when acting separately, the same value of  $\chi$  should still apply when they are acting together.

#### IV.5 Principal Stress Magnitudes

In a perfectly isotropic material under stress, the relative retardation of the ordinary and extraordinary rays is a function only of the difference in magnitude of the principal stresses in the plane normal to the ray, and is directly proportional to the optical path length through the stressed material. That is, the values of the stress birefringence coefficient,  $C_1 - C_2$ , is independent of stress directions in an isotropic solid. However, in the case of crystalline solids, including MgO, the stress birefringence coefficient is dependent on both the direction of the principal stresses and the direction in which the observations are made. Giardini and Poindexter (1958), working with synthetic MgO, determined the stress-optical constant and reported the



| $\Theta$       | $W_{mm}$ | $t_{mm}$ | $C_1-C_2$ |
|----------------|----------|----------|-----------|
| $0, 90^\circ$  | 2.42     | 2.83     | 5.11      |
| $15, 75^\circ$ | 2.55     | 2.63     | 4.53      |
| $30, 60^\circ$ | 2.43     | 3.21     | 3.97      |
| $45^\circ$     | 2.30     | 3.00     | 2.35      |

The observations were made through the thickness  $t$  of specimens of width  $W$ , and  $C_1-C_2$  is given in the arbitrary units: fringes  $\times 10^5 / g/mm^2/mm$

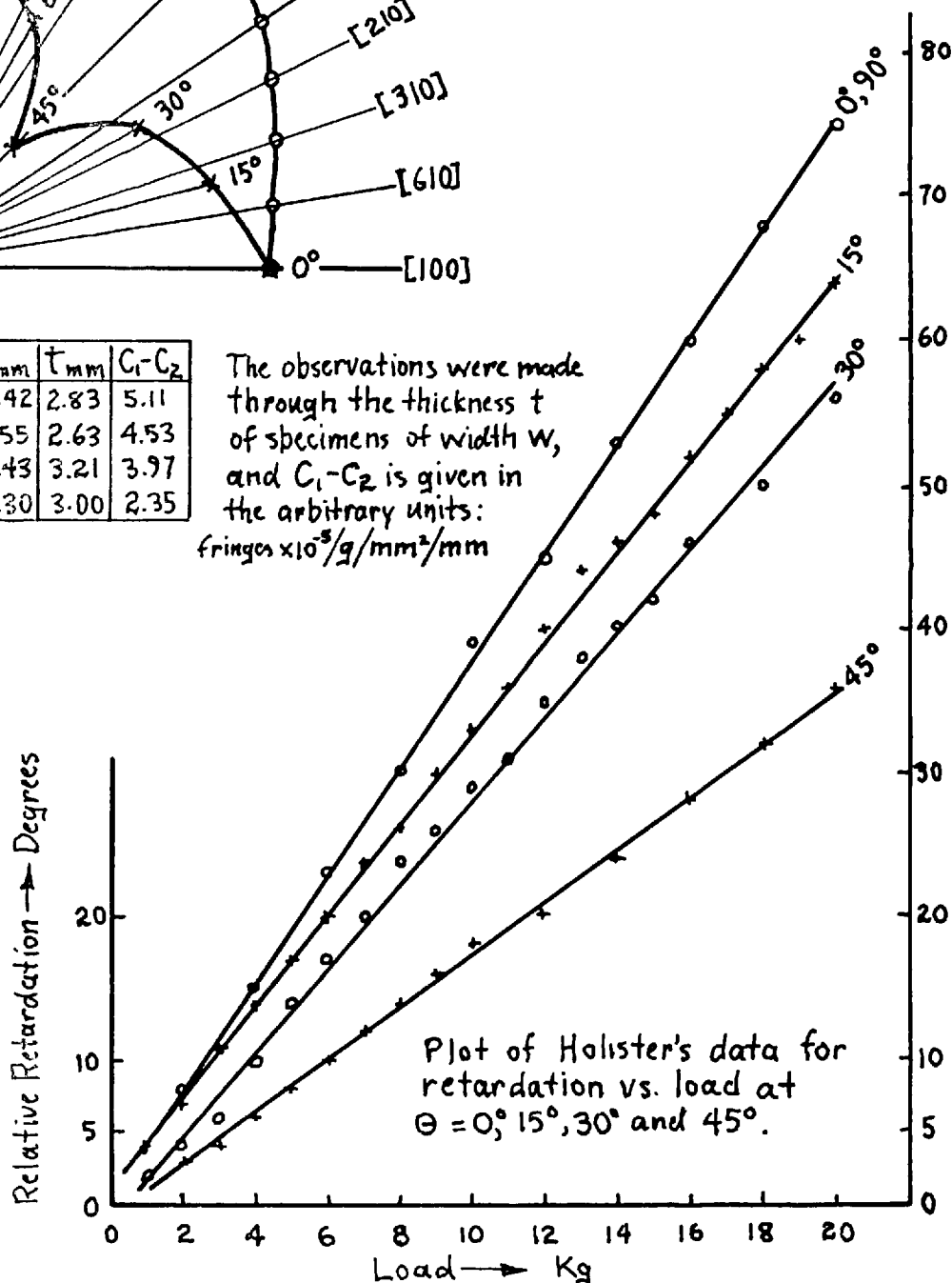


Fig. 52

values  $\pi_{11} - \pi_{12} = -1.25$  and  $\pi_{44} = -.66$ , both given in units of  $10^{-13} \text{ cm}^2/\text{dyne}$ . (The sign convention and the symbols used here are those established by Nye (1957) and not those used by the authors). Furthermore, they separated the values  $\pi_{11}$  and  $\pi_{12}$  giving the values  $\pi_{11} = -0.9$  and  $\pi_{12} = +0.3 \times 10^{-13} \text{ cm}^2/\text{dyne}$ . Using these values in the equations given by Nye (1957), the birefringence coefficients  $C_{11} - C_{12}$  and  $C_{44}$  were calculated and are compared below to those given by West and Makas (1948).

|                   | W. and M. | G. and P. |
|-------------------|-----------|-----------|
| $C_{11} - C_{12}$ | 3.26      | 3.28      |
| $C_{44}$          | 1.64      | 1.73      |

These values serve to indicate how widely the birefringence coefficients may vary with stress direction, although the variation in MgO is extremely small compared to that encountered in AgCl and other solids. Since stresses lying only in the (001) plane are considered to be present in the beams, only the value of  $C_{11} - C_{12}$  is of any direct interest in the present application. However, it does not give any indication of the variation with stress direction of the birefringence coefficient  $C_1 - C_2$  in the (001) plane, but simply gives its value at points where the principal stress directions correspond to the cube axes.

When measuring  $\chi$ , Holister (1962) also measured the relative retardations of the slow and fast rays for the same values of  $\theta$ . He used a  $\frac{1}{2}$  wave plate as a compensator to measure the retardation in the fashion described by Jessop and Harris (1949), and his data are plotted in Fig. 52. From the slopes of the lines, relative values of  $C_1 - C_2$  can be obtained for the values of  $\theta$  investigated. By applying this variation to the values of  $C_{11} - C_{12}$  determined from the data of Giardini and Poindexter (1958) given above, absolute values of the stress-optic coefficient can be calculated at  $\theta = 0^\circ, 15^\circ, 30^\circ$  and  $45^\circ$ .

#### IV.6 Birefringence Observations

Observations of the birefringence resulting from the stress in the beam cross-section were made on the Zetopan microscope. Suitable

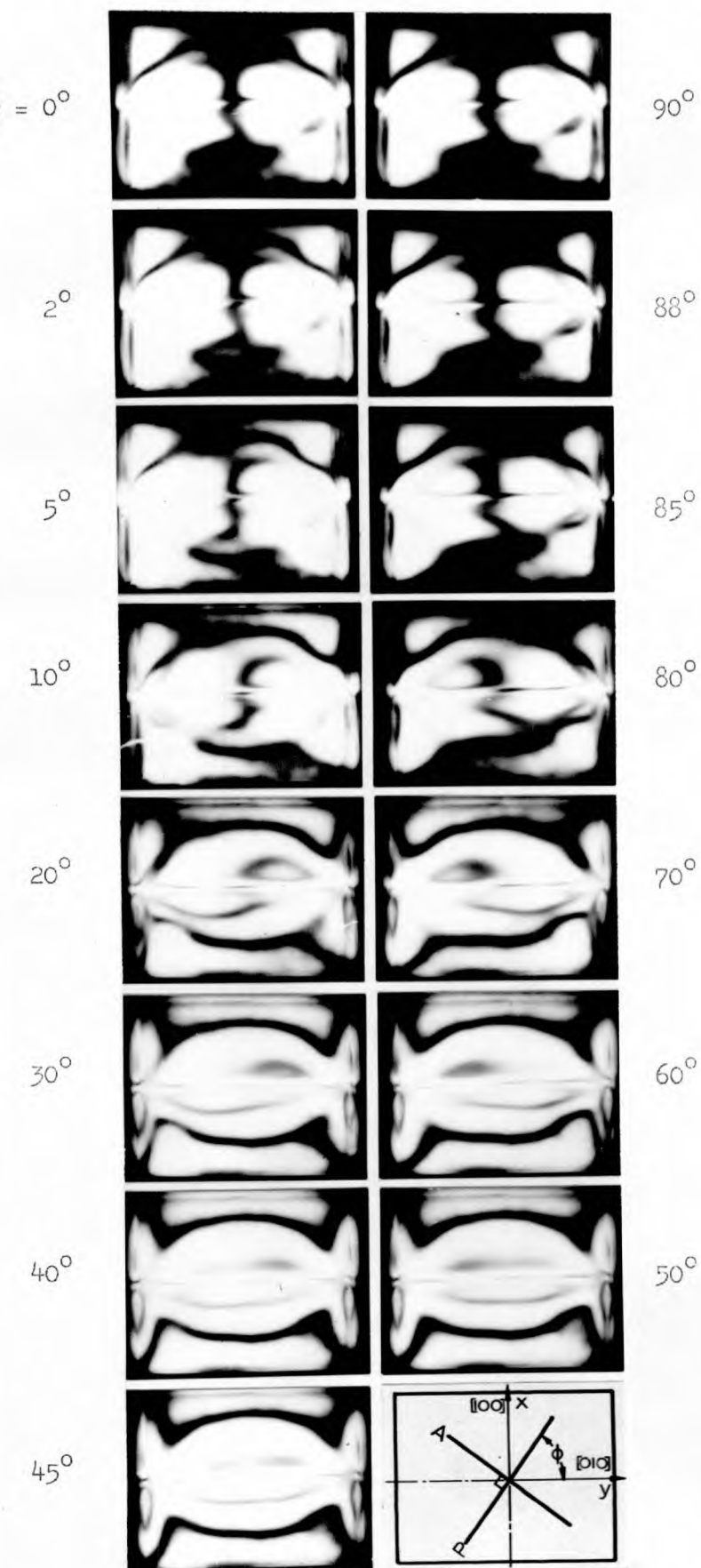
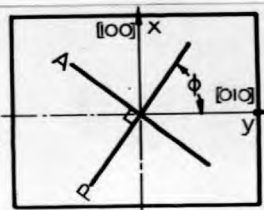
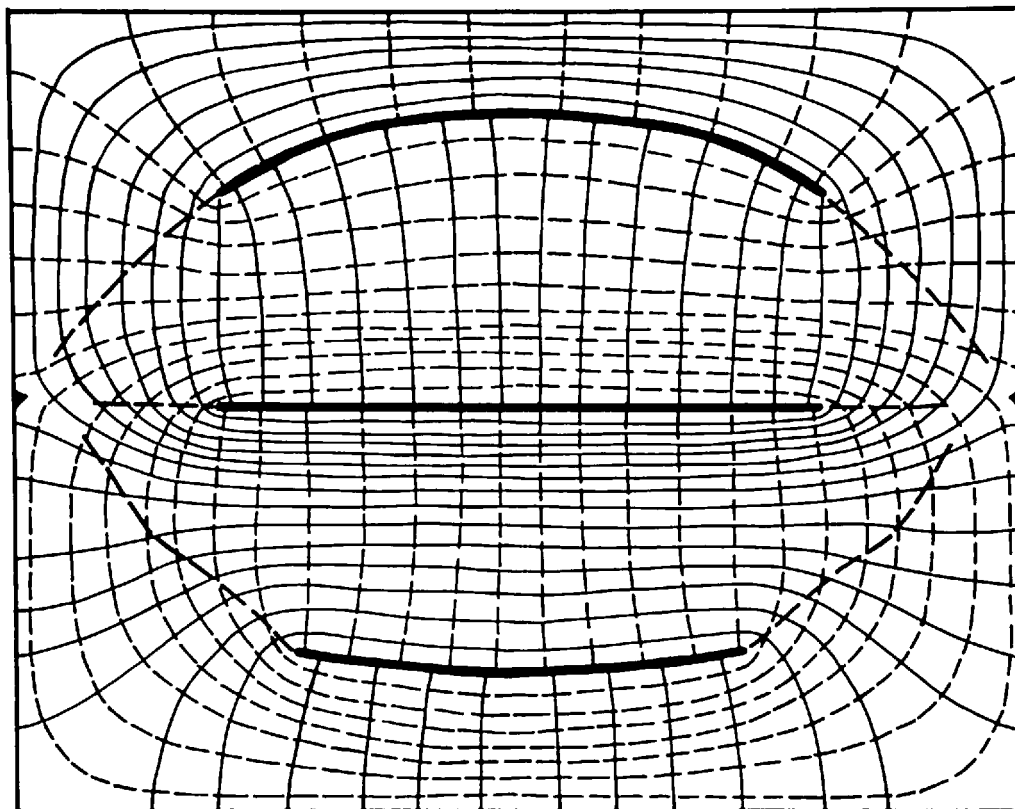


Fig 53.  
The isoclinics observed longitudinally down a beam when the angle between the crossed nicols and the  $[100]$  is that indicated.





LINES OF ZERO STRESS **————** PRINCIPAL STRESS TRAJECTORIES  
 LINES OF ZERO STRESS P SET **-----**  
 DIFFERENCE:  $|P|=|Q|$  **----** Q SET **————**

Fig 54. The principal stress trajectories in the plane of cross-section of a beam deformed approximately 0.3% with slip in the 3D mode. These were determined from the isoclinic fringes shown in Fig 53. The tension surface is at the top of the figure.

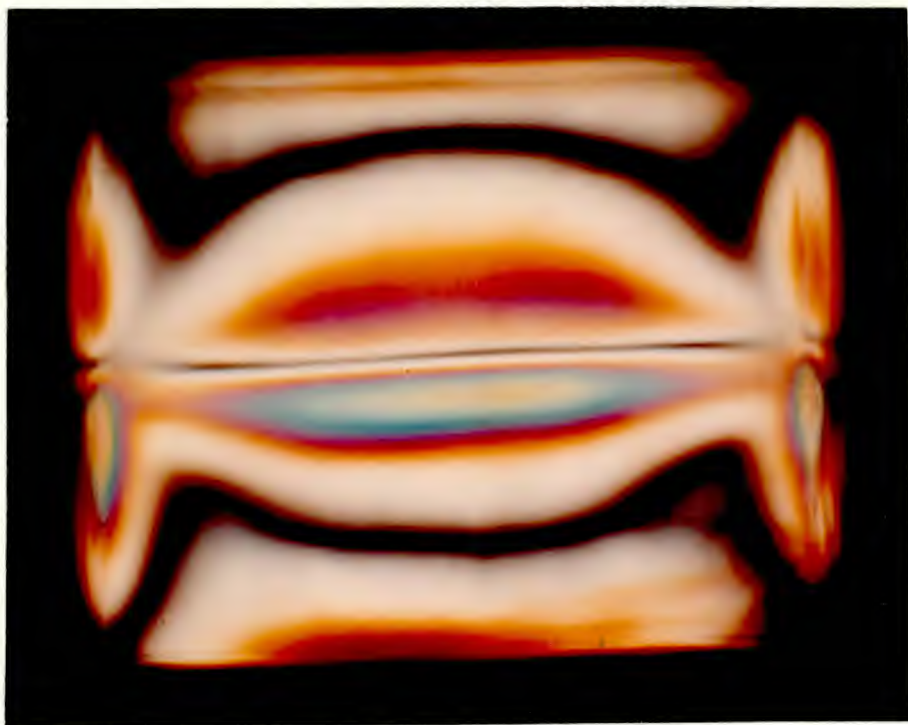


Fig 55. The birefringence observed lengthwise down a beam section with nicols crossed in  $\{110\}$ .  $\times 45$



Fig 56. The same view as Fig. 55, but with  $\frac{1}{4}$  wave plates added to the optical system in a "mixed" orientation.  $\times 45$ .

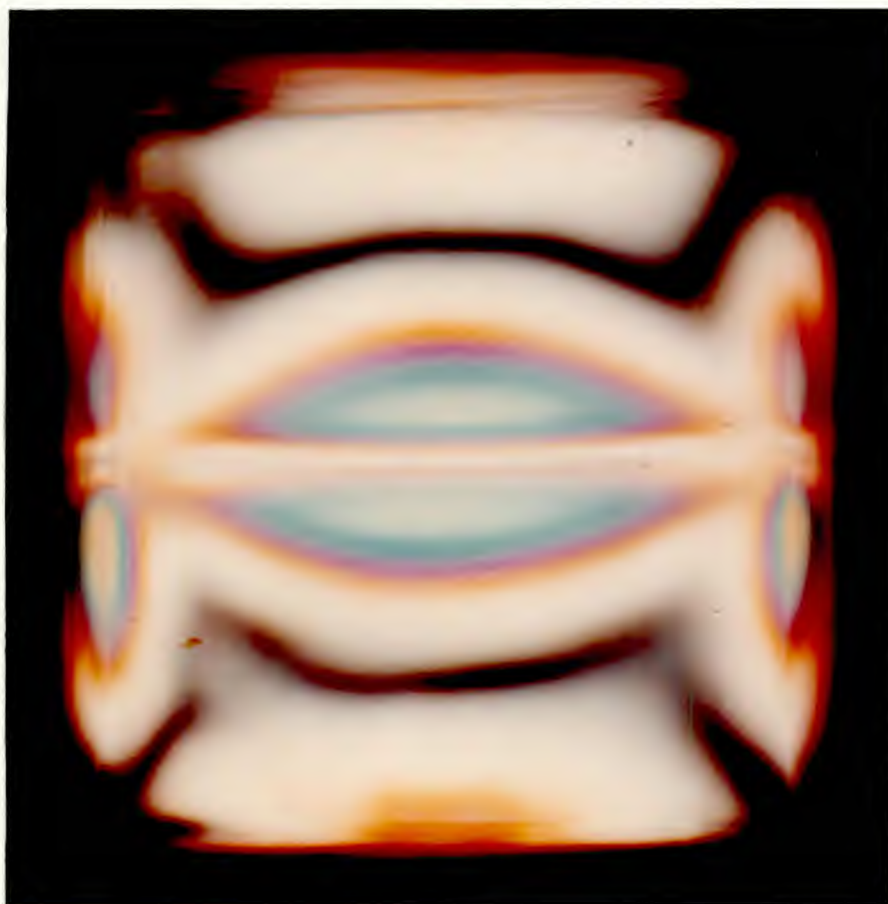


Fig 57. The birefringence observed  
lengthwise down a beam section with  
nicols crossed in  $\{110\}$ . x65.

beam sections were stood on end in a bath of di-iodo methane, which has a refractive index close to that of  $\text{MgO}$ , and examined with transmitted polarized light.

To observe and record the isoclinic fringes for various values of  $\phi$ , the nicols were set in a crossed position and the beam section rotated on the stage through  $90^\circ$ . In Fig.53 the isoclinic fringes at a number of values of  $\phi$  are shown for one specimen. By adding the correct value of  $\chi$  to  $\phi$ , the stress direction in each of the fringes is established. The principal stress trajectories are plotted in Fig.54.

A colour transparency was taken on type F Ektachrome of the same beam at  $\phi = 45^\circ$  under exactly the same conditions as was the photograph for  $\phi = 45^\circ$  in Fig.53, and is reproduced in Fig.55. By comparing the two it is immediately obvious that what appear as faint isoclinic fringes near the centre of the beam on both sides of the neutral axis in Fig.53 are in fact isochromatic lines corresponding to the first tint of passage. This is a result of the slight oversensitivity of the black and white film in the yellow region. The primary reason that the isoclinic fringes appear so diffuse and are scarcely visible in the central region of the beam is that the trajectories of the principal stresses in that region are changing direction only very slowly. Hence, what is recorded in Fig.53 is the combined effect of both the isoclinic fringes and the isochromatic fringes. A reproduction of another transparency taken of a beam of different cross-section, but very nearly the same strain is reproduced in Fig.57.

To make observations of the isochromatic fringes without the interference of superimposed isoclinic fringes is possible by adding  $\frac{1}{4}$  wave plates to the optical system, one between the polarizer and specimen, and one between the specimen and analyser. If the  $\frac{1}{4}$  wave plates are arranged with their axes perpendicular and rotated  $45^\circ$  relative to the direction of polarization their only effect will be to remove the isoclinic fringes. If they are arranged with their axes parallel, in addition to removing the isoclinics they will add a half wave length to all the relative retardations and so make the isochromatic fringes appear

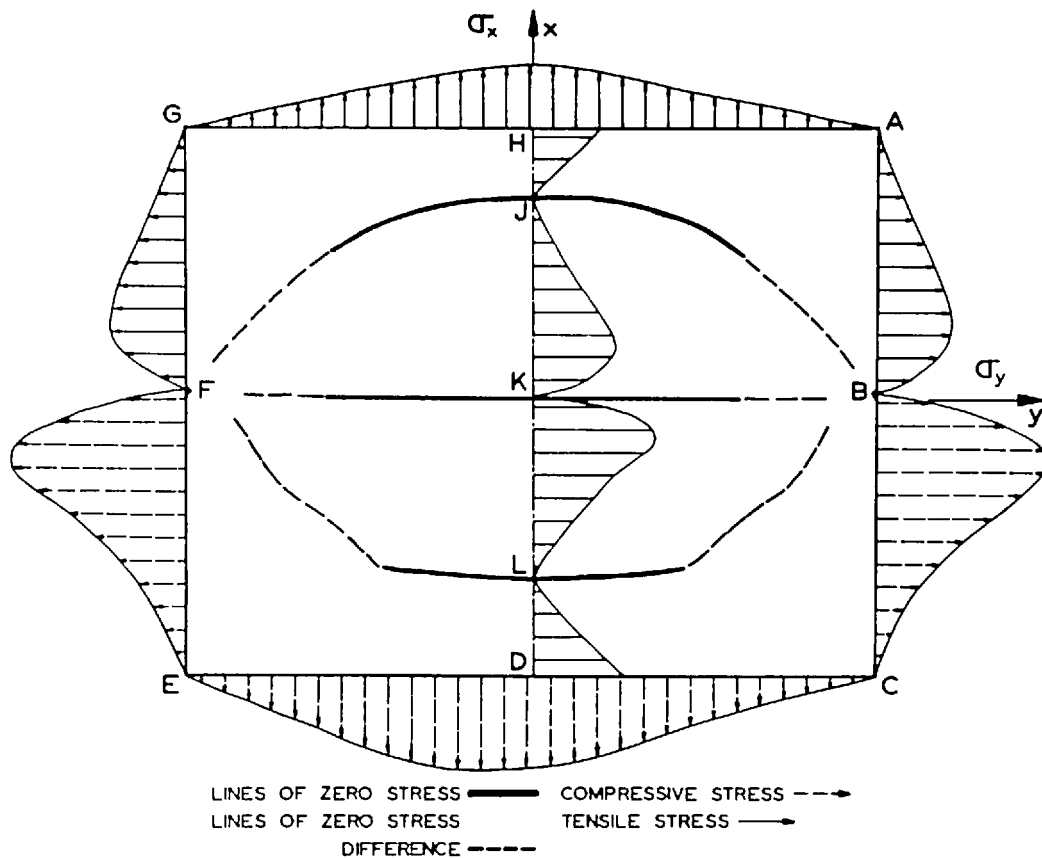


Fig 58. A plot of the magnitudes of the tangential stresses at the boundaries of the beam cross-section. Along the centre-line HD the principal stress differences - (P-Q) are plotted. The tension surface is at the top of the figure.

at points where the retardations due to the stresses are  $(n+\frac{1}{2})\lambda$  instead of  $n\lambda$ , where  $n=0, 1, 2, \dots$  etc. The latter arrangement was used when the transparency reproduced in Fig.56 was taken.

The relative retardation of the slow and fast rays could be determined at any point in the beam cross-section from the isochromatic fringes. Thus, the difference in the principal stresses could be calculated at all points where the directions of the principal stresses were those for which  $C_1-C_2$  were known. However, since the actual depth of specimen contributing to the isochromatic pattern was not known, the stresses were calculated entirely on a relative basis, the stress difference giving rise to the first tint of passage being arbitrarily set to one.

#### IV.7 The Determination of the Stresses

At the boundaries of the cross-section one of the principal stresses must be zero and the other parallel with the edge. Consequently, the retardation at the boundaries gives a direct indication of the magnitude of the tangential stress. This is plotted in Fig.58 along with the difference in principal stress down the centre-line of the cross-section. By applying small compressive loads to the cross-section and observing the resultant motion of the isochromatic fringes it was determined that the P lines of principal stress represent a compressive stress everywhere in the cross-section, whereas the Q lines a tensile stress. This permits a sign to be given to the stress plotted in fig.58 everywhere along the boundary.

Since it has been assumed that there is no variation of the stresses with  $z$ , the stress distribution in the cross-section can be expressed entirely with the stresses  $\sigma_x$ ,  $\sigma_y$  and  $\tau_{xy}$ . Along the beam boundaries both the shear  $\tau_{xy}$  and the normal stress must be zero. Therefore, along GHA and CDE  $\sigma_y$  is the only stress acting, and it is negative (tensile) along the tension face and positive (compressive) along the compression face. Along EFG and ABC only  $\sigma_x$  acts and is positive from E to F and B to C while negative from F to G and from A to B.

The centre-line, HJKLD, is a line of symmetry in the cross-section. Therefore  $\tau_{xy}$  must be zero everywhere along its length, and the principal stresses, P and Q, correspond directly with the stresses  $\sigma_x$  and  $\sigma_y$ . Considering first  $\sigma_y$ : along HJ,  $\sigma_y = Q$ ; along JK,  $\sigma_y = P$ ; along KL,  $\sigma_y = Q$ ; and along LD,  $\sigma_y = P$ . Since P is everywhere a compressive stress and Q a tensile stress  $\sigma_y$  changes sign at L, K and J and must fulfil the condition

$$\int_D^H \sigma_y dx = 0$$

By a simple examination of Fig.58 it can be seen that this condition is very nearly met by the differences of the principal stresses along the centre-line. This observation leads to the conclusion that  $\sigma_x$  must not vary greatly along the line and since  $\sigma_x$  is zero at all the points D, L, K, J and H it must be nearly zero everywhere along the length of the line.

This conclusion is substantiated by a simple application of the Lamé-Maxwell equation

$$\frac{\partial P}{\partial S} = \frac{Q-P}{\rho}$$

which given in difference form is

$$\Delta P = \frac{(Q-P)_M}{\rho_M} \Delta S$$

where  $\Delta P$  is the increase in P in moving along the P stress trajectory a distance  $\Delta S$ , from point M to point N;  $\rho_M$  is the radius of curvature of the orthogonal Q trajectory at M; and  $(Q-P)_M$ , the principal stress difference at M. If M and N are successive points of intersection of the principal stress trajectories drawn in Fig.58 proceeding from H to J, it is obvious  $\rho_M$  is very much greater than  $\Delta S$  and hence the change in P is very small compared to the principal stress difference,  $-(P-Q)$ , along the line. Since P must be zero at point H, P is everywhere nearly zero. Essentially, the same analysis can be applied to Q from J to K to show that it too does not vary greatly and is everywhere nearly zero along the line. It can thus be concluded that the

difference in principal stresses plotted along line HD in Fig.58 gives a reasonable indication of the values of  $\sigma_y$  along the line.  $\sigma_y$  is tensile from H to J and K to L, and is compressive from J to K and from L to D. Along this line the maximum value of  $\sigma_y$  is reached on the compression side of the beam near the neutral axis. In this region it is a tensile stress.  $\sigma_y$  does not vary rapidly with  $y$  in the region near the neutral axis until close to the sides of the beam where it decreases fairly rapidly to zero.

CHAPTER FIVEDISCUSSIONV.1 Introduction.

There are essentially two important problems which present themselves in the foregoing observations on the plastic bending of beams. The first is to determine why some beams deform almost wholly in the 2D mode while others, apparently similar in every respect, deform almost exclusively in the 3D mode. The second problem is to explain the marked difference in the work necessary to achieve the same strain in beams deformed in the two different modes. In the attempt to deal with these major problems it is hoped some light may be shed on the related topics of kinking and the fracture mode.

V.2 The Predominance of one Slip Mode.

Throughout the experimental work beams were observed to deform almost exclusively in a single slip mode under a wide variety of conditions. Some of the factors which undoubtedly played an important part in this behaviour were the number and orientation of slip sources, the interference of established slip bands with subsequent slip, and the modification of the stress within the beam by the initially formed slip. In almost every case all three of these factors played at least some part in the establishment of the eventual slip pattern, and only rarely could their effects be clearly separated. However, in order to apply some organization to the discussion it will be arranged around these three factors.

V.2 (a) Slip Sources.

One very obvious way of controlling the slip mode in a beam is to make certain that all probable sources of slip are so oriented that upon activation the slip is in the required mode. This method was employed for some of the beams of MgO and was described in III.4. This procedure is possible in MgO because all the grown-in dislocations that might otherwise move and multiply

to form glide bands are exceptionally well pinned. The effectiveness of this pinning was clearly demonstrated by Stokes (1962) who reported achieving stresses as high as six to eight times the normal flow stress before any slip occurred in carefully polished, dog-bone, tension specimens of as-received material. Much the same procedure for controlling the slip mode could be employed with beams of LiF, were this necessary, for once again the grown-in dislocations were never observed to move. Gilman and Johnston (1957) reported the immobility of the grown-in dislocations in LiF and attributed it to the associated impurities. However, the problem which must be answered is not how the slip mode can be controlled, but why when there is a random distribution of slip sources in both orientations one eventually becomes the dominant mode.

In the case of a beam with a vast number of dislocation loops that can act as slip sources, as in all the as-cleaved beams, the loops in the 3D orientation invariably expanded appreciably before the loops in the 2D orientation even began to expand. This might be expected since edge dislocations were observed to move at lower stresses than screw dislocations both in the work described here and that reported by Stokes et al. (1959 (b)). Consequently the slip in the 3D orientation should be the first to expand across the tension and compression surfaces. When the 2D slip does start to form the dislocations in the 3D mode should be moving across the beam at a velocity at least an order of magnitude greater than those in the 2D mode, according to the result of II.2. W.G. Johnston is reported by Keh (1960) to have found that the linear velocity of the edge component of a dislocation half-loop is approximately 200 times that of the screw component of another loop in the same homogeneous stress field in MgO. If the difference in velocities is in fact this great, the formation of slip in the 3D mode at the tension and compression surfaces would be even more favoured. Therefore the predominance of 3D slip observed at low strains in as-cleaved beams and illustrated in Figs.20 and 21 should be expected.

On the other hand when a beam with all obvious surface sources removed is deformed, the factors governing the distribution and mode of slip are more complicated. The contact of the knife-edges undoubtedly damages the crystal surface, producing fresh dislocation loops which should be the first to expand. This damage permits slip to start on the compression side of the beam, but does not initiate any slip on the tension side. If there are no sources activated at the low stresses on the tension side an outward displacement of the neutral axis will probably result with the stress on the compression side not exceeding that needed to move dislocations while the stress on the tension side continues to increase until a sufficiently high stress is reached to nucleate slip. The influence of the knife-edges is particularly important in three-point bending and often results in beams straining to fracture with no slip nucleated at the tension side of the beam. Such behaviour in carefully polished beams deformed in three-point bending was reported by Stokes (1962).

However, should some internal dislocation loop start to expand when a relatively high stress is reached in an otherwise slip-free beam, it will expand very rapidly, multiplying profusely, and result in a very heavy slip band. It is likely that such an expanding slip band will also produce slip on orthogonal planes after the manner described by Stokes (1962), and in so doing perpetuate its own slip mode. It was the rapid growth of slip bands in beams whose other slip sources were nearly exhausted that produced the marked load drops seen in the plots for beams (b) and (c) in classification II in Fig.22.

Normally it should be expected that several sources be activated at about the same stress with no particular preference for slip in one mode or the other. Hence the resulting slip should, at an early stage of the deformation in the polished beams, be a combination of few, but fairly heavy bands in both orientations. The relative predominance of one slip mode should depend

largely on the chance orientation of the activated slip sources. The resulting random distribution and orientation of slip might in addition be expected to substantially modify the stress distribution in the beam, and probably provide slip intersections suitable for the nucleation of microcracks and the ensuing fracture.

Most of these phenomena can be seen in Fig. 23. Along the tension side almost all the bands are in the 3D orientation and although few in number are relatively heavy. The great preponderance of slip has originated at the compression surface, possibly because of the influence of the knife-edges, and apparently displaced the neutral axis towards the tension surface.

Following the same argument and assuming that the slip sources are randomly oriented, it might be expected that only on very rare occasions would one mode of slip completely dominate, and that even in beams in which this was the case, fracture would probably be nucleated at the intersection of orthogonal slip bands long before the beams were full of slip. On even rarer occasions all the slip might be in the same direction, thereby increasing the probability of higher strains being reached before fracture. The behaviour of the MgO beams deformed at room temperature reported in III.3 substantiates these predictions. The postulated arguments of course only apply to beams deformed in pure bending, since in three-point bending the resolved stress is greater for one direction of slip in the 2D mode than for any of the other possible slip directions at every point in the beam. Consequently, in three-point bending the probability of single slip occurring in the 2D mode is substantially greater than in a beam deformed in pure bending since the velocity of the dislocations is so extremely stress sensitive.

An effect which probably plays some part in the establishment of one slip mode over the other is the propensity observed in all three of the materials discussed here to slip in such a direction that the glide direction is the shortest possible.

When deformed in compression all three materials slip so that slip steps appear on the wider face of the specimen while the direction of slip is parallel with the narrow face. This behaviour was reported in NaCl by Dommerich (1934), Wolff (1934) and Smekal (1934) and more recently by Pratt (1951). Wu and Smoluchowski (1950) found in some cases in their experiments on single crystals of aluminium that the first slip system to operate was not that predicted by the usual criterion of maximum resolved shear stress, but was that for which the length of the glide path was a minimum.

It is not clear what effect this phenomenon might have in a linearly varying stress field of the sort encountered in a beam before any plastic flow has occurred, but should the same criterion apply, the beams would have to be twice as deep as wide to give equal probabilities of expansion for slip in the two modes. In the case of NaCl there apparently was a greater tendency for beams to deform in the 3D mode as the depth to width ratio of the specimen was increased. However, this effect was often masked by many others and no specific conclusion drawn on the basis of the experiments reported here. This may suggest a profitable line of experiments in which the effect of the depth to width ratio is investigated.

It may be concluded for the case of MgO deformed at room temperature that the orientation and number of slip sources plays a very important part in determining the slip behaviour of the beam. If many randomly oriented sources are present the probability of the beam deforming with slip predominantly in the 3D mode is increased, whereas if all the obvious surface sources are removed the orientation and density of the slip is directly dependent on the number and type of the unseen sources that are eventually activated.

The role of the slip sources in determining the eventual mode of bending in NaCl and LiF was not as clearly defined as in

MgO at room temperature. All the beams of NaCl, LiF, and MgO for the high temperature tests, were polished until all obvious surface sources were removed and were then deformed. After about 0.2% or 3% strain both the NaCl and LiF beams contained a great number of fairly light slip bands in both orientations, but by the time strains of approximately 0.5% were reached, the slip in one orientation had virtually ceased operating. Although it was impossible to observe the slip during the high temperature tests on MgO, the final appearance of the beams was much the same as that of the LiF and NaCl. Thus it might be assumed that all these beams deformed in much the same way.

As a result of these tests it may be concluded that the number and orientation of the sources activated at the outset of plastic flow did not have as direct a bearing on the eventual slip mode as they had in the case of MgO at room temperature. Rather, it appeared that the established slip, by the time a strain of 0.5% was reached, in some way completely prejudiced the further formation of slip in favour of one mode or the other. Very seldom was there a preponderance of slip in one mode at the very early stages of deformation, as was sometimes observed in polished beams of MgO at room temperature. This observation may be explained at least in part by the fact that in LiF and NaCl the activated sources were far more numerous, thus reducing the probability of there being a significant difference in the proportion of sources in the two modes.

## V.2 (b) Interaction of Slip

The intersection and interaction of slip dislocations in crystals of the NaCl structure have long been known to influence the motion of the dislocations and hence the mechanical properties of such materials. Since the slip in the three materials considered occurs in  $\{110\} \langle 1\bar{1}0 \rangle$  slip systems, all the possible intersections can be divided into two groups according to the angle at

which the slip planes intersect. In one group all the intersections occur between normal slip bands while in the other the slip bands are always inclined to each other at an angle of  $120^\circ$ .

The first group of interactions may all be represented by the Burgers vector equation,

$$\frac{1}{2} [\bar{1}01] + \frac{1}{2} [101] \rightarrow [001],$$

assuming that the dislocations coalesce to form a single dislocation. However, it is doubtful that the newly formed dislocation will be stable since this reaction does not involve a lowering of the elastic energy. Nevertheless, by assuming that it does exist and that its formation causes a symmetrical pile-up of dislocations on the intersecting slip planes, Chou and Whitmore (1961) evaluated the shear stresses on the slip planes at the tip of the pile-up. They found that the stress reached a level of  $374$  times the applied shear stress when there was a pile-up of only 60 dislocations on the two glide planes, and that a pile-up of 120 dislocations was sufficient to reach the theoretical fracture stress. Obviously, to support such high stresses, the slip barrier must be extremely effective. Therefore it is most unlikely that the dislocation reaction given above plays a significant role in the blocking of slip.

In their electron microscopy studies of MgO Washburn et al (1960) observed little evidence of any interaction or piling up of dislocations at such intersections, and were it possible to carry out the same examination on LiF and NaCl the same observations would probably hold true. However, Stokes et al (1958) and Washburn et al (1959), and the author, in the work reported in III.3, observed the formation of cracks at such intersections in MgO deformed at room temperature. These observations probably imply that when the dislocation density is low in both the intersecting slip bands, the dislocations can quite readily slip past each other and continue on virtually uninhibited, but that as the number of dislocations in the slip bands increases they more effectively

hinder the slip in the orthogonal system and may eventually block its passage possibly leading to the nucleation of fracture. This corresponds to the observations mentioned above and to those made by Parker (1959), but does not explain why fracture was nucleated at the intersection of the very light slip bands as illustrated in Fig.26. In this case it seems unlikely that either of the slip bands were heavy enough to block the passage of dislocations on the other, and in addition there is no obvious pile-up of dislocations on either of the slip planes, leading to the conclusion that some other mechanism which contributed to the localization of a high stress at the intersection must have been operative.

For the case of MgO deformed at room temperature it may be summarised that orthogonal slip bands do not interfere greatly with each other as long as they are not very heavy, but with the band thickness the interference increases, and may lead to pile-ups sufficient to nucleate fracture. The same conclusions probably apply to NaCl and LiF except that there is little evidence of cracks ever being formed along such intersections. Instead, in these materials kinks of the type originally described by Brilliantow and Obreimow (1934) and later Orowan (1942) normally occur. In this kinking mechanism it appears that the dislocations in a light band interfere with the passage of dislocations in the dominant orthogonal slip thereby causing pile-ups of dislocations of opposite signs on the opposite sides of the interfering band. These pile-ups distort the lattice causing it to buckle and the crystal is said to have kinked.

Relating this behaviour to the slip in beams it is immediately obvious that it plays no direct part in the establishing of one slip mode as the dominant mode, since the slip intersections between the two modes are always at  $120^\circ$  and never at  $90^\circ$ . Therefore the part played by this type of intersection is limited to the behaviour of slip within either of the two modes, and of course to the nucleation of fracture. If reasonably heavy slip

bands are formed in one direction they may inhibit the passage of slip in the orthogonal direction as suggested by Parker (1959) and Stokes et al (1961), thereby promoting single slip. This behaviour was often observed in the beams of NaCl, LiF and the MgO deformed at high temperatures. The beams of MgO tested at room temperature normally fractured before the slip in one direction had become completely dominant. The fracture in almost all the beams tested in the polished condition originated at such a slip intersection before the slip had become very dense, at outer fibre strains less than 2%.

Thus the primary importance of this slip intersection in the bending tests was its ability to nucleate fracture and thereby terminate the test before either of the slip modes had become dominant.

The second group of slip interactions may be represented by the Burgers vector equations

$$(1) \quad \frac{1}{2} [0\bar{1}1] + \frac{1}{2} [10\bar{1}] \rightarrow \frac{1}{2} [1\bar{1}0],$$

or 
$$(2) \quad \frac{1}{2} [0\bar{1}\bar{1}] + \frac{1}{2} [10\bar{1}] \rightarrow \frac{1}{2} [1\bar{1}\bar{2}].$$

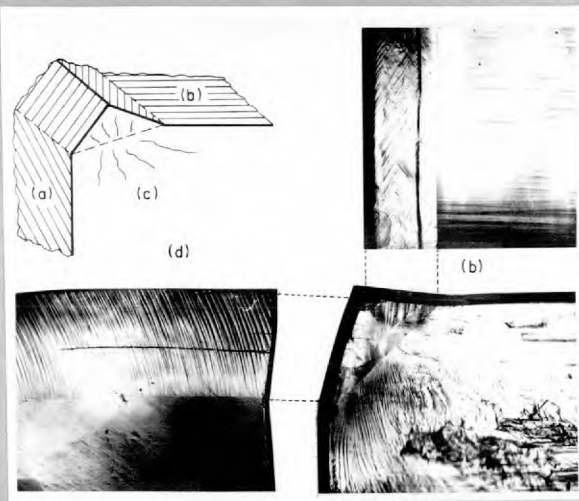
Chou and Whitmore (1961) have shown that 38% of the elastic energy is released when two dislocations combine as in the first equation. Thus the new edge dislocation  $[1\bar{1}0]$  (112) should be relatively stable. However, the interaction of dislocations as defined by the second equation involves an increase in the elastic energy, and hence the interacting dislocations probably will not coalesce, but will try to repel one another along the line of intersection of their slip planes.

In a theoretical paper Kear, Taylor and Pratt (1959) discussed both of these intersections in some detail. They postulated that the first would occur giving rise to the new edge dislocations which would be in a position of minimum energy when lying in  $[\bar{1}11]$  one above another in (110). This arrangement would give rise to a pure tilt about  $[\bar{1}11]$ . Furthermore, they predicted for the second interaction that the dislocations would not coalesce, but

that when many parallel slip planes intersect in this manner a crossed grid of mixed edge and screw dislocations would be formed. The resultant complex dislocation boundary should lie in  $\{110\}$  and act as a barrier through which the slip dislocations can be driven only with difficulty.

The formation of these boundaries lying in  $\{110\}$  between regions slipped in the different directions was originally made by Brilliantow and Obreimow (1937) when they compressed squat specimens of NaCl. More recently the same phenomenon has been reported by many others working with NaCl and other materials; see for examples Stepanow and Nanskoi (1954), Klassen-Neklyudova et al (1957), Vaughan et al (1958) and Taylor and Pratt (1958).

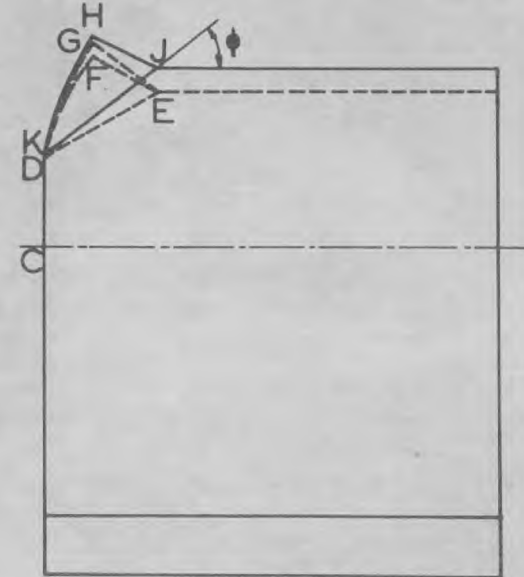
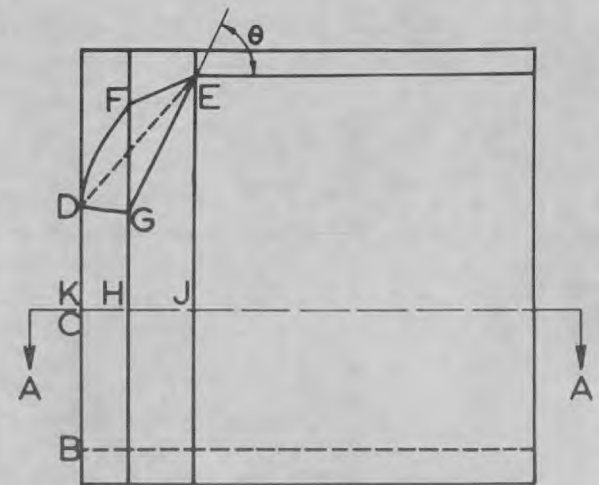
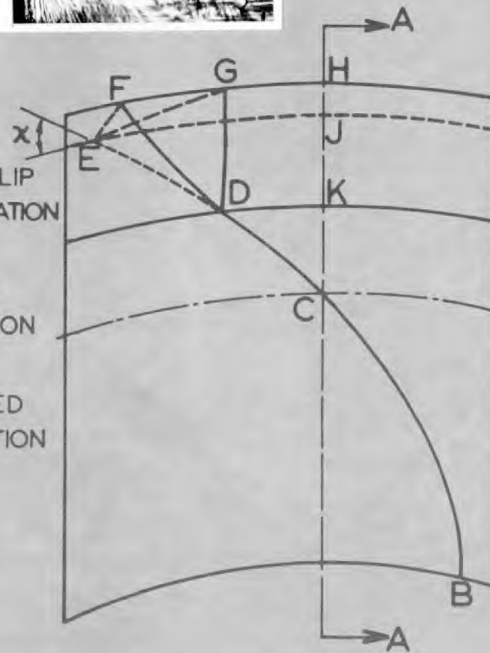
Thus it is evident that this slip interaction does provide an effective barrier to the propagation of slip, but its role in the nucleation of fracture is not as well established. Chou and Whitmore (1961) showed that if dislocations pile up on the intersecting slip planes at such a boundary, only 62 dislocations in each slip band are sufficient to raise the stress at the intersection to a level sufficient to nucleate a microcrack. Washburn et al (1960) reported seeing dense tangles of dislocations at these intersections in MgO, but reported that there was no evidence of dislocations piling up against the intersections as was assumed by Chou and Whitmore (1961). Nevertheless microcracks were reported to have been nucleated in  $\{110\}$  planes at such intersections by Keh et al (1960) and Keh (1960) for specimens of MgO indented at room temperature. Phillips (1962) observed cracks nucleated at the intersection of  $\{110\}$  slip planes in compression specimens of LiF, NaCl and MgO, and stated that the cracks propagated parallel to a  $(110)$  plane in regions of high stress concentration. He did not make clear whether the intersection was between slip bands at  $120^\circ$  or at  $90^\circ$ , but it is probable that in a region of high stress concentration there would be significant slip intersections of both types. In three of the five examples given by Parker (1959)



BCDFE: A (101) 2D SLIP  
PLANE AFTER DEFORMATION

DGE: A (011) 3D SLIP  
PLANE IN KINKED REGION

KHJ: FORM OF KINKED  
REGION IN CROSS-SECTION



SECTION A - A

Fig 59. The form of a kink along the tension edge of a beam bent with slip in the 2D mode except for the kinked region. The diagram is based on the slip models of II.5 and 6.

of cracks nucleated at the intersection of slip bands, intersections at both angles had taken place, and hence the mechanism responsible for the nucleation of the crack was not unambiguously determined. Cracking was observed by Stokes (1962) along the slip bands radiating from the line of contact of the inner knife-edge in his bending tests, and similar observations were reported in III.4. In these last two cases the crack was not in the same orientation relative to the intersecting slip as in the observations of Keh et al, but one feature common to all the examples cited is that  $\{110\}$  cracks have only been observed to originate in regions where intersections at both angles occurred and in which the stress field was complex and not well understood.

Now, relating the effects of the slip intersections at  $120^\circ$  to the geometry of slip in beams it is immediately obvious that there must be significant interference between the slip in the two bending modes. One way in which this interference was manifest was by the formation of the  $\{110\}$  boundaries between regions slipped in different modes. This was first observed by Stokes et al (1961, 1962) in beams undergoing bending and was frequently observed during the course of the work reported in III.5 and 6. In the light of the current experimental observations the explanation for their formation given by Stokes et al does not appear entirely satisfactory. They called this phenomenon kinking and postulated that the kinks were formed to accommodate the lateral contraction of the beam. The beams they reported always slipped in the 2D mode, as might be expected in 3 point loading, and the smaller region, that shall be referred to as the kinked region, slipped in the 3D mode. This slip orientation can readily be seen in Fig.59 which is a reproduction of their original prints.

Consider the beam of NaCl pictured in Fig.59. Since  $\chi$ , the angle at which the 2D slip bands are inclined to the beam fibre, is very nearly  $35^\circ$  at the bottom of the kink it might be

safely assumed that this beam had been severely bent to the limiting curvature described in II.5. . Further, assuming that the 3D slip in the kinked region had developed from the outset of plastic flow and at an early stage became the completely dominant slip mode in the kinked region, the slip planes should have rotated until inclined to the cross-section at an angle of  $55^\circ$ . The observed value of  $\theta$  is as close to this as can be measured. Furthermore, if the kink is assumed to lie in a plane that had been (110) in the undeformed beam, but became distorted by the lattice rotation as the deformation proceeded, then at the strain necessary to produce the observed values of  $\chi$  and  $\theta$  the boundary should be inclined to the tension surface at  $40^\circ$ . The measured value of the angle is as nearly  $40^\circ$  as can be determined on the original print.

The other part of Fig.59 is a projection drawing of a kink formed at the rectangular corner of a beam. The drawing was made for a beam strained as assumed in the preceeding paragraph, and on the basis of the slip models given in II.5 and 6. In other words, it is the theoretical form of the kink shown in the inset figure. One of the obvious differences between the two is that the 3D slip steps on the side of the kinked beam are substantially more curved than the one drawn in the diagram and in fact even appear to be curved the wrong way. Another difference is that the tilted edge of the beam is not inclined as sharply to the tension surface as in the diagram. Both these differences are due to the excessive rounding of the beam edge during the chemical polishing carried out before bending. In all other respects the two are very much the same.

As was shown in II.5 a slip plane in a beam deformed in 2D bending is the involute of a circular cylinder located with its centre at the centre of curvature of the beam. Thus the slip plane is a simple curved surface whose curvature never changes sign, but increases from a minimum at the tension surface to a maximum at the compression surface. Assuming the lattice between the slip

planes is elastically bent, additional constraints must be applied to it if it is to remain a plane curve and not become an anticlastic surface. However, when kinking occurs, if the secondary slip is in the right direction, the surface does become anticlastic and some of the elastic energy will be released. If the slip occurs in the other possible direction the elastic strain energy of the surfaces parallel to the slip surfaces will be increased. Thus, in a beam slipped in the 2D mode the slip in the direction that releases some of the elastic strain energy should be preferred in the kinked material. The idealized form of such a plane, with the kink in the right direction, is drawn in Fig.59. Wherever it could be distinguished in the experimental work the direction of kinking was that expected on the above basis. This indicates that the constraint leading to the formation of the kinks is not entirely accounted for by the transverse stress arising from the major bending stress, for were this the case neither of the two slip directions in the kinked material would be favoured.

Furthermore, if the kinking is related to the curvature of the slip planes as suggested above, since these are most highly curved at the compression face of the beam it should be expected that kinks be formed at the compression edges even more frequently than along the tension edges. Insufficient experiments were carried out to determine if this preference could be established, but it was found that the kinks occurred along the compression edges of the NaCl beams just as regularly as along the tension edges. In MgO insufficient kinks were observed to permit the drawing of any conclusions, but again kinks were observed along both the tension and compression edges.

On one occasion, a kink in the reverse orientation to those discussed so far was formed in a beam of MgO deformed at high temperature. It formed in a beam that had slipped exclusively in the 3D mode save for one edge at which the slip was the 2D mode, as can be seen in Fig.47. This observation, and the not uncommon

branching of the kinks, as seen in Fig.47, together with the observation of separate parallel kinks along the edges of some beams, lead to the conclusion that the formation of these kinks is in part dependent simply on the orientation in which the initial slip occurred in different parts of the beams. The stresses developed as the straining proceeds undoubtedly influence the kinks, and particularly the direction of slip, but probably plays only a secondary role in the actual formation of the kink.

More weight might be given to this argument by a simple examination of the stresses in the cross-section of a beam that arise from the major bending stress. These stresses will be at a maximum near the centre of the beam and not at the edges where the kinking normally was observed. Furthermore, the work hardening rate of beams deformed in single slip in the 2D mode was very low, as can be seen in the curves of Fig.22, III, and hence the longitudinal elastic stress never reaches a level much more than twice the macroscopically observed flow stress. This estimation of the stress is based on the assumption that the stress still increases linearly from the neutral axis out to the tension and compression surfaces, which of course is no longer true, and hence this estimate is high. Therefore, since the transverse elastic stresses are only Poissons Ratio times the longitudinal stress it is unlikely that they even reach the magnitude of the observed flow stress. Such a stress level is obviously inadequate to account for the cracking and other phenomena attributed to it by Stokes et al(1961), but undoubtedly does play some part in the deformation. It is postulated that the orientation of the initially activated slip sources, and the sign of the curvature of the lattice play more significant roles in the formation and development of kinks.

So far it has been established that stable boundaries in  $\{110\}$  do occur in bent beams, separating regions slipped in the two bending modes. However, the two possible types of  $120^\circ$  slip intersections undoubtedly have a significant effect even when it

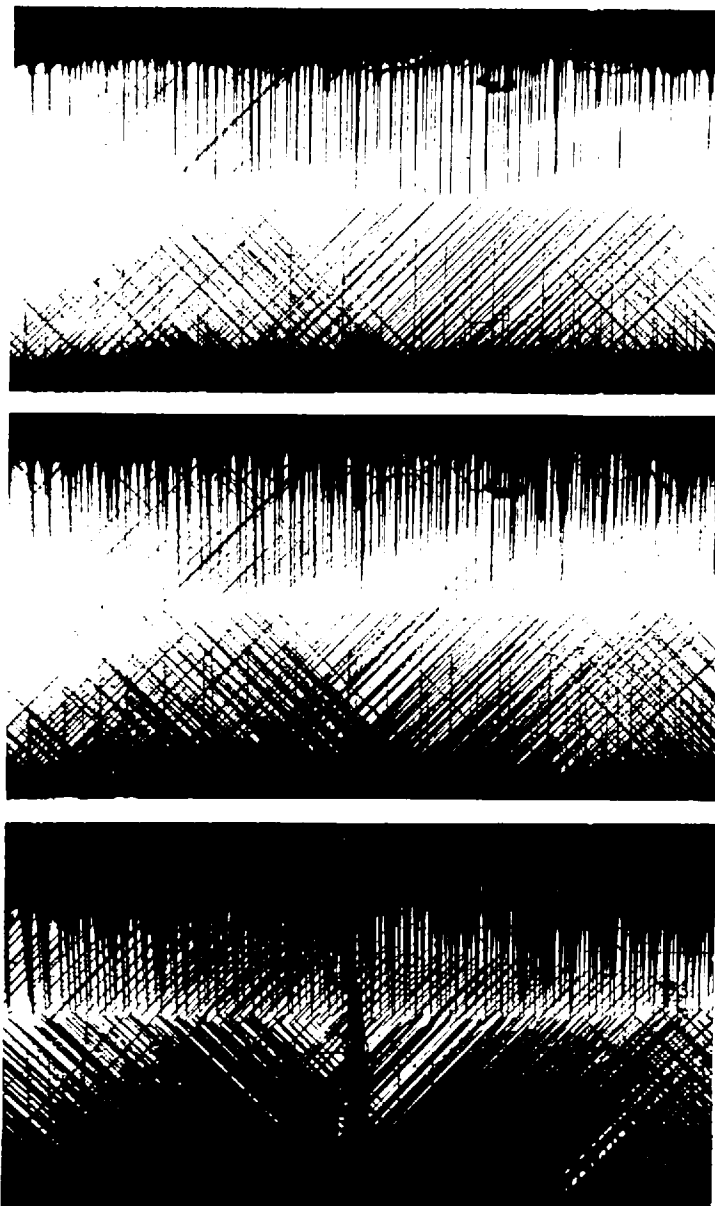


Fig. 60. The etched side of a beam at strains of 0.5%, 1.2% and 1.8%. Note the predominance of 3D slip on the tension side and 2D slip on compression side. x23.

is not manifest as a kink. Whenever an intersection between slip takes place at this angle the passage of the dislocations must be hampered. Thus if initially a preponderance of sources producing slip in one slip mode are activated this slip will hinder and may effectively block the propagation of slip in the other mode. This behaviour is illustrated in Fig.60. The sequence of plates were taken of the slip pattern in a beam at strains of 0.5%, 1.2% and 1.8%. The beam had first been polished, was then sprinkled on its tension and compression faces with 45 mesh carborundum and finally strained. In order to take the pictures it was necessary to remove the beam from the bending jig, etch it, take the plate, and then replace it in its original position for continued straining. It fractured at 1.8% strain. It can be seen that although the slip was generally mixed in the first of the pictures, the predominance of the 3D mode is evident on the tension side while on the compression side the reverse is true. From that point in the strain onwards it can be seen that the slip occurred almost exclusively in the mode that was predominant at 0.5% strain. It is also evident that the formation of the 2D slip on the compression side was primarily in a single direction in any given area after the first plate.

The hindrance of slip by these  $120^\circ$  interactions favours the predominance of slip in the 3D mode since it can expand across the tension and compression surfaces at a lower stress than can the slip in the 2D mode, and even when the stress is high enough for the 2D slip to expand, the slip in the 3D mode will be expanding at a much higher velocity. Therefore, assuming a large number of random sources the 3D slip should effectively criss-cross the surfaces before the 2D slip has had time to expand appreciably and thus be in a better position to hinder than be hindered. The type of slip pattern predicted is illustrated in Fig.14.

On the other hand, when there are no fresh slip sources and a high stress must be reached to activate the potential sources, the interaction between the slip in the two modes assumes less importance relative to that of the orientation of the sources eventually activated. It nevertheless still plays a significant role in the eventual domination of one slip mode in such beams, as long as fracture does not terminate the test prematurely.

#### V.2 (c) Modifications of Stresses.

The final factor influencing the eventual mode of slip is the modification of the stress in the beam by the initial slip. Up to this point it has been assumed that the stress driving the dislocations in the two slip orientations is the same for each at any point in the beam. This condition no longer exists once any slipping has taken place. The use of stress birefringence to observe the slip is possible only because of the modifications to the stress pattern in the beam, and thus clearly demonstrates the changes which accompany any plastic deformation.

The precise nature of the changes in the stress distribution are extremely complicated and are the result of so many factors, that for a rigorous examination of their effects each case must be individually investigated. Nevertheless, certain general observations can be made on some of the changes in the stress pattern. Normally the stress pattern was most severely distorted in beams that, before bending, had all their obvious slip sources polished away, and consequently slipped in relatively few, but quite heavy bands. In such beams the neutral axis, as observed by birefringence, was often displaced away from the side of the beam in which the slip in any given area originated, and in some beams became so distorted that it was unrecognizable. Furthermore, in the regions where slip intersections occurred both within a mode and between slip in the two modes, the local stresses were often very high and the birefringence pattern extremely complex. All these phenomena

can be seen in the beam pictured in Fig.23. In this figure it is virtually impossible to determine the position of a neutral surface from the etchpit pattern, for although slip in the 3D mode originating on opposite faces of the beam stays fairly well apart, and therefore might be used to indicate the position of the neutral axis, when the 2D slip is considered, any semblance of a neutral axis is destroyed. The slip in the 2D mode originating at opposite faces frequently intersects and in some cases very nearly runs completely across the beam, intersecting bands of 3D slip that apparently formed under stresses of the opposite sign. This particular example is typical of the behaviour of many beams, and though it presents a confused picture, it gives some indication of the importance of the changes that take place in the stress distribution once slip has occurred.

On the other hand, when there are a great many slip sources, as in the as-cleaved beams, the distribution of slip is far more nearly homogeneous, and during bending the specimen behaves much more as a simple beam with a well defined neutral axis. Nevertheless birefringence observations reveal that even in these cases there still is substantial elastic strain energy resulting from the slip. However, on the rare occasions when the slip was in a single direction in any specific region of a heavily strained specimen, the birefringence pattern corresponded very nearly to that expected in an elastically deformed beam, indicating that the resulting elastic strain energy was very nearly the minimum possible value.

Thus on the basis of a simple photoelastic examination, it is evident, as might be expected, that the more nearly the slip distribution approaches the condition of homogeneous single slip, the less elastic strain energy is needed to achieve the same strain. Hence, once slip in one direction is established, the energy necessary to form slip in any other direction is increased, even if the energy associated specifically with the intersection of the dislocations is not considered.

The role of the secondary elastic constraints in influencing the orientation of the slip in beams predominantly deformed in the 2D mode was discussed in the previous section. However, in beams deformed in the 3D mode the magnitude of the stresses in the cross-section is very much higher for the same strain, and their direction and distribution far more complex. Using the stress convention established in IV.7, and referring particularly to Fig.58, it can be seen that at the beam surfaces, where slip would most probably be nucleated, there is only one stress that need be considered. Furthermore, since only the effect on the nucleation and propagation of slip in the 2D mode is to be considered, and  $\sigma_y$  has no component resolvable in the direction of slip, only  $\sigma_x$  need be examined.

Since  $\sigma_x$  is normal to the tension and compression faces it must be zero at the faces, and, as shown in IV.7, does not vary rapidly in the x-direction. Therefore, if the sources are considered to be lying in the beam surfaces, ideally they are unaffected by  $\sigma_x$  at both the tension and compression faces. However, at the sides of the beam, particularly near the neutral axis,  $\sigma_x$  becomes very large. In this area, on the tension side of the neutral axis it is a tensile stress while on the compression side a compressive stress. Thus when  $\sigma_x$  is resolved in the direction of 2D slip at the sides of the beam it is of the opposite sign to the resolved component of the simple bending stress and reduces the stress applied. At high strains, i.e. 1.5%, it may even exceed the simple bending stress by a sufficient magnitude to nucleate slip in the 2D mode of the wrong sign to accommodate the major bending curvature. This may explain the appearance of the 2D slip birefringence at the sides of the beam in Fig.38 and the 2D slip lines near the neutral axis on the side of the beam shown in Fig.60. (These stresses will be discussed more fully in the next section.) It further explains why the 2D slip in the as-cleaved beams pictured in Figs.20 and 21 did not propagate completely out to the edges of the tension and

compression faces. The loops in the 2D orientation propagate deeply into the beam and thus are retarded and eventually stopped significantly below the surface by the  $\sigma_x$  stress component before they reach the sides of the beam.

One further case that bears mentioning before leaving this topic is that of the beam shown in Fig.24 in which two parallel 3D slip bands crossing the tension surface blocked the propagation of slip both in the 2D mode and on orthogonal planes in the 3D mode. It is evident that the local stresses resulting from these slip bands must be high, and when resolved in the direction of slip in the three intersecting systems must be of the opposite sign to the simple bending stress. Assuming that no identical slip bands existed on the compression side of the beam in the same planes as the two bands pictured, thereby allowing the compression side of the beam to bend sharply at the same place, but that the slip on the compression side though equivalent in total magnitude was fairly evenly distributed, then a tensile stress normal to the neutral axis would result in the immediate neighbourhood of the heavy slip bands. Such a stress would reduce the resolved shear stress in the direction of the 2D slip and thus may account for the blocking of the slip in this orientation. The blocking of the orthogonal 3D slip is not as easily explained, since, if the dislocation density in the two heavy bands is assumed constant, there should be no resulting shear stresses in orthogonal planes, according to the stress analysis of Cottrell (1953). However, it is highly probable that the necessary conditions for his analysis do not apply, primarily because of the constraints applied by the compression side of the beam. As a result shear stresses capable of blocking the orthogonal slip may exist.

To summarise, in beams in which there are many random sources, slip in the 3D mode is favoured at the outset of plastic flow, both because of the ability of edge dislocations to start moving at a lower stress than screw dislocations, and because of

their substantially higher velocity even when the stress is sufficient to move the screw dislocations. Since the initial slip can hinder the motion of subsequently formed intersecting slip the probability of 3D slip becoming the dominant mode is quite high. On the other hand, for specimens with no fresh dislocation sources the eventual slip mode depends in large part on the chance orientation of the initially activated sources. Were these predominantly in the 3D orientation very often the beams continued deforming in this mode, the established slip offering sufficient resistance to the formation of slip in the 2D mode to overcome the attraction of the more favourable inherent work-hardening rate.

These effects were more clearly demonstrated in the beams of NaCl and LiF than in MgO at room temperature, since the elastic interactions of the slip were not masked to the same extent by the hardening effects of the impurities. Nevertheless, the MgO deformed at high temperatures exhibited much the same properties as the NaCl and LiF, this change in characteristic behaviour mainly being due to the reduced effect of the impurities and the greater ease of dislocation climb and cross-slip.

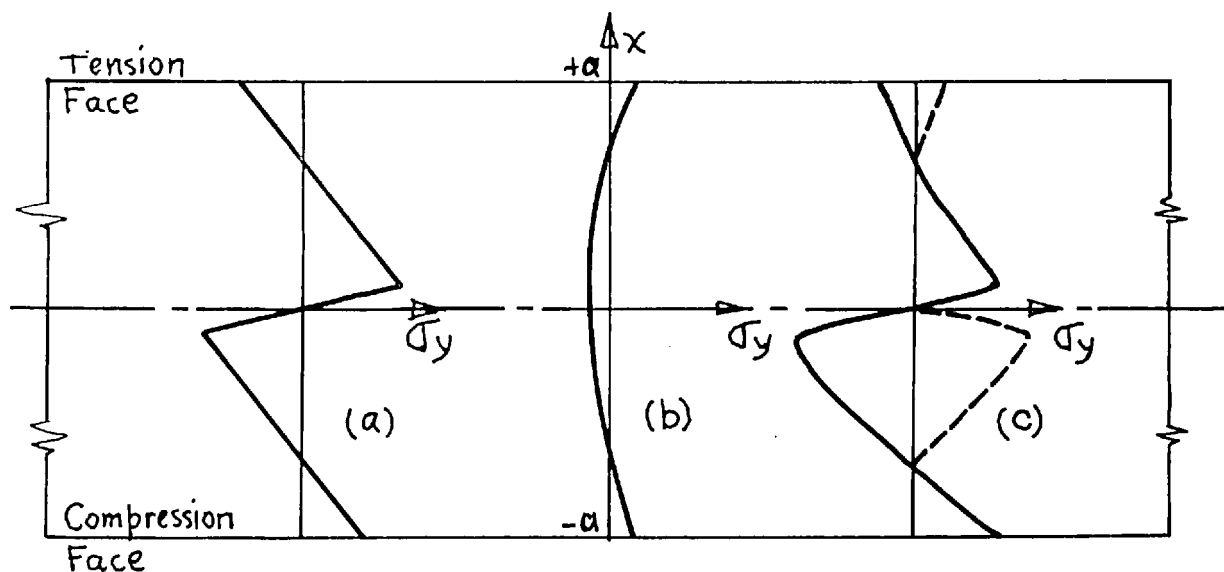
### V.3 Work Hardening Rates

When a beam is plastically bent with slip entirely in a single direction in the 2D mode, ideally, the form of the load-strain curve, at low strains, should depend only on the relative values of the stresses necessary to nucleate and to move the dislocations, as derived by Read (1957). At higher strains the change in the shape of the slip planes and the increased moment of inertia of the beam cross-section lead to a significant increase in the rate of work-hardening. However, for the strains at which comparisons shall be made between the two bending modes these effects are entirely secondary and need not be considered. The observed rate of work-hardening up to strains of about 6% shall be considered to be simply a combination of the geometric factors derived by Read, and

the hardening effects associated with interactions between dislocations, and dislocations with point defects. These last two factors should contribute very little if the deformation is entirely in single slip.

Similarly, when a beam deforms plastically in the 3D mode the same factors should influence the work-hardening rate to much the same extent. However, upon referring to the load strain curves in Figs 22, 32, 39, 43 and 45, it is immediately obvious that some additional factors contribute far more significantly to the work-hardening rate. To determine the reasons for this difference and its significance, a more careful examination of the cross-section of beams deformed in the 3D mode is considered important.

Assuming the stress differences plotted along the centre-line of the typical beam in Fig.58 to be very nearly representative of the relative magnitude of  $\sigma_y$ , as suggested in IV.7, the sign and magnitude of  $\sigma_y$  bears a marked similarity to the longitudinal bending stresses in the relaxed, plastically deformed beam described by Nye et al (1957). This stress distribution might have been expected since the plastic strain of the cross-section varies with the longitudinal strain, but is of course at any element in the beam of the opposite sign. For any incremental change  $\Delta\theta$  in  $\theta$ , which is the angle between the cross-section and the slip plane, the incremental longitudinal strain is  $\sin(\theta+\Delta\theta) - \sin \theta$  while the transverse strain is  $\cos(\theta + \Delta\theta) - \cos \theta$ . It is evident that the magnitude of these strains are equal only at  $\theta = 45^\circ$  and that as  $\theta$  either increases or decreases from this value, the difference in the strains increases at an ever increasing rate. Neglecting, for the time being, the fact that all the surfaces originally parallel with the neutral plane have become hyperboloids of one sheet, and assuming that the tension and compression surfaces are simply sections of circular cylinders, the difference in the magnitudes of the strains  $\sin(\theta + \Delta\theta) - \sin \theta$  and  $\cos(\theta + \Delta\theta) - \cos \theta$  must be accounted for by elastic deformation if the beam volume is to



Plot of  $\sigma_y$  in cross-section of beam slipped in the 3D mode.

- (a) Stress distribution in a relaxed plastically bent beam.
- (b) Stress distribution arising from transverse elastic strain.
- (c) Sum of (a) and (b): Plotted showing sign of stress. ———

Plotted as  $-(P-Q)$  in Fig. 58. - - - - -

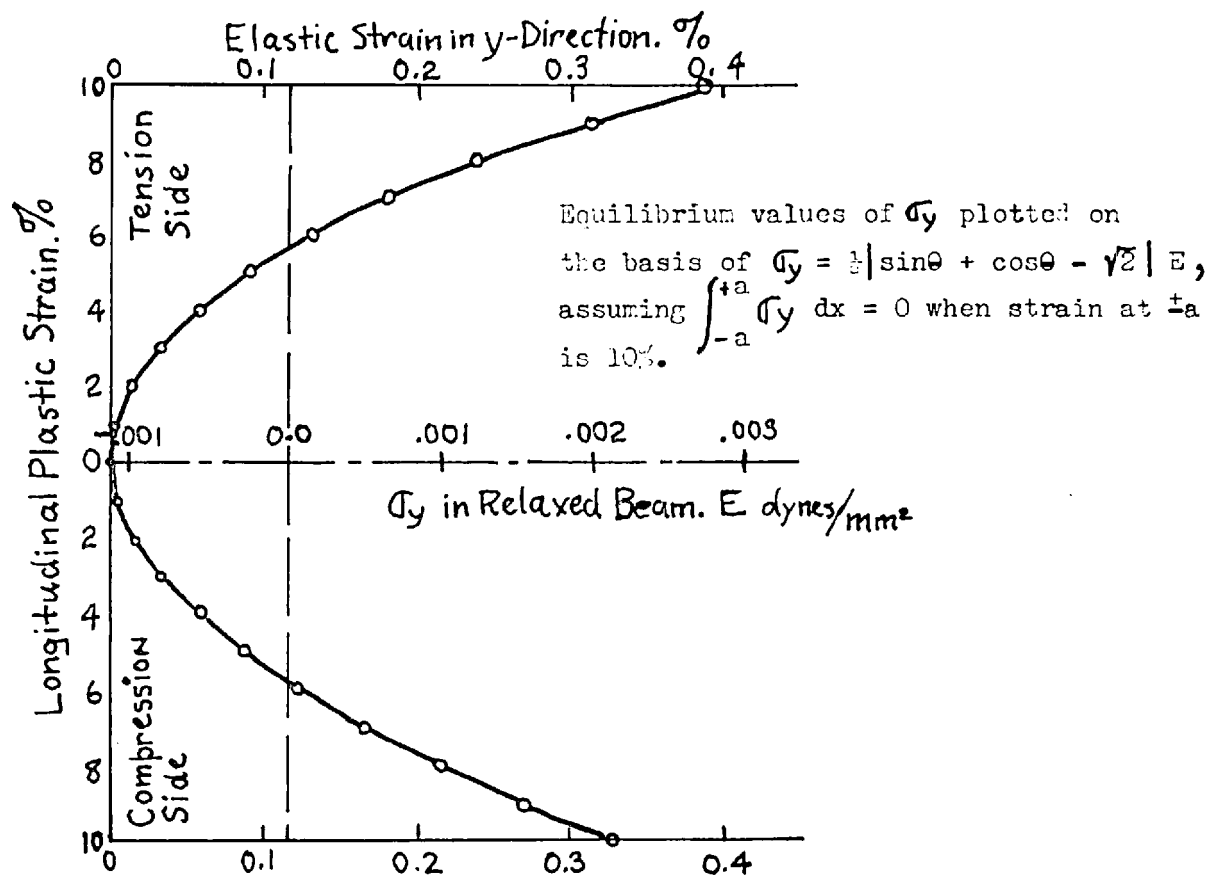


Fig. 61

remain constant.

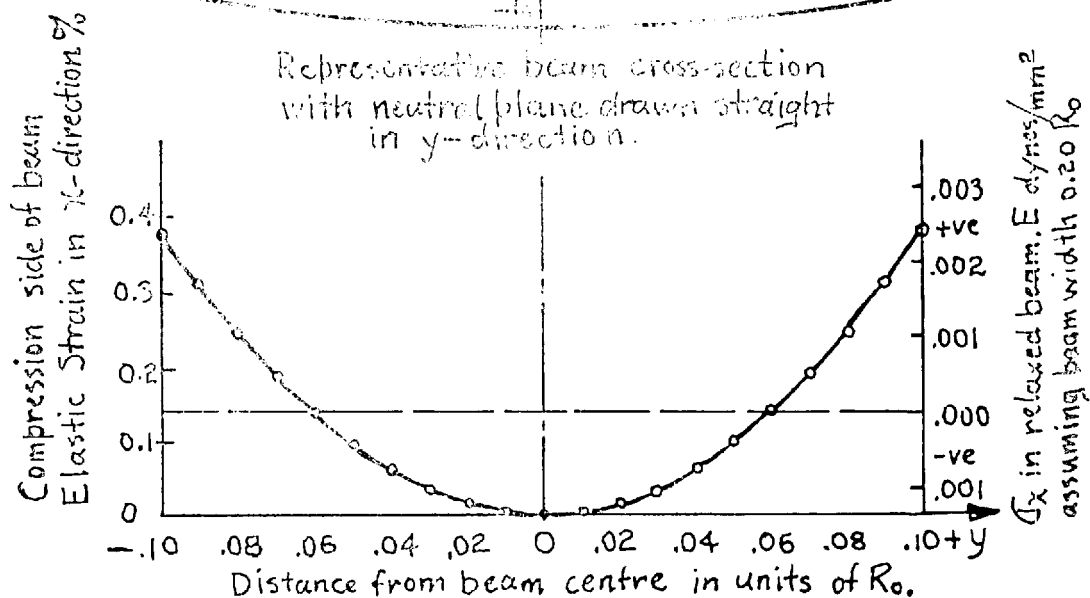
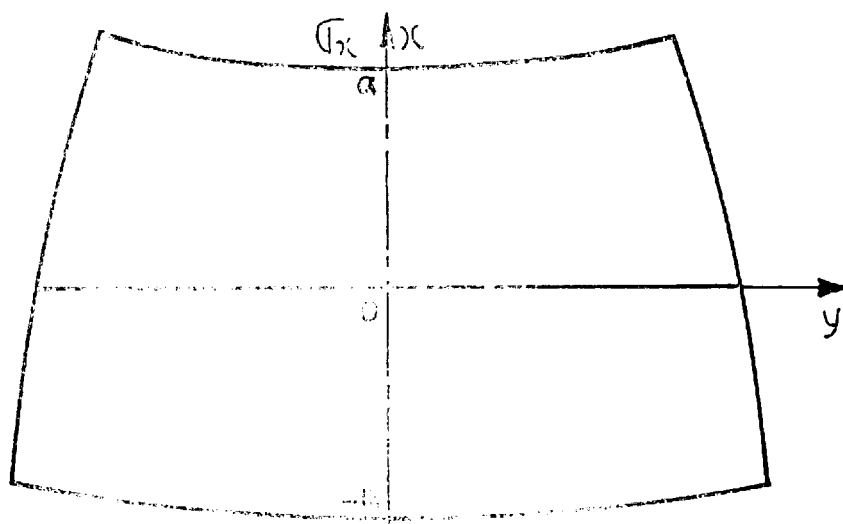
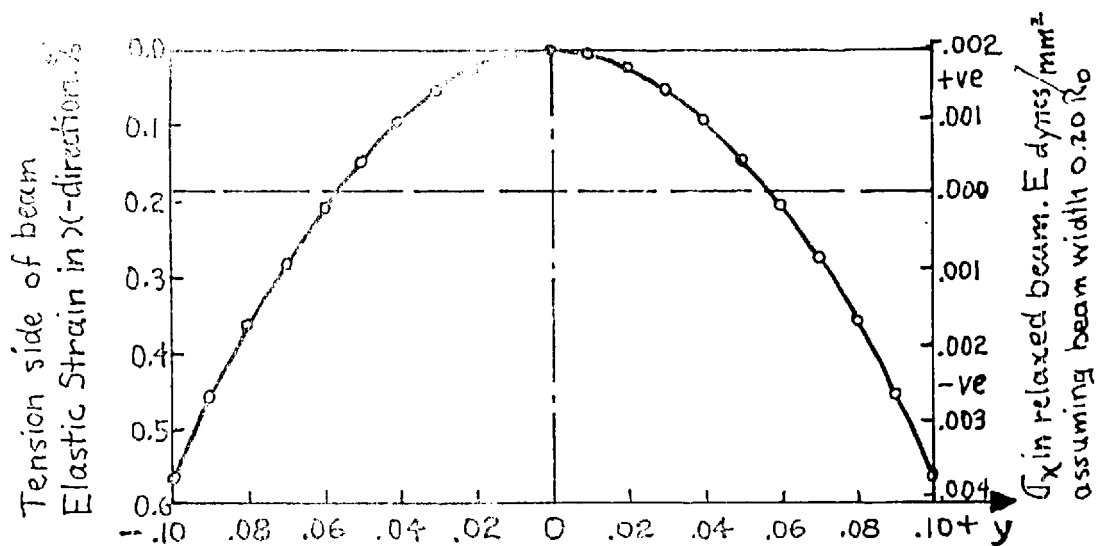
Since  $\theta > 45^\circ$  on the tension side of the beam, the transverse strain is greater than the longitudinal strain, while on the compression side of the beam  $\theta < 45^\circ$  and the transverse strain is less than the longitudinal strain. Such strains would lead to a transverse compressive stress superposed on the beam cross-section, but since the conditions of static equilibrium must be maintained, the resulting elastic stress remains a compressive stress at and near the tension and compression faces, but reverses sign to become a tensile stress in the intermediate region as shown in Fig.61. When such a stress distribution is superposed on that of a relaxed, plastically deformed beam, the stress pattern resulting is very nearly that observed along the line HKD in Fig.58. Thus it can be seen that even at strains as low as 0.3% the incompatibility of the plastic strain with beam volume, resulting from the lattice rotation, contributes significantly to the strain energy in the beam.

If plastic bending is to proceed at any point in the deformation, the component of the longitudinal bending stress resolved in the direction of slip must equal the sum of the component of the transverse stresses resolved in the direction of slip and the actual stress necessary to move the dislocations. Thus it can be seen that as the plastic strain increases the elastic strain of the cross-section increases, this increase requiring a greater stress, which in turn is reflected in a higher work-hardening rate. The magnitude of the stresses that may result in this deformation mode are extremely large, as might be at least indicated by the fact that in a beam deformed ideally as defined in II.6 to a strain of 5% the resulting elastic strain is approximately  $1 \times 10^{-3}$ , leading to a stress approximately  $1 \times 10^{-3} E$ . Though of the right order of magnitude, this estimate is high, for the slip surfaces will only rotate to a position of equilibrium at which the longitudinal elastic strain is slightly larger, but very nearly equal to that in the plane of the cross-section. Thus it may be assumed that as a result of this

elastic strain equilibrium the transverse elastic stress can be represented by the expression  $\frac{1}{2} (\sin \theta + \cos \theta - \sqrt{2})E$ . A plot of these values for strains up to 10% is given in Fig.61. Hence, the very rapid rate of work-hardening can in large part be attributed to the elastic strain energy necessary to maintain the beam at a constant volume throughout the deformation.

Moreover, it can be seen that the maximum tensile stress achieved anywhere in the beam is probably the transverse stress in the cross-section just below the neutral axis, in the region in which the longitudinal cracks were observed to nucleate and propagate.

Finally, an account must be given of the very high stresses observed at the sides of the beams deformed in the 3D slip mode, as they obviously represent significant elastic strains. Since at the beam sides only  $\sigma_x$  acts, all the strains may be neglected save those in the x-direction. A simple examination of the theoretical shape of a beam deformed without elastic constraints, as given in II.6, reveals that the depth of the beam measured along the lattice lines increases from the undeformed beam depth at the centre of the section to a maximum at both sides. This increase in depth must be accommodated elastically if only slip in the 3D mode is operative, and consequently should result in a tensile stress  $\sigma_y$  which increases from zero at  $y=0$  to a maximum at the beam sides. However it was found that on the tension side of the neutral axis,  $\sigma_x$  was a tensile stress, but on the compression side became a compressive stress. This change in sign at the neutral axis can be accounted for by assuming that the neutral axis does not in fact become a hyperboloid, but remains a section of a circular cylinder. Since theoretically no slip has taken place at the neutral axis, this assumption may be at least partially justified, but doubtless, under the influence of the elastic constraints it becomes at least slightly curved. However, assuming that its transverse curvature is substantially less than that of the



All values computed with equations of II.6, assuming the neutral axis straight in  $y$  direction, and 10% outer fibre strain.

Fig. 62

rectangular hyperboloid predicted,  $\sigma_x$  would be expected to be a tensile stress on the tension side and a compressive stress on the compression side of the beam, and further to increase rapidly in magnitude as the sides of the beam are approached.

Under static equilibrium conditions  $\sigma_x$  should reverse sign in the central portions of the beam, but maintain the same sign at and near the sides of the beam where its maximum values are attained. Such a stress distribution is shown in Fig. 62. Doubtless these stresses make a further significant contribution to the elastic strain energy of beams that have been deformed exclusively in the 3D bending mode. Furthermore they probably account for the appearance of slip in the 2D mode at the sides of some of the beams otherwise completely deformed in the 3D mode. This secondary slip was observed at strains as low as 1.5% and is probably of the wrong sign to accommodate the major bending curvature.

Thus it can be concluded that the much greater amount of work necessary to deform a beam in the 3D mode as opposed to the 2D mode may be accounted for by the elastic strain energy remaining in the beam after deformation.

Undoubtedly one of the most convenient ways of plastically deforming single crystals which cleave into orthorhombic specimens is by bending. This method of deformation has been widely used by many investigators to determine the mechanical properties of crystals of the rock-salt structure and innumerable conclusions have been drawn from their load-deflection curves. However, as a result of the work reported here it is concluded that of all the factors influencing the work hardening rate, the one with the most profound effect in specimens undergoing pure bending deformation is simply the orientation of slip.

The effect of slip orientation is independent of the material investigated and depends simply on the components of the Burgers vectors when resolved in the cross-section plane of the beam. If the component is normal to the neutral surface the slip may be defined as 2D and the beam will deform as described by Road (1957) with a relatively low rate of work hardening, but if the component is not normal to the neutral surface the slip must be classified as 3D. Assuming that the beam is not allowed to twist when slip in the 3D mode occurs, the lattice must rotate about the major bending radii to permit the necessary extension and contraction. As the lattice rotates the transverse plastic strain is incompatible with the longitudinal plastic strain and elastic strains result. These elastic strains may be expected to be very small if the cross-section component of the Burgers vector is nearly normal to the neutral surface, but to increase to a maximum when the component is parallel with the neutral surface as in cleaved beams of MgO, LiF and NaCl. Though zero when the angle between the direction of slip and the cross-section plane is  $45^{\circ}$ , the elastic strain increases rapidly as the angle either increases or decreases from this value.

Thus it may be concluded that the use of bending tests to determine the energy associated with any of the aspects of plasticity in single crystals is highly suspect unless very careful attention is paid to the elastic energy associated with the orientation of slip. Furthermore, it may be stated that the use of three-point bending

to deform single crystals of the materials described herein, and probably any other materials with few slip sources, is particularly unsuitable for determining their intrinsic plastic properties, the load-deflection curves rendering little information other than the characteristics of a three-point bend test.

However, in a more positive vein, the results indicate a possible method of increasing the strength of single crystal beams. This may be done by preparing beams so that when they are stressed in bending slip occurs only in a direction parallel with the neutral surface and inclined to the cross-section at an angle different from  $45^{\circ}$ . The details of how this might be accomplished will vary from material to material and must be worked out for each individual case.

Although in the work reported here the investigation of the effect of slip geometry was limited principally to  $\text{MgO}$ , with some work being done on  $\text{NaCl}$  and  $\text{LiF}$ , it undoubtedly should be extended to other materials that are commonly deformed in bending. However the feasibility of employing photoelastic techniques to investigate internal stresses makes the continued use of transparent ionic crystals most attractive for further investigations of the bending phenomena.

An attempt should be made to examine the stresses in the cross-sections of beams while they are still under load, and a method should be devised for preparing beam sections of known lengths in order to permit a rigorous quantitative examination of the stresses. This investigation should be fully extended to  $\text{LiF}$  and  $\text{NaCl}$ , and even better from the photoelastic standpoint,  $\text{AgCl}$ , since its stress-optic constants are very high. Such an investigation should provide sufficient information to permit the accurate checking of the stresses predicted by the slip model, and additionally an experimental estimate of the elastic strain energy in the beam.

Another method of investigating the effect of slip in the 3D mode that might prove fruitful would be the development of a technique for accurately determining both the shape of the beam cross-section and

the rotation of the slip planes, preferably while the beam is still under load.

A series of experiments to determine the effects of the relative lengths of the glide paths on the establishment of slip in one mode or the other might provide further insight into the problem of the eventual predominance of slip in one mode. Related to the same problem, an investigation of the effects of changes in the strain rate on the pattern of slip in beams would probably provide additional valuable information and may also shed some light on the phenomenon of kinking.

Finally, the author feels, as these conclusions indicate, that the work presented here though far from being complete will both permit better understanding of the plastic properties of bent crystals and provide a starting point for many fruitful investigations.

ACKNOWLEDGEMENTS

The work described in this thesis was carried out in the department of Metallurgy under the general direction of Professor J.G. Ball to whom appreciation is expressed for laboratory facilities and the arranging of financial support.

The author is also greatly indebted to his supervisor, Dr. P.L. Pratt, for his advice and guidance throughout the course of the work.

Thanks is due to the group at Harwell working on MgO under the supervision of Dr. F.J.P. Clarke for helpful criticism and discussion, with particular thanks to G. Halister, for providing the photoelastic data for MgO.

The advice and aid of Miss P. Martins is all things photographic was greatly appreciated.

Finally, the author wishes to express his thanks to U.K.A.E.A. Harwell for their generous provision of both the necessary funds and equipment.

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