The behaviour of face centred cubic metals and alloys
under simple reversed stresses.

A thesis submitted to the University of London for the
degree of Doctor of Philosophy

by

MARTIN PATRICK DESVAUX
This thesis is an account of slip line investigations carried out at room temperature on single crystals of pure copper and a 12 at. % copper-aluminium alloy deformed under reversed stresses in tension and compression. Measurements on copper were made by the statistical determination, from surface replicas viewed in the electron microscope, of tensile and compressive slip line densities before and after a compressive strain which followed a tensile prestrain. Slip line densities were counted on a small region of the surface which had been re-electropolished after the tensile prestrain, and on an adjacent area which had not been re-electropolished. The difference found between compressive slip line densities in the two regions suggests that partial reverse slip takes place in copper.

For copper-aluminium the electron microscope was used in conjunction with a new replica technique which enabled small changes in slip step height to be detected. New light is thrown on the mode of deformation of this alloy in tension, and under compression following a tensile prestrain, reverse slip is shown to occur. This reverse slip has been investigated in relation to the Bauschinger effect and it is caused by the dislocations generated during the tensile half cycle of deformation. Data concerning the Bauschinger effect is given for the alloy and the Bauschinger strain is found to be sensitive to the ageing time. Obstacles to dislocation movement which are set up in tension, break down in compression. The results are discussed in the light of current theories of cyclic strain hardening and the Bauschinger effect. A short review of the theories and experimental work which are relevant to cyclic strain hardening and to the Bauschinger effect are given.
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The stresses quoted in table 2.2, p.31, are critical resolved flow stresses, whilst those quoted in table 2.3, p.32, are applied stresses.
CHAPTER I

A brief review of the principal experimental findings relevant to the cyclic hardening of f.c.c. materials.

1.1 Introduction.

If an annealed single crystal of a pure metal is deformed in unidirectional tension, the stress required to continue the deformation must always be increased, until the crystal approaches the point at which it will fracture. This effect is known as unidirectional work-hardening. If the same crystal had been deformed by cycling under reversed stresses, the behaviour would be qualitatively the same and is known in the literature as cycle strain hardening. For alloys, the same behaviour is generally observed except that under some conditions hardening is initially absent (see section 1.2). For single crystals cyclic strain hardening can be defined as the increase in resolved shear stress per cycle of deformation required to continue the plastic deformation to the end of the cycle, divided by the glide strain in that cycle; the glide strain being summed irrespective of sign.

The plastic deformation of a metal, be it of an unidirectional or cyclic nature, involves the movement and interaction of dislocations. These can interact with each other, or with point defects, and it is generally accepted that their behaviour is the fundamental cause of work-hardening in metals and alloys. When dislocations, which in f.c.c. materials travel on close packed \( \{111 \} \) planes in close packed \( \langle 110 \rangle \) directions, intersect the crystal surface a step is formed whose magnitude is equal to the Burger's vector of the dislocation \( (1,2,3) \). The behaviour of dislocations is therefore to a certain extent
reflected in the pattern of slip lines on the surface of the material in question. This thesis describes experiments which, through a study of the slip line patterns on the surface of copper and a 12 at. pct. copper-aluminium alloy deformed under reversed stresses in tension and compression, throw new light on the behaviour of the dislocation processes involved in cyclic hardening.

A typical cycle of deformation proceeds in the following stages: The crystal is pulled in tension, yields plastically; it is unloaded and compressed so that plastic flow sets in and is unloaded again. The relevant literature concerning the behaviour of f.c.c. metals and alloys during this cycle will be reviewed in the next section. It is not possible, or even relevant, to give a complete review of the facts, but most of these will be found in the review articles cited.

1.2 The work-hardening of single crystals of f.c.c. materials in unidirectional tension.

The stress-strain curves of f.c.c. materials exhibit three distinct regions called respectively Stage I, Stage II and Stage III (4,5,6). Stage I represents a strain interval during which hardening is low for pure metals or zero in the cases of some alloys. Stage II is an interval of rapid linear strain hardening and in Stage III the hardening rate decreases gradually to zero with strain, and becomes negative just before the crystal fractures.

The dislocation distribution in pure copper, as found from etch pit studies and observations by thin foil electron
microscopy, in Stage I is very non-uniform. Dislocations are arranged in bundles separated by $\sim 5-100 \mu m$, the intervening region being virtually free of dislocations ($7,8,9,10$). The dislocations are mainly of edge nature and it is thought that screw dislocations may annihilate through cross-slip ($7,8,9$). Many edge dipoles on the primary system are seen ($7,8,9,11,12$). Livingston (13) has shown with etch pit techniques, that pile-ups begin to occur against sub-boundaries towards the end of Stage I in pure Cu. The presence of impurities has the effect of increasing the flow stress and decreasing the length of the easy glide region (14). Replica studies of the surface of copper in Stage I (24,25) show long slip lines of height $\sim 30 \AA$ which have an average spacing of $\sim 300 \AA$ which remains constant with strain at glide strain values of $\sim 2-3\%$.

In copper alloys Stage I begins after an initial yield point (15,16). The rate of hardening as shown by the load–elongation curve is, to a first approximation, zero (15,16). This foil electron microscopy shows dislocations arranged in bundles and piled-up groups (32,33,7) both of which contain edge dipoles (7). Optical microscopy reveals that slip lines on $\alpha$-brass and $\alpha$-CuAl appear as large distinct steps, and cross-slip is always present during easy glide. Replica studies on $\alpha$-brass (19) have shown that the slip step faces are single $\{11\}$ planes and that the slip lines have widths which range from $\sim 50$ to $\sim 500 \AA$ (19,20,21). Etch pit studies on $\alpha$-brass show dislocations lying in piled-up groups along the primary and cross-slip planes (22,23).
In Stage II, dislocations in pure metals and low solute alloys arrange themselves into cell walls (27), the slip line length, and therefore the mean free path of the dislocations, decreases with the strain (26), and strong slip lines form on the crystal surface (24). In α-brass slip lines grow in height (23). In pure Cu (7,3), α-brass (4) and α-CuAl (8) barriers of the Lomer-Cottrell type are observed, and the density of dislocations on systems other than the primary increases rapidly (7,8,10). In CuAl, of high solute content, the slip distance is estimated to be $\sim 400\mu(7)$. Stage III in pure metals is accompanied by the formation of fragmented slip bands and cross-slip (6), and this effectively increases the mean free path of the dislocations. Further details of Stage III which is not important to the present work will be found in references 4,5 and 6.

1.3 Stability of deformation during unloading.

When the stress is taken off a crystal deformed in tension and reapplied in the same sense a small amount of hysteresis is observed (30) indicating that some dislocation rearrangement takes place during the unloading. A small amount of reverse plastic deformation has been noted by other workers (31) during the unloading interval. Galligan (35) has observed a small low hardening region on reloading copper single crystals in Stages I and III, which is attributed to stress relaxation. On cyclically deformed crystals the unloading curve is seen to exhibit plastic flow in the sense opposite to the prestrain (45,41).
1.4 **The Bauschinger effect.**

An intrinsic feature of cyclic strain hardening is that after a tensile prestrain many materials are less resistant to a compressive stress. This hardness anistropy is called the Bauschinger Effect after its discoverer (36). The effect has been studied by many workers, earlier experiments being centred on polycrystalline materials (36, 37, 38). Later, single crystals were investigated and a Baushinger effect has now been established in polycrystals and single crystals of α-brass (39, 40), Aluminium (41, 42, 43, 44, 45, 46, 48), copper (38, 45, 46, 47) and other materials (38, 45, 43, 49, 50).

To understand the principles and parameters involved in the Bauschinger effect, consider an annealed crystal of, say, copper. The unstrained state in fig. 1.1 is represented by 0. If the crystal is plastically deformed to a stress of \( \sigma_0 \) and unloaded at B down to C then a forward plastic prestrain of 0C has occurred. Redeforming in tension will cause the curve CBE to be followed, but redforming in compression gives rise to CDE (plotted positive for convenience). The strain represented by \( \varepsilon_B \), is called the 'Baushinger strain'. The value of this depends on the value of \( \sigma_B^* \), which was chosen to be \(-0.75\sigma_0^*\) and \(-\sigma_0^*\) by Buckley and Entwistle (44) and \(-0.75\sigma_0^*\) by Woolley (45). In this thesis \( \sigma_B^* \) will be used to denote the stress value at which \( \varepsilon_B \) was measured.

The most thorough measurements of the Bauschinger effect carried out so far were made by Buckley and Entwistle (44).
on aluminium single crystals. The main features as summarized by van Bueren (51) are briefly:

a) $\epsilon_b$ is approximately proportional to $\sigma_0$, the prestress level.

b) $\epsilon_b$ is uniquely related to the degree of work-hardening. The higher the rate of hardening in tension, the lower is the rate of increase of $\epsilon_b$ with strain.

c) $\epsilon_b$ is not affected by electropolishing after the prestress and is not correlated with the slip markings on the surface. During the compression half cycle, new slip markings, as observed optically, appear only after $\sigma_b = -\sigma_0$ has been exceeded.

d) The maximum value of $\epsilon_b$ is about a tenth of the value of the prestrain and the Bauschinger effect appears to be resistant to a high temperature anneal.

The last point is surprising since the Bauschinger effect is presumably due to dislocation arrangements which are set up during the tensile prestrain, and it is known that these anneal out quite well at the temperatures used by Buckley and Entwistle (820 °C) in aluminium. Furthermore, Corten and Elsesser (40) have found that the Bauschinger effects in low-carbon steel and $\alpha$-brass anneal out after heating the former to 820 °C and the latter to 149°C for 20 hours. This is contradictory to Buckley and Entwistle's statement, which is based on the observation that occasionally a specimen strained initially in compression had a higher flow stress than one (cut from the same crystal) strained
initially in tension, thus constituting a residual Bauschinger effect in tension. This being the exception rather than the rule, they assumed that the single crystals in question had been strained in the mould (this would have had to be a compressive strain) and that the pre-test anneal at 320°C did not remove the residual strains. A more plausible explanation would be that the as-grown crystals had had an impurity gradient present. This could have arisen from occasionally imperfect raw material, or from impurities occasionally being injected during the growth process. The modified Bridgeman crystal growing technique would then have had a zone refining effect on the impurities and a higher impurity content in one end of the crystal would have resulted. Since it is well known that impurities raise the flow stresses of crystals (14), then the compression specimen was cut from the more impure end of the single crystal. The fact that this anomaly did not occur regularly, and that the shapes of the stress-strain curves are sensibly the same in both directions of deformation makes this explanation feasible. Charlsley (66) has also observed this type of anomaly on aluminium single crystals.

The single crystals of Buckley and Enwistle were exhaustively investigated, and their polycrystalline measurements on Aluminium are in good qualitative agreement with those of Woolley (45) on polycrystalline copper. Furthermore Woolley finds no grain size dependence, in copper, of the Bauschinger effect, and the assumption that single crystals of copper have the same qualitative Bauschinger effect behaviour as aluminium
is a reasonably safe one. This reasoning is confirmed by the measurements of Phillips (40) on copper single crystals. His measurements on aluminium single crystals agree quantitatively with those of Buckley and Entwistle.

1.5 Cyclic strain hardening.

The field of cyclic strain hardening can be thought of as being entered once the compressive stress exceeds the value of \( \sigma_0 = -\sigma_c \). The two main types of experiment which can be performed can be summarized as:

a) Constant stress amplitude tests, and
b) Constant plastic strain amplitude tests.

In the first type, hardening is indicated by the gradual decrease of the hysteresis loop width and in the second by the increase in stress required to deform the crystal to the end of the cycle.

The first type of test has been carried out on copper (31, 47, 52) and aluminium (54) single crystals, and the second type on copper (41, 55) and aluminium (42, 43) single crystals. Constant stress amplitude experiments show that the rate of hardening depends on the stress amplitude and crystal orientation. The same kind of variation of cyclic hardening is found in both types of tests and the results of Kensley and Patterson (47) which are of type (b) and typical of pure metals will be summarized here. The main features are:

(i) The cyclic plastic strain hardening rate is lower than unidirectional strain hardening rate.

(ii) For glide strain amplitudes of less than \( \approx 0.004 \)
the rate of cyclic hardening has an orientation
dependence which is similar to that of unidirectional
strain hardening. Orientations near the centre of the
stereographic triangle have low hardening rates whilst
as the \([100]\), \([311]\) and \([111]\) corners of the triangle are
approached, the hardening increases. Hardening is
higher for \([100]\) and \([111]\) orientations than for \([110]\).
For higher strain amplitudes the rate of hardening is
lowest for the near \([110]\) orientations and highest for
\([111]\) and \([110]\), the centre of the triangle giving
intermediate values of hardening.

(iv) The cyclic strain hardening increases with plastic
strain amplitude.

(v) Optical microscope observations on slip lines
indicate that their spacing was sensibly constant.
Single glide orientations give uniformly distributed
slip on the primary slip system. The appearance of
secondary slip, for orientations near the \([110]\) - \([100]\)
symmetry line is accompanied by a marked increase in
the hardening rate. Lower strain amplitudes give less
intense slip. For high hardening rate crystals
considerable amounts of secondary slip occur and this
was generally for orientations near the \([100]\) - \([111]\)
symmetry line, where a noticeable clustering of slip
lines is also observed. The latter effect decreased
for orientations nearer to the centre of the stereographic
triangle where only uniform diffuse slip was observed.

Avery and Bockofen (53) have shown by constant strain amplitude tests in bending that the cyclic strain hardening rate falls off exponentially with stacking fault energy (usually denoted by $\gamma$) over the range $\gamma < 100 \text{ ergs/cm}^2$, there being an anomalous point at $\gamma = 20 \text{ ergs/cm}^2$.

1.6 Dislocation behaviour during cyclic hardening.

Dislocation arrangements during cyclic straining have been studied in Cu (56,57), Al (59,54), Ni (56), Au (56) and $\alpha$-brass (58,67). In all cases many dislocation loops are found even after 1 cycle of deformation (54). After 100 cycles, pure metals show that dislocation tangles form (54), and cell structures form after several thousand cycles of deformation (55,57). In $\alpha$-brass after 50 cycles dislocations are still grouped on well defined slip planes (58).

Hirsch et alia (60) have shown that dislocations just beneath the surface correspond directly to the surface slip steps in strain-cycled stainless steel, which has similar dislocations arrangements to high solute Cu alloys. In some cases dislocations are observed where there is no slip step. This result indicates that the dislocations have undergone equal amounts of to and fro movement, a phenomenon which will be called 'reverse dislocation motion', and which gives rise to 'reverse slip'; the slipping back in compression of a slip step formed in tension or vice versa. Pond and Harrison (61) used optical cinematography to investigate reverse slip in aluminium single crystals and claim to have
evidence for it. Charsley and Thompson (63) have looked at the slip line structure on cyclically deformed Al single crystals, using the optical microscope but found no evidence of reverse slip. Some of Charsley and Thompson's crystal surfaces were mechanically polished prior to electropolishing, which would have caused a certain amount of hardening, especially since fine slip was absent. Louat and Hatherly (63) reported one observation on Al which suggested the occurrence of reverse slip, but like all the observations made in this field, the horizontal resolution was not better than 2000Å. This means that the argument that slip takes place on an adjacent plane cannot be discarded, especially in view of Mader's (24) observations on slip line separations on copper. Brown (64) has made electron microscope replica observations on slip bands in Al, and has found that compressive slip bands often appear near those formed in tension. Phillips (46) and Buckley and Entwistle (44) report no apparent reversal of slip in the Bauschinger strain region of the stress-strain curve. However the latter (44) readily admit that, due to the smallness of the Bauschinger effect, strain reversal could occur without being noticed. Phillips and Robertson (13) examined crystals of α-brass with optical microscope techniques but could see no evidence for the reversal of slip lines within the slip clusters.

1.7 Concerning this thesis.

In the remaining chapters of this thesis, experiments will be described in which pure Cu and 12 at. pct. Cu-Al
(referred to as CuAl throughout) are deformed through up to four cycles of plastic deformation at a temperature of \(200 \pm 5^\circ\text{K}\). The slip line patterns, as observed by optical microscopy and by replicas in the electron microscope, and their relationship to the Bauschinger effect and reversed stresses exceeding the \(\sigma_C - \sigma_0\) region, will be described. The results will be viewed in the light of modern theories of cyclic hardening. Copper was chosen as a material since a considerable amount of data concerning its tensile and cyclic hardening properties has already been published. In addition it was thought that since the oxide film is not as serious as in say Al, the only other well investigated f.c.c. pure metal, the slip line results would have more meaning. CuAl was chosen since it had much larger slip steps on the surface and slip step heights and changes of the same could be more accurately detected. The use of such a material enabled a new technique to be used to study slip lines, thus giving more information on the behaviour of this type of alloy. It is also useful to compare CuAl with Cu since their stacking fault energies are considerably different.

Chapter II describes the preparation of single crystal test pieces, and chapter III describes the experimental techniques used to obtain the results. The CuAl results are separated into two chapters, chapter IV describing the slip line patterns obtained in simple tension and chapter V giving the reversed stressing results. Chapter IV is included since replica observations of slip on CuAl have not been reported in the
Furthermore, new information on the mode of deformation of this material in tension, is presented.

Chapter VI gives results obtained on pure Cu, being divided into unidirectional tension results and reversed stressing results.

Chapter VII gives a short review of the theories involved in the type of cyclic hardening observations described in this thesis.

Chapter VIII discusses the reversed stressing results in the light of the current theories to account for cyclic strain hardening.
Diagrammatic Representation of the Mauzinger Effect.
CHAPTER II

The preparation, mounting and deformation of single crystals.

2.1. Introduction.

When studies are made on the mechanical behaviour of a metal, it is important that the previous history of the metal in question is known, otherwise little value can be placed on the results obtained. To perform a series of experiments, such as those to be described, it is desirable to have a reasonable degree of uniformity in the crystal preparation. Care must be taken to reduce any unwanted impurities, internal stresses, strains or any changes in the fixed parameters such as orientation, dislocation density and specimen dimensions. If any fluctuations occur they their magnitude should be known. This chapter deals with the growth and preparation of a single crystal specimens used in this research, the method laid out being adhered to for all the crystals examined. It was found, in practice, that despite the apparent uniformity of preparation, variations of flow stress and easy glide, especially in copper crystals, were observed. These will be discussed in the relevant results chapter.

2.2. Growth and seeding of single crystals.

All single crystals were grown by a modified Bridgeman technique. The details of the raw materials and their purity are given in tables 2.1a and 2.1b; all were supplied in 7mm. diameter, rod form, the only exception being crystal CA1 which was grown from raw material of 3mm. diameter.

After a 15 cm. length of Copper raw material had been electropolished (details are given in Sect. 2.5.) and washed, to remove surface contamination, it was placed in a mould of high purity graphite (obtained in rod form from the Morgan Crucible Co. Ltd.) of dimensions shown in fig 2.1. The mould was placed in an evacuated,
aluminous porcelain furnace tube, which was suspended from a motor and hung inside a vertical tube furnace. The furnace had an 18cm. long, uniform temperature region whose temperature was held constant at 1150°C to within ±5°C. The growth rate was 2cm. per hour, and the molten metal first nucleated in the 'V' at the bottom of the mould, and then grew into a single crystal of the same orientation as the nucleus.

The procedure for CuAl was the same, except that the graphite mould was enclosed in a stainless steel casing (see fig 2.2). This was to prevent a composition change of the alloy by evaporation of aluminium from the melt. Any air left inside the mould did not seem to affect the crystal.

Crystals grown in the above manner had random orientations; with copper there was a tendency for the crystal axis to grow near the (001)-(111) symmetry lines. Since a single glide orientation was required, a suitable seed of each material was selected, and used to grow others of the same orientation. The 7cm. long seeds were cut using the Servomet (Type SMC) spark machine on range 4, and electropolished before insertion into a 22cm. long mould. 15cm. of raw material was placed on top of the seed and grown to the required orientation. After furnace cooling, the crystals were etched in a mixture of 2:1 methanol/fuming nitric acid. This etch revealed grain boundaries and sub-grain boundaries, and crystals containing a serious number of these were rejected.

2.3 Orientation of single crystals.

Crystals were oriented using the Laue back reflection method. The spots obtained on the photographic plate were usually circular, but occasionally showed a small degree of splitting,
indicating sub-boundaries, which was never more than $1^\circ$ of arc. The maximum error in the tensile axis determination was $\pm 2^\circ$ of arc. The orientations of the crystals used are given in fig. 2.3.

2.4. Preparation and annealing of the test pieces.

The as-grown crystal was spark-cut to three 7.3cm lengths, one of which was the original seed and was kept for further crystal growth. The other two were lathed into tensile test pieces (dimensions of which are shown in fig. 2.4) using a Gervomet Lathe attachment. The spark lathing was done on progressively finer ranges and the gauge length was always finished on range 7 which provided a smooth finish. The variation in diameter along the gauge length was always less than 0.0003cm. After a light electropolish, all the test pieces were annealed in a vacuum of $10^{-3}$ atm., according to the data given in tables 2.2 and 2.3. Since there was no recrystallization during the anneal, the whole method was considered to produce a single crystal test piece which was strain and damage free after annealing. To check this an additional test was carried out. A spark machined test piece of copper, and an as-grown crystal of copper, of the same orientation, were annealed together at 970$^\circ$C for 16 hours, and pulled in tension. The critical resolved shear stress of the former was 128 gms./mm$^2$, and of the latter 148 gms./mm$^2$, showing that spark machine damage was successfully removed by the subsequent anneal.

2.5. Electropolishing method.

The accurate observation of slip lines in the electron-microscope can only be carried out if the surface conditions of the
metal, from which the replicas are to be taken, are favourable. Hence, prior to deformation the surface must be smooth and free from any thick oxide layers. This was fulfilled in practice by electropolishing the gauge length prior to deformation. The details of the electropolishing system used throughout this work were as follows:

Solution: Equal parts of orthophosphoric acid (S.G. = 1.75) and distilled water.

Cathode: Copper.

Volts across cell: 3.2.

Current density: 0.25 amps./cm².

Temperature: 20°C.

Time: 3-15 mins. according to the amount of surface removal required.

Washing solution: Distilled water followed by pure methanol.

Stopping off medium: Lacomite (Cannings & Co. Ltd., Birmingham)

These conditions gave a smooth relatively oxide-free surface on both materials. The method was essentially taken from Tegart (34), adjustments being made to obtain the best operating conditions.

Crystals F8, F9, and F9 had to be polished at 1.3v otherwise excessive pitting occurred making the surface unsuitable for observation. These three crystals were the only ones which showed this peculiarity and it was thought to be due to the presence of sulphur impurities in the crystal, which were acquired by the accidental usage of a contaminated furnace tube. On analysis of the composition of F9 (carried out by Johnson, Matthey & Co. Ltd.) the purity was found to be as shown in the second column of table 2.4. This confirmed the author's suspicions and also accounted for the high flow stresses observed in these crystals (see also section 6.4).
Since the polish attack, above the electropolishing plateau, was only serious in Copper containing a high degree of impurities, it was concluded that the impurities are a necessary part of the pitting process. Electropolished surfaces were covered with a layer of Formvar to protect them from oxidation by the atmosphere. This was found to be an effective method of keeping the surfaces from deterioration.

2.6 Selection of the observation region

On Copper, observations were usually made along the centre 2mm. part of the gauge length. It was therefore necessary to mark this region in a non-destructive way before proceeding further. This was usually done (experiment 02 was the only exception) by polishing a trapezium shaped area (2mm. longside x 2mm.) at the centre of the gauge length, the long side of which lay along the plane containing the Burgers vector as shown in fig. 2.5. This was so that the slip lines could be observed where their height was a maximum. The amount of surface removal was about 10μ. The whole gauge length was then electropolished leaving a shallow trapezium shaped indent on the crystal surface. The indent could be seen on replicas taken from the surface later on, and thus enable the same region of gauge length to be viewed successively in the electron-microscope.

The plane containing the Burgers vector and the crystal tensile axis was determined by X-rays, in conjunction with a special crystal holder which allowed rotation of the crystal, through any angle, about its axis.

Observations on CuAl were made along the entire gauge length, and hence it was only necessary at this stage to determine
the Burgers vector azimuth and to electropolish the gauge length.

2.7 Mounting of the test pieces.

The electropolished test pieces were mounted into a pair of accurately machined knobs which had a 6.50 mm. internal diameter. To ensure a close fit the ends of the test pieces were always machined to about 0.002 cm. oversize to allow for surface removal during the various electropolishes. If they were still oversize, by the time they were ready to mount, then the ends were electropolished down until a sliding fit into the knobs was obtained. This method of preparation ensured that the specimens were a good fit into the knobs, thus reducing the risk of buckling under compression. The test piece was held in each knob by tightening three grub screws in rotation so that the correct alignment could be achieved. The alignment was checked by gently rolling the knobs, with the inmounted test piece, along an accurately flat steel plate, and looking for air gaps between the knobs and the plate. The alignment could then be adjusted by tightening the appropriate grub screw. The final alignment of the test piece with respect to each knob was always better than about 20 seconds of arc. The amount of bending strain on the crystal surface would therefore always be less than 0.0005 which is well below the experimental error of the results to be described.

The gauge length of one copper crystal was checked by replica for slip as a result of mounting by the grub screw method. No slip could be detected on the gauge length except at the fillet, so that observations made at the centre of the gauge length were free from any end effects. In CuAl no deformation could be observed, anywhere along the gauge length, as a result of mounting.
At this stage the crystal was ready for deformation.

2.3 The deformation of test pieces.

After mounting but before replication, the crystals were deformed in tension in a Hounsfield Tensometer (fig. 2.6a) at a strain rate of 0.0088 per sec. The accuracy of the stress measurements using this machine were ±0.3 kgm with the 31.5 kgm spring, ±0.6 with the 62.5 kgm spring and ±1.2 kgm with the 125 kgm spring. Strains were measured by means of traveling microscope determinations of the separation of two fiducial marks at the gauge length extremes of the crystal. The accuracy of strain measurements over a 3 cm. gauge length of the crystal was ±0.0007. Copper crystals 01 and 02 and CuAl crystals CA6, CA7 and CA9 were deformed in a Hounsfield type E tensometer with the same strain rate as above. Copper crystals were deformed using the 25 kgm load cell while CuAl crystals were deformed using the 250 kgm load cell. The accuracy of the stress values given by this machine is ±1% of the full scale deflection used. All stress values quoted for these crystals will be accurate to at least ±3% of their magnitude.

Compression was carried out using the tensometer compression attachment (fig. 2.6b) using the same strain rate as in tension. To reduce the chances of buckling, the knobs were keyed into the endplates of the compression attachment. This was effected by the insertion, into each of the knobs, of an accurately machined brass cylinder (fig. 2.6c), which was also located into a hole in the relevant end plate. The parallelism of the endplates was periodically checked by inserting a parallel ended block between them and checking for air gaps. Adjustments could be made by tightening the end plate locking nuts which can be seen in fig. 2.6a.
Once aligned no further adjustment was found to be necessary. The brass peg which can be seen in the left-hand knob in fig. 2.6a and the lower knob in fig. 2.6b prevented the brass cylinder from dropping out during deformation. The maximum possible movement of the knobs relative to each other in the compression attachment was 0.002 cm. The brass end pieces also served to maintain the knobs parallel to the crystal axis.
TABLE 2.1a. Details of raw materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Supplier</th>
<th>Purity</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>Johnson, Matthey &amp; Co. Ltd.</td>
<td>99.999</td>
<td>Spectroscopically pure.</td>
</tr>
<tr>
<td>CuAl</td>
<td>International Research and Development Ltd.</td>
<td>99.999</td>
<td>CA 1 – CA4: Between 11.6 and 11.7 at. pct. Al.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>CA 5 – CA9: 12.5 at. pct. Al.</td>
</tr>
</tbody>
</table>

TABLE 2.1b. Spectroscopic Analysis of copper.

<table>
<thead>
<tr>
<th>Impurities present</th>
<th>Quantity in p.p.m.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>1</td>
</tr>
<tr>
<td>Iron</td>
<td>1</td>
</tr>
<tr>
<td>Magnesium</td>
<td>1</td>
</tr>
<tr>
<td>Silver</td>
<td>3</td>
</tr>
</tbody>
</table>

- 30 -
<table>
<thead>
<tr>
<th>Crystal</th>
<th>Annealing time (hrs)</th>
<th>Annealing temp. °C</th>
<th>Length of Stage I</th>
<th>Orientation</th>
<th>Flow Stress ( \sigma )</th>
</tr>
</thead>
<tbody>
<tr>
<td>E1</td>
<td>16</td>
<td>1000</td>
<td>0.040</td>
<td>E1</td>
<td>130</td>
</tr>
<tr>
<td>E2</td>
<td>16</td>
<td>1000</td>
<td>0.030</td>
<td>E2</td>
<td>155</td>
</tr>
<tr>
<td>B1</td>
<td>16</td>
<td>370</td>
<td>0.012</td>
<td>B1</td>
<td>124</td>
</tr>
<tr>
<td>C4</td>
<td>15</td>
<td>950</td>
<td>0.018</td>
<td>C4</td>
<td>153</td>
</tr>
<tr>
<td>01</td>
<td>368</td>
<td>1020</td>
<td>&gt;0.010'</td>
<td>01</td>
<td>100</td>
</tr>
<tr>
<td>02</td>
<td>368</td>
<td>1020</td>
<td>0.013</td>
<td>02</td>
<td>33</td>
</tr>
<tr>
<td>F1</td>
<td>20</td>
<td>1000</td>
<td>0.005</td>
<td>F</td>
<td>200</td>
</tr>
<tr>
<td>F2</td>
<td>16</td>
<td>1000</td>
<td>0.000</td>
<td>F</td>
<td>253</td>
</tr>
<tr>
<td>F3</td>
<td>20</td>
<td>1000</td>
<td>0.016</td>
<td>F</td>
<td>144</td>
</tr>
<tr>
<td>F6</td>
<td>15</td>
<td>1000</td>
<td>0.016</td>
<td>F</td>
<td>150</td>
</tr>
<tr>
<td>F7</td>
<td>15</td>
<td>370</td>
<td>0.063</td>
<td>F</td>
<td>210</td>
</tr>
<tr>
<td>F8</td>
<td>112</td>
<td>1000</td>
<td>0.000</td>
<td>F</td>
<td>155</td>
</tr>
<tr>
<td>F9</td>
<td>112</td>
<td>1000</td>
<td>0.010</td>
<td>F</td>
<td>215</td>
</tr>
<tr>
<td>F10</td>
<td>438</td>
<td>1010</td>
<td>&gt;0.016'</td>
<td>F10</td>
<td>145</td>
</tr>
<tr>
<td>F12</td>
<td>438</td>
<td>1010</td>
<td>0.030</td>
<td>F</td>
<td>149</td>
</tr>
<tr>
<td>F15</td>
<td>438</td>
<td>1020</td>
<td>0.000</td>
<td>F</td>
<td>130</td>
</tr>
</tbody>
</table>

\[\text{Cyclically annealed crystals...Time of cycle 1 hr.}\]

\[\text{Crystals which were reverse stressed before the end of Stage I.}\]

**TABLE 2.2** Annealing and Deformation Data for Cu crystals.
### TABLE 2.3 Annealing and deformation data for CuAl crystals.

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Annealing time (hrs)</th>
<th>Annealing temp. °C</th>
<th>U. Yield Stress(_2) kgm/(\text{cm}^2)</th>
<th>L. Yield Stress(_2) kgm/(\text{cm}^2)</th>
<th>Yield drop kgm/(\text{cm}^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CA 1</td>
<td>--</td>
<td>---</td>
<td>2.80</td>
<td>2.54</td>
<td>0.26</td>
</tr>
<tr>
<td>CA 3</td>
<td>42</td>
<td>930</td>
<td>4.35</td>
<td>4.10</td>
<td>0.25</td>
</tr>
<tr>
<td>CA 4</td>
<td>16</td>
<td>900</td>
<td>3.04</td>
<td>3.04</td>
<td>---</td>
</tr>
<tr>
<td>CA 5</td>
<td>16</td>
<td>920</td>
<td>3.60</td>
<td>3.23</td>
<td>0.32</td>
</tr>
<tr>
<td>CA 6</td>
<td>16</td>
<td>920</td>
<td>4.25</td>
<td>3.73</td>
<td>0.53</td>
</tr>
<tr>
<td>CA 7</td>
<td>16</td>
<td>920</td>
<td>3.62</td>
<td>3.34</td>
<td>0.32</td>
</tr>
<tr>
<td>CA 9</td>
<td>16</td>
<td>920</td>
<td>3.27</td>
<td>2.97</td>
<td>0.30</td>
</tr>
</tbody>
</table>

### TABLE 2.4 Estimate of impurities present in crystals F9 and B1 as given by Johnson, Matthey & Co. Ltd.

<table>
<thead>
<tr>
<th>Element</th>
<th>Crystal F9</th>
<th>Crystal B1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cd</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>Cr</td>
<td>&lt; 1</td>
<td>-</td>
</tr>
<tr>
<td>Fe</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Mg</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>Ni</td>
<td>20</td>
<td>5</td>
</tr>
<tr>
<td>Ag</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>S</td>
<td>20</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>O</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>Si</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
</tr>
</tbody>
</table>
graphite cap

stainless steel cap

hole for suspending wire

graphite mould

stainless steel casing

FIG. 2.1

FIG. 2.2

SCALE 1:1
FIG. 2.3 Orientation of Single Crystals

LEGEND

CA 1

CA 5, 6, 7 and 9.

01 and 02

B 1

CA 4

All F crystals except F 11

F 11

EG 1 and EG 2
FIG. 2.4. Test Piece Dimensions

FIG. 2.5. Position of Trapezium shaped indent on the gauge length
CHAPTER III

Experimental techniques for the observation of deformation under reversed stresses.

PART I

3.1 Introduction.

Experiments concerning dislocation behaviour in materials have been tackled in many ways. The method chosen for this investigation was to observe slip line patterns on the crystal surface, by means of electron microscope replica techniques. Other methods, etch pit studies and thin foil electron microscopy, could also be used to obtain information in this field, but the chosen method was considered adequate and a more efficient method of obtaining results. There are objections to all three methods mentioned, but the advantage of slip line studies, is the lack of complicated techniques involved in obtaining results; replicas being simple to prepare in comparison with etch-pitting and the preparation of thin foils. The main objections to replicating slip lines is that the resolution of the replica is ~30 Å (67) and that there are conflicting views in the literature as to how representative slip lines on the crystal surface are of the dislocations in the bulk material. These will now be briefly reviewed.

3.2 The effect of the crystal surface on dislocations.

Dislocations which are near to the crystal surface experience attractive or repulsive forces according to the surface conditions (i.e. whether or not a surface oxide film is present; if the former case, then the elastic modus of the surface film determines the type of force that is experienced.) Friedel (2)
Kramer (69) has shown that the effect of some surface active agents is to increase the extent, and to decrease the hardening rate of, Stage I in Copper single crystals. Also Kramer and Demer (70) have shown that surface removal during deformation has this same effect on aluminium single crystals, indicating that the continual removal of the surface helps dislocations to leave the crystal. It was therefore proposed that, in deformed Cu and Al crystals, the dislocation configuration just below the surface was different from in the bulk metal. Alpha-iron (71) and Cu (55), in fatigue, have been shown to have surface dislocation arrangements which differ markedly from the bulk arrangement. In contrast to these observations, Swann (72) has shown, by looking at thin foils prepared from the crystal surface, that "at strains <15% the surface of electropolished Copper is not a strong barrier to dislocation motion compared to obstacles in the bulk". Hirsh et al. (60) have shown that, in stainless steel deformed in tension and fatigue, dislocations have a direct correspondence to the slip steps and that surface dislocation arrangements are similar to the bulk. Essmann and Kronmüller (73) show this for neutron irradiated Cu deformed in tension. Grosskreutz has found surface strains at slip steps, in cyclically strained Au (which is reputed to have no oxide film) and Al. He attributes this to the "incomplete relaxation of the crystal lattice around dislocations which have moved out of the surface during deformation" (74).

In the light of these observations it seems that the slip
line behaviour of pure Cu, deformed in tension or under reversed stresses of a few cycles, should be representative of the bulk dislocation distribution provided the total strain is <15%. The experiments to be described fulfill these requirements.

In CuAl, whose internal dislocation arrangements resemble those of stainless steel, it is easy to see how the dislocation arrangements in thin foils could give rise to the surface slip markings. Long pile-ups on slip planes separated by distances equivalent to the surface slip line separations can be seen. The number of dislocations in the pile-ups (33) account for the lowest range of slip step heights (100Å). Since only limited areas of thin foils can be observed then it is reasonable to assume that longer pile-ups exist to account for the larger slip steps.

**PART II**

The analysis and theory of slip line shadowing on Replicas.

3.3 **Introduction.**

Since the observation of slip under reversed stresses has, up till now, not been investigated in detail with replicas, it is necessary to give an analysis of replica slip line observations, which finds its application in the next three chapters. A general analysis of slip steps, formed under tension has been given by a few workers (19,76,77), but these are concerned with the analysis of large slip steps to determine the
degree of homogeneity of slip and the faithfulness of replicas in reproducing the crystal surface. Furthermore, an analysis of compressive slip lines in relation to already existing tensile slip lines has not been published and it is this aspect of replicas with which we shall now be concerned.

3.4 The assumptions involved in the shadowing of slip steps.

In what follows, two assumptions will be made. They are:

a) that the replica gives a faithful reproduction of the surface and does not buckle in such a way as to render the slip steps misleading.

b) The shadowing (Pt-C was used see section 3.12) intensity obeys a cosine intensity law analogous to Lambert's law for the intensity of illumination of a surface; the carbon arc is considered to be a point source as far as the slip steps are concerned.

Assumption (a) may not be true if large areas of replica are considered, but it should be valid over small regions (~10 microns sq) so that each slip line is represented faithfully with respect to its surroundings. The smaller the slip step height, the more valid this assumption be. The results in section 4.2 will be seen to support this assumption for CuAl, and it will therefore also be valid for Cu which has smaller slip steps.

3.5 Types of shadowing and contrast.

Slip line shadowing can be split into two main types
from which others may be built up. In the first case the step is shadowed face-on (called hereinafter "front shadowing") causing the step to appear darker than the surroundings, when it is viewed in the electron microscope. Fig. 3.1a illustrates this case. "Back Shadowing" is the second case, is when the slip step is shadowed from behind (Fig. 3.1b) and thus appears lighter than its surroundings. However if the step in 3.1a were shadowed from a direction normal to the crystal surface, i.e. front shadowed, a contrast condition equivalent to back shadowing would arise, so it is necessary to define these quantities more rigorously. This will now be done mathematically.

3.6 Mathematical representation of shadowing.

Consider fig. 3.2a $AB$ is a ray from a point shadowing source, $\Psi$ is the shadowing angle, $CDE$ is the slip plane and $\chi$ the angle between the slip plane and crystal surface at the point of observation. $\chi$ lies in the range $\Psi - \chi < \chi < \frac{\pi}{2}$ where $\omega$ is the angle between the tensile axis and the normal to the slip plane. Let $I_o$ be the normal intensity of $AB$ when it is near the slip step and $I_s$ the shadowing intensity on the surfaces $FC$ and $DG$.

Assuming Lambert’s cosine law, then:

$$I_s = I_o \cdot \cos (\Psi - \chi)$$

3.1

Calling the intensity of shadowing on $CD$, $I'$, it follows:

$$I' = I_o \cdot \cos (\Psi - \chi)$$

3.2

Defining the shadowing contrast, $S$, by

$$S = \frac{I'}{I_s} - 1$$

3.3

it can be seen that

$$S = \frac{\sin (\Psi + \chi)}{\sin \Psi} - 1$$

3.4
Equation 3.4 gives the contrast between the slip step and the surrounds in terms of slip step angle and shadowing angle, and $S$ should be as large as possible for the best observation conditions.

Consider now some important cases:

a) If $\psi \to 0$ (shadowing at grazing incidence) then $S \to \infty$.

The contrast increases to infinity, since in the limit there is no Pt-C deposit on the surfaces FC and DG, but a finite amount on the step CD. In practice it is not easy to get good results with this kind of shadowing since the unevenness of the replica causes it to become self-shadowed thus losing much of the replica to view. It was found that a good compromise was $\psi = 12^\circ$, which gave good contrast and ensured that the whole replica was shadowed.

b) Dangerous cases are solutions to the condition $S=0$. This condition must clearly be avoided, or no slip steps will be visible, and would lead to a misinterpretation of the results. The two solutions to eqn. 3.4 for the condition $S=0$ are, $\chi = 0$ and $2\psi = 180 - \chi$. $\chi = 0$ is unrealistic since the active slip planes in f.c.c. materials are never parallel to the crystal axis. The second case is when AB bisects angle CDG. CD, DG and FC then have equal shadowing intensities, to a first approximation, thus rendering the slip lines invisible.

The foregoing has been for the case of 'positive contrast', i.e.; $S>0$. It can be seen from eqn. 3.4 that if $\psi > 90 - \frac{\chi}{2}$ then $S$ becomes negative and this constitutes what will be called 'negative contrast'.

Summarising:

Positive Contrast: \[ I_s < I \quad \ldots \ldots \quad \psi < 90 - \frac{\chi}{2} \] 3.5

Negative Contrast: \[ I_s > I \quad \ldots \ldots \quad \psi > 90 - \frac{\chi}{2} \]

Relations 3.5 formulate a mathematical definition of the two types of contrast, for a tensile step. Compressive steps can be treated similarly and three cases are shown in fig 3.3a – 3.3c.

In CuAl only 3.3c was used, but for Cu, 3.3b and 3.3c were used.

3.7 Measurement of strain from slip line widths.

In fig. 3.2a the projection of DC on the tensile axis is ND. Assuming that the slip line is perpendicular to the tensile axis, then:

\[ ND = CD \cos \chi \quad \ldots \ldots \ldots \ldots \quad 3.6 \]

This is only an approximation since it does not take into account the fact that as slip takes place, lattice rotation occurs and the surface becomes slightly inclined to the tensile axis. ND is the width of the slip line as seen on the final image in the electron microscope, and is also the contribution of the slip step CD to the macroscopic strain. Hence for large slip steps:

\[ \sum \text{slip line widths on replica} = \frac{\text{total macroscopic strain}}{\text{gauge length}} \quad 3.7 \]

This relation will be used in section 4.2 and applies only to replicas with the shadowing angle in the range \(90 - \chi > \psi > 0 \).
If \( \Psi > 180 - \chi \), the image of the slip line is wider than the step, as can be seen from fig. 3.1b. For the compressive step shown in fig. 3.2b, \( w \) is the observed slip line width, but the contribution of the step \( BD \) to the strain is \( PD \). It can be shown, from fig. 3.2b, by elementary geometry that

\[
P_D = \frac{w \tan \Psi}{\tan \chi}
\]

3.8 Elementary structure slip steps on copper.

The foregoing only applies to slip steps which are large compared with the Pt-C layer. It applies, therefore, to CuAl, but not to pure Cu which has slip steps which are very small. In this case the situation depicted in fig. 3.4 arises. It can be seen that there would be expected to be an accumulation of Pt-C around the step, causing a broadening of the slip line. This was confirmed by measurements made on elementary structure slip lines on copper. Graph 6.2 shows that at a glide strain of 0.01 (which corresponds to a tensile strain of \( \sim 0.005 \)) there is a slip line density of \( \sim 7 \) lines per micron on the surface. Assuming a uniform slip line density over the gauge length the strain represented by 10 lines per micron is:

\[
\text{ND.7} = 0.0007 \text{ND} \frac{10,000}{10000}
\]

Where ND is in \( \frac{1}{\text{cm}} \). But this must be equivalent to the tensile strain (eqn. 3.7) so that:

\[
\text{ND} = \frac{0.005}{0.0007} = 7.3 \frac{1}{\text{cm}}
\]
This corresponds to a step height (CD in fig. 3.2a) of 11£. The measured value of $M_D$ from a micrograph taken from a region containing ~7 lines per micron is ~100£. Hence at low strains, slip line broadening occurs in copper, and the effective resolution of the replicas in this case is ~11£, in good agreement with Mader's estimate, at a strain 0.08, of 15 - 20£ (24).

**PART III**

The experimental technique used on copper crystals.

3.0 Combinations of tensile and compressive lines as seen on replicas.

When a metal is reverse stressed the compressive slip lines can arise very close to the tensile slip lines as in figs. 3.5 and 3.6. Consider first of all fig. 3.5. This is an intrusion on the metal surface, which appears as an extrusion on the replica. When the extrusion is shadowed it will appear as a black and white line close together. Since the minimum separation of slip lines formed in tension in Stage I is 100£ (24) and the slip steps height in the region is 50£ max. (this is a generous estimate in view of section 3.8) these should easily be replicated and not collapse, assuming that slip lines in compression do not form closer than 30£ to the tensile lines. (If they were any closer the replica would probably not reproduce them accurately). Fig 3.6 shows an extrusion which appears as an intrusion on the metal surface. It can be seen from 3.6c that this would be difficult to shadow if the tensile and compressive slip planes are
close together. Applying the ideas so far enunciated it can be shown that the black and white lines, caused by shadowing the intrusion (on the replica), would be beyond resolution if the slip planes concerned were closer than \( \sim 100\lambda \), thus an extrusion (on the metal) would appear like reverse slip. All the above arguments only apply in the cases of \( \psi > \sim 40^\circ \). If back shadowing were used then the extrusions would appear like white lines and would therefore be counted as a tensile line. The intrusion case would turn out the same but with opposite contrast.

Three other possibilities could occur and are shown in figs. 3.7b - 3.7d. They are partial reverse slip, total reverse slip and over-reversal respectively, and are self-explanatory.

3.10 Mathematical treatment of slip under reversed stresses.

Reversed stress measurements on Cu were usually carried out as follows. The crystal was pulled in tension to some forward strain \( \varepsilon_t \). A replica was taken from the surface region which contained the trapezium shaped indent (see section 2.6); and the slip line density, called hereinafter \( n'_t \), was measured. After region b was electropolished, the crystal was compressed by some strain \( \varepsilon_c \). Replicas were taken from both regions a and b. Region a contained both tensile and compressive lines, while b contained only compressive lines. After compression let \( n'_a \) be the compressive slip line density in region a, \( n'_b \) the compressive slip line density in region b, and \( n'_t \) the tensile slip line in region a.

When the stress is reversed there are six ways in which
the compressive stress could be accommodated. Most of them were discussed in the last section, but are summarized now for convenience.

(i) Compressive slip occurs on new planes remote from tensile slip. Let the no. of these events be N.

(ii) A fraction \( \alpha \) of the \( n_t^1 \) lines partially reverse, but not enough to shrink below the resolution of the electron microscope. (Fig. 3.7b)

(iii) A fraction \( \beta \) either totally reverse or become small enough as to be unresolvable. (Fig. 3.7c)

(iv) A fraction \( \gamma \) over reverse. (Fig. 3.7d)

(v) A fraction \( \delta \) form extrusions. (Fig. 3.6c)

(vi) A fraction \( \varepsilon \) form intrusions (Fig. 3.5c), but these will appear as black and white lines on the replica and will be counted as \( n_t^a \) and N respectively.

The no. of compressive lines \( n_c^b \) comes from the sum of cases (i) - (v):

\[
\dot{n}_c^b = (\alpha + \beta + \gamma + \varepsilon) n_t^1 + N 
\]

The value of \( n_c^c \) is the sum of (i) and (iv)

\[
\dot{n}_c^c = \gamma n_t^1 + N 
\]

and the value of \( n_t^e \) can be seen to be

\[
n_t^e = n_t^1 \left[ 1 - (\alpha + \varepsilon) \right] 
\]
The condition
\[ n'_t > n^a_t \]  
indicates a disappearance of tensile lines in region a after compression. This would be the case of reverse slip or very thin extrusions. If condition 3.10a is present, then
\[ n^b_c > n^c_c \]  
must also hold, since if slip line reversal occurs, then it must do so at the expense of compressive slip lines. It is possible for the condition
\[ n^b_c > n^a_c \] \[ n'_t = n^a_t \]  
to be present. This expresses that partial reversal of tensile lines has taken place. ie: no tensile slip lines completely reverse, and a 30\(^{\circ}\) slip line, say, shrinks to 15\(^{\circ}\). The 15\(^{\circ}\) line should still be visible in region a, and there will now be a 15\(^{\circ}\) compressive line present in region b. Hence the no. of tensile lines will be the same before and after compression, but the no. of compressive lines in region b will be higher than in region a.

If all the slip occurs on new planes in case (i) then no tensile lines disappear, and the compressive slip line density will be the same in both regions. ie:
\[ n'_t = n^a_t \] \[ n^b_c = n^c_c \]  
Consider now what happens when conditions 3.10a - 3.12 are applied to equations 3.9a - c. If the condition 3.12 is applied to equations 3.9a - c then it can be seen that:
\[ \alpha = \beta = \gamma = 0 = \delta \] and \( N = n^b_c = n^b_c \) \hspace{1cm} 3.13

Condition 3.11 gives the solutions that:
\[ n_t (\beta + \delta) = 0 \] \hspace{1cm} 3.14
\[ n^b_c = N - n'_t \] \hspace{1cm} 3.15

and \[ n^b_c - n^a_c = (\alpha + \beta + \delta) n'_t \] \hspace{1cm} 3.16

The possible solutions to 3.14 are:
\[ n'_t = 0 \] \hspace{1cm} 3.17
\[ \beta = \gamma = \delta = 0 \] \hspace{1cm} 3.18
\[ -\beta = \gamma + \delta, \gamma + \delta = 0 \] \hspace{1cm} 3.19

3.17 is not an acceptable solution. 3.19 has no meaning since it implies that a negative no. of compressive slip lines form under compression. (This is also true when any one of \( \beta, \gamma \) or \( \delta = 0 \) and the other two are finite). Therefore, 3.18 is the only realistic solution and is accepted, giving finally for this case that:
\[ \alpha = \frac{n^b_c - n^a_c}{n^b_c} \] \hspace{1cm} 3.20
\[ N = n^b_c \] \hspace{1cm} 3.21
All the quantities on the right hand side of eqns. 3.20 and 3.21 are known and hence the no. of tensile slip lines that partially reverse and the no. of new slip planes that become active under compression can be found.

Equations 3.9a-a cannot be solved explicitly for $\alpha$, $\beta$, $\gamma$, $\delta$, and $N$, when the conditions 3.10a and 3.10b are imposed, since there are five unknowns and only three equations. However it will be seen in Chapter VI that the only case that occurs is given by condition 3.11, and for this reason conditions 3.10a and 3.10b will not be considered further.

**PART IV**

3.11 Experimental techniques carried out on CuAl crystals.

Slip lines, which appear in tension, on CuAl vary in height from 50 - 3000$^\circ$2. This enables a different and more direct slip line study to be carried out on this alloy, when the above two area, electropolishing technique is employed. The method was the same as for copper as to the polishing and deformation procedure, except that the boundary between a and b extended along the whole gauge length. This was so that the whole gauge length could be examined; the reason for this will be clear in the next chapter. Replicas were taken from the boundary between a and b after subjecting the crystal to an "incremental" strain (i.e.: after redefining in tension or compression). This way the relative positions of the lines formed in the prestrain (visible in region a) and those formed
after the incremental strain (visible in region b) could be studied directly. Fig. 4.19 shows a portion of the boundary after the crystal had had a tensile incremental strain. The black tensile lines in b can be seen in some cases to originate from those in a. On analysing the micrograph, taking into account the shadowing direction, it is seen that some of the lines in b come from the 'base' or 'downhill' side of the original line in a, which is what would be expected if the original slip step had grown. Fig. 3.6 shows this in diagramatic form. The slip step formed in tension (shaded black) becomes progressively polished away as it goes into the re-electropolished region. The 'increment' of slip comes from the base of this step. The curvature of the slip increment is due to the strong local surface curvature at the boundary: a result of the electropolishing is that the same crystallographic plane as the surface is no longer being observed. The electropolished part of the original line (in region b) is sometimes bent in the opposite way to the incremental line curvature. This is due to a tendency for the replica to lie flat on the grid.

When the crystal is subjected to an incremental strain in compression, the positions of compressive slip lines can be studied in an analogous manner, as will be seen in Chapter V. This method of looking at slip steps in CuAl is extremely sensitive, and many of the results obtained could not have been observed since it shows up slip line width growth increments of as little as 30Å, which can be observed individually. The method is therefore more sensitive in this application than the
selected area techniques of Fourie (77) and Bradley (68), and has the additional advantages that no area invisible to the naked eye has to be selected, and only one replica need be made.

3.12 Preparation of Replicas.

Slip lines were studied in the electron microscope by use of a two-stage platinum-carbon replica technique, the intermediate stage being Fornvar.

After the crystals had been pulled in the tensile machine, a 2% solution of Fornvar in chloroform was poured onto the observation area. This was allowed to dry for about a minute, during which time the chloroform evaporated leaving a thin film of Fornvar plastic on the surface of the crystal. If the plastic was left for much longer than this, it was found to be difficult to subsequently strip off. After breathing on the plastic film to condense moisture on it, the former was stripped off using Scotch tape. It was found that if Sellotape was used, the cleaning procedure described below was virtually impossible, due to the fact that the gum used on Scotch tape was more soluble in petroleum ether than that used by the Sellotape manufacturers. The Scotch tape with adhering replica, was then mounted (with replica facing upwards) on a microscope slide and placed in an Edwards Coating Unit 12E/6, in such a position as to ensure the correct shadowing angle. The pressure was reduced to less than one micron and the replica was coated with a thin film of platinum-carbon. After letting the Bell jar down to atmospheric pressure, the specimen was moved to
directly underneath the evaporation source to avoid shadowing effects, and was then coated with a thin film of carbon at a pressure of about five microns. This ensured molecular scattering of the evaporated carbon, to form a uniform thin film of carbon over the entire replica. After removal from the Coating Unit, the area of the replica to be observed was cut out, using a fine scalpel blade. This was then placed in a Petri dish containing petroleum ether, to dissolve away the gum of the Scotch tape. When the dissolution was complete, the shadowed Formvar replica floated away and was caught on a 3mm grid, care being taken to ensure that the carbon surface was uppermost.

The grid together with the replica, was placed on a sloping copper gauze and chloroform from a drip source, was allowed to run along the gauze, thus dissolving the Formvar from between the grid and the platinum-carbon replica. If the replica had been placed upside down on the grid, then the Formvar did not dissolve away satisfactorily and the specimen was rendered useless. The final product was then ready for observation in the electron microscope.

3.13 Electron Microscopy

Replicas were examined using a Siemens Elmiskop I electron microscope. It is unnecessary to describe the mode of operation employed, since standard procedures were used throughout and no special attachments were required. It suffices to say that the replicas were mounted on 3mm grids and viewed with the microscope aligned on 60KV. After each
service the magnification was checked using a criss-cross diffraction grating replica, with 2160 lines per cm. in two perpendicular directions. The grating was obtained from E.J. Fullam & Co. Ltd. Photograph magnifications quoted in this thesis are accurate to ±2%. 
(a) Front shadowed

(b) Back shadowed

FIG. 3.1
FIG. 3.1c

(c) Critical shadowing

Image intensity

Negative contrast
Electron beam shadowing and electron beam direction

Pt-C film

Replica

Zero contrast

Positive contrast

Negative contrast

\( U = \text{Image intensity} \)

FIG. 3.3
FIG. 3.4
FIG. 3.5

- Shadowing direction
  - Pt-C film
  - Replica

- Intrusion

- Initial Step

- Graphs showing variations in u over x
**FIG. 3.6**

- **Initial Step**

- **Pt-C film Replica**

- **Extrusion**

- **Shadowing direction**

**u**

**x**
FIG. 3.7
Diagram of Electropolished Boundary

- Unpolished region
- Boundary formed during repoling
- Old slip line
- Incremental slip line
- Slip plane

b = Burgers vector
CHAPTER IV

The mechanical behaviour of CuAl under tension.

PART I Results.

4.1 Introduction.

Slip line observations in \( \alpha \)- CuAl alloys have been confined to optical microscopy (13, 17, 53). Koppenaal and Fine (16) have made observations with the optical microscope, and by the use of interferometric methods measured the slip step heights on the surface of a deformed 14 at. pet. CuAl crystal. They found that the observed steps accounted for only 42\% of the macroscopic strain, and suggested that optically unresolvable slip in other apparently undeformed parts of the specimen accounted for the major part of the macroscopic strain. Optical microscopy is therefore not sufficiently powerful to investigate the detailed slip line behaviour of this alloy, and replica work was undertaken to obtain more accurate information on the behaviour of CuAl under tension before going on to reversed stressing experiments. This chapter presents in part I, and discusses in Part II, results which throw new light on the way in which CuAl deforms in tension and shows that the suggestion of Koppenaal and Fine is not necessary.

4.2 The stress-strain curves for CuAl.

The results of this chapter and the next are compiled from seven crystals of CuAl which were all prestrained in tension. All the crystals, except CA4, showed a yield drop. Values of the yield stresses are given in table 2.3. Fig. 4.1 gives the stress-strain curve for crystal CA7. The yield drop, whose height will be referred to as \( h \), and width \( W \) was always followed by a zero
hardening yield point elongation zone as is usual for this type of alloy (16).

4.3 The change in shape of the crystal during deformation.

Attempts were made to correlate the stress-strain curve with the formation of slip markings. Ca5 will be described in detail since it was a typical example of how these crystals deformed. As the upper yield stress was reached, intense slip markings formed in a localized region near one end of the gauge length (fig. 4.2b). As the flow stress dropped to the lower yield stress, finer more widely spaced markings spread towards the centre of the gauge length, whilst the region which had initially slipped necked down (fig. 4.2c). At the lower yield point the necking stopped, and further deformation resulted in the shoulders of the necked region moving along the gauge length at constant load as shown in fig. 4.2d. It will be convenient if the deformed crystal is regarded as being comprised of three regions:

a) The lightly deformed region.
b) The transition region.
c) The heavily deformed region.

It was generally observed that the sharper the neck, the longer was the zero hardening region. Because of the strain being non-uniform along the gauge length, the macroscopic strain had little meaning when a localized region of the gauge length was being discussed, and for this reason the macroscopic strain in CuAl experiments will often not be quoted.
4.4 The lightly deformed region.

Optical microscope observations showed that this portion of the gauge length consisted of narrow slipped regions (fig. 4.3) separated by undeformed regions of average width 0.002 cm, measured on crystal CA5. The average separation varied in some undetermined way from crystal to crystal. Electron microscope observations showed that these narrow slipped regions were clusters of between ~3 and ~20 slip lines of varying widths, and no slip was visible between the individual clusters. Towards the transition region, the clusters became broader causing their separation to decrease. In general these clusters encircled the crystal, except at the part where the lightly deformed region faded out into completely undeformed material (marked as x on fig. 4.2c). Here the narrow clusters were only seen on one side of the crystal. The dislocations responsible for these were presumably crossing the slip clusters to spread across the crystal. Optical microscopy failed to show any cross-slip joining the individual slip clusters.

4.5 The transition region.

This region contained slip clusters in various stages of development. The nearer the heavily deformed region the broader the cluster, fig. 4.4 giving a typical example, and again replicas failed to show any fine slip in between the clusters. Optically visible cross-slip could be seen within the clusters and could occasionally be seen joining individual clusters.

4.6 The heavily deformed region.

When viewed with the optical microscope, this region
had the appearance shown in figs. 4.5, 4.6, and 4.7. The slip
clusters like those in fig. 4.4, had broadened and joined
together to form an almost continuously slipped region. The
dark regions involved with the 'long range' cross-slip in figs.
4.5 and 4.7 are parts of the specimen where little primary
slip has taken place. This is also seen in fig. 4.6 where the
region in the centre of the micrograph contains only a small
amount of primary slip. These 'blank' regions were always
traversed by heavy cross-slip and the latter will be called 'long
range' cross-slip since it extends over many tens of microns.
In the lower part of fig. 4.5 a much shorter type of cross-slip
can be seen and will be distinguished from the above type by the
name of 'short range' cross-slip.

The heavily deformed region of crystal CL5 was X-rayed
and a crystal axis rotation of $10^\circ$ towards $[110]$ was measured.

4.7 Cross-slip associated with etch pits.

In fig. 4.8 a large electro-polishing pit shows up as
a large black area. This type of observation was quite frequent
and in many cases cross-slip (figs. 4.4, 4.5, 4.8, 4.9 and 4.10)
was associated with the pit edges. This phenomenon could be
seen more clearly in regions of the crystal which had been
repolished and incrementally strained after a prestrain. This
type of cross-slip had also been observed in alpha-brass (18,20)
and in aluminium (91,90). The pit appears to divert the slip
into or around it, and fig. 4.11 shows an electron-micrograph
of this effect. Slip on the critical system can be seen in the
vicinity of the pit. This 'U'-shaped cross-slip pattern is not always near a pit and fig. 4.12 shows an electron-micrograph of an example which has taken place on the cross-slip system. Dislocations on the cross-slip system have double cross-slipped onto a parallel plane, and then back again. The 'cross-slip' plane in this case is on the primary slip system.

4.8 Long and short cross-slip.

Long range cross-slip also occurred in the transition region as in figs. 4.13 and 4.14. In fig. 4.13 the cross-slip is connected to a slip cluster at the right, (indicated diagrammatically) and out of view of the micrograph, and it extends over several tens of microns. Along the cross-slip traces shown in fig. 4.13, several primary slip lines are generated. At A the cross-slip stops abruptly and a primary slip line is connected to it. In reactions like at B the cross-slip plane remains active on the other side of the primary slip line. The primary slip line at C is an unusual observation since it was the only slip line formed in unidirectional tension which was seen to fade out. The line D is a scratch on the photographic plate and not a third slip system. The overall picture conveyed by these two micrographs of long range cross-slip is that the latter is a means of activating new primary slip planes. Similar observations have been made by Schwink and Neuhäuser (92) in irradiated Cu. In a movie sequence they observe cross-slip spreading out first from a primary slip cluster and then initiating new primary slip at ~5 microns from the original cluster.

An extreme example of short range cross-slip is shown
in fig. 4.15. Lines P and Q are connected by very faint cross-slip at various points along their length, and as one line broadens the other becomes thinner. Fig. 4.16 also shows short range cross-slip joining three primary slip lines, which is joined to the intersection of the black primary system and the white secondary system.

4.9 Distribution of slip line widths.

Crystal C 5 was pulled in five stages as indicated in table 4.1, and after each macroscopic strain (shown in col. 2) 12 micrographs were taken at random from within the slip clusters. The widths of the slip lines on the micrographs were measured and the frequency (f) of a given width (W) was plotted as f(W). The distribution curve obtained after test 5 is given in fig. 4.17. From the average width and density of the slip lines, the strain represented on each batch of the 12 micrographs was calculated (Col. 3, table 4.2). These strains bear little relationship to the macroscopic strain, since their value depends on the regions which were photographed, and any particular slip line width has a certain probability of occurrence, depending on which part of the cluster was photographed, hence a large error in the strain calculation could be obtained. For a good strain correlation many more plates should be taken, thus giving a more reliable average. If each distribution curve thus obtained is divided by the strain of col. 3 in table, 4.1, then the points fall on a 'universal' curve as shown in fig. 4.18 except for the tail of the curve obtained after tests 5. Furthermore if the log of f is plotted against the log of W for the tail of each distribution
curve, a straight line of slope $n$ is obtained. The value of $n$ for each curve is given in col. 4 table 4.3. The distribution expressed in the tail of this curve is of the form:

$$f = \frac{B}{\gamma^n}$$

where $B$ is a constant.

It is possible that one of the standard distribution equations (i.e., Maxwellian, Poisson, etc.) could be fitted to the complete curve, but this was not attempted but is left as a suggestion for further work. Since all the distribution curve peaks coincide when expressed as a universal curve, the latter expresses that the most probable slip line width depends linearly on the strain, which can be interpreted as meaning that the slip lines grow in tension with strain, a conclusion borne out by the findings of section 4.12.

Fourie and Wilsdorf (21) have carried out a detailed investigation of the $f$-$W$ curves for $\alpha$-brass over various strains and their histograms have roughly the same shape as those described here.

Crystal CA1 was pulled $0.067 \pm 0.001$ in tension and a neck 2 cm. long formed on the 7 cm. gauge length (crystal diameter $= 3.1 $mm). From the clusters in the necked region, 12 micrographs were taken at random, and the average slip line width calculated. The percentage of slipped gauge length was determined by measuring the widths of the slip clusters with an optical microscope. The strain calculated from the average slip line density and width and percentage of slipped gauge length was.
0.070 ± 0.007, in good agreement with the macroscopic strain. A histogram (compiled in the same way as for the distribution curves above) of slip line widths of this crystal is given in fig. 4.19. The dotted curve gives the continuous distribution of slip line widths for this crystal and it is similar to those obtained in figs. 4.17 and 4.18.

4.10 Further observations on slip lines using the electropolish technique.

The electropolish boundary technique, described in section 3.11, was carried out on crystals CA5 and CAT which had been incrementally deformed in tension. The results showed that:

a) slip occurred on previously inactive planes.

b) slip line growth took place.

c) some slip lines remained the same height.

Fig. 4.20 shows a typical section of the boundary and illustrates cases (a) (b) and (c) indicating them by N, I and O respectively. The lines marked N represent new slip lines since the slip lines are the same height in both regions. The lines marked I in the centre of the photograph represent incremental slip coming from the base of the step. This was found to be the case on the opposite side of the crystal; in crystal CA7 a boundary was polished on diametrically opposite parts of the crystal to verify that slip always came from the base of the steps. The lines marked O are ones which remained inactive throughout the incremental strain, since no incremental slip can be seen to come
For crystals which had received small incremental strains the new (N) and incremental (I) slip occurred predominantly at one stage of the cluster and on the 'downhill' side of it. (Throughout this work the word 'downhill' when referring to slip clusters or slip lines, will be taken to mean 'the base-of-the-step' side of the slip cluster or line). For larger incremental strains, slip clusters broadened from both sides, (see fig. 4.21)

4.11 The distribution of new and incremental slip lines.

After crystal CA7 had been prestrained in tension by 0.0481, it had a boundary polished along the entire gauge length. It was then given two total incremental strains, one of 0.0044 followed by one of 0.0155. After each incremental strain replicas were taken from along the boundary. An analysis of the number of incremental slip lines and new slip lines was carried out as laid out below. First, it is necessary to establish a notation for the following quantities:

Let \( N_0 \) be the number of lines per micron present after the prestrain.

Let \( N_I \) be the number of incremental lines per micron present after the incremental strain.

Let \( N_n \) be the number of new lines per micron present after the incremental strain.

The total number of lines formed during the incremental strain, in the repolished region, is \( N_I + N_n \) and the total in the other
Since the strain varies along the gauge length in a way described in section 4.3, the value of $N_0$ increases progressively from the lightly deformed region to the heavily deformed region. From the replicas each of which covered 2 mm of the boundary, $N_t$, $N_i$, and $N_n$ were counted, and the value of $N_0$ was calculated. Usually the complete replica was used in such counts and each value to the above quantities were averaged out over a 2 mm part of the gauge length boundary. By plotting $N_i/N_0$ and $N_n/N_0$ against $N_0$, the variation of incremental slip lines and new slip lines with local prestrain could be found, the results obtained after both incremental strains being shown in figs. 4.22 and 4.23.

If the average width of the slip lines is assumed constant over the gauge length then the value of $N_0$ is proportional to the strain. In fig 4.23 both the sets of points fit a straight line if the point marked A is ignored.

When $\log N_n/N_0$ is plotted against $N_0$ the graph in fig. 4.24 is obtained. The discontinuities both occur at about the same value of $N_0$ (2.3 lines per micron). This will be called the 'deformation transition point'. The slope of both the curves in fig. 4.24, below the deformation transition point is $-1.0.1$ which gives a hyperbolic relationship between $N_n/N_0$ and $N_0$ showing that $N_n$ is constant. Above the transition point, the slope is $-3.6.2$ for both graphs giving that $N_n = A N_0^{-2.6}$, $A$ being a constant which will be different for both graphs.
If the straight lines in fig. 4.23 are expressed as $N_i = f(N_0)$ then the functional relationships are:

$$N_i = 0.44N_0 - 0.103N_0^2 \quad \text{(for incremental strain $0.0155$)}$$

$$N_i = 0.20N_0 - 0.046N_0^2 \quad \text{(for incremental strain $0.0044$)}$$

where the numbers are taken from the slopes and intercepts of the straight line graphs and are subject to an error which is not greater than $\pm 0.004$ for the slope and $\pm 0.05$ for the intercept.

Differentiation of these equations gives maxima at:

$$N_o = 2.28 \quad \text{for the 0.0155 strain increment}$$

$$N_o = 2.18 \quad \text{for the 0.0044 strain increment}$$

The value of $N_i$ is zero when $N_o$ is zero and 4.3 lines per micron for both curves.

A schematic diagram of these variations is shown in fig. 4.25.

The value of $N_i$ is independent of $N_o$ up to the transition point after which it falls off rapidly with $N_o$. The value of $N_i$ increases from zero to maximum at the transition point and then falls to zero again at the values quoted above. The transition point of $N_o = 2.3$ lines per micron coincides with about the centre of the transition region (as defined in section 4.5) but the exact position has not been determined.
4.12 Discussion of results.

The progress of the deformation of CuAl can clearly be traced through the pattern of the results given in the last part of this chapter. As the crystal is subjected to higher and higher stresses the appearance and initial propagation of slip markings corresponds to a drop in the applied load. The yield drop corresponds to the formation of a neck and since this forms in a localized part of the gauge length the slip planes rotate (10° in Ca5) causing a reduction in the cross-sectional areas of the crystal, and a change in the resolved shear stress acting on the primary slip planes, the result being that work-hardening takes place. This last point can be more clearly understood if the post yield drop behaviour of the crystal is considered when the neck spreads along the gauge length causing lattice rotation and changing undeformed crystal into a deformed state at constant load. Appendix A gives a treatment of the resolved shear stress in the neck as a function of the crystal lattice rotation, the result being expressed in eqn. A7 as:

\[ \sigma_d = \frac{\cos \chi_a}{\cos \chi'_a} \sigma_u. \]

Where \( \sigma_u \) is the applied resolved shear stress on a primary slip plane in the undeformed region, \( \sigma_d \) is the applied resolved shear stress on a primary slip plane in the heavily deformed region, \( \chi'_a \) is the angle between the primary slip system Burgers vector in the deformed crystal and the tensile axis and \( \chi_a \) is the corresponding angle in the undeformed crystal. The value of \( \sigma_d \) for crystal Ca5 is 1.16 \( \sigma_u \) showing that although the load is constant, hardening has taken place and must do so during.
the rotation of the crystal lattice. The degree of hardening must be the controlling factor in the amount of necking that takes place so that the crystal in slipping locally, hardens and stops slipping after which the neck propagates along the gauge length. If the crystal did not harden the neck would become increasingly thinner and local fracture would occur without any further deformation of the rest of the gauge length.

The slip markings themselves are clusters of individual slip lines which start off as fine or small clusters in the lightly deformed region and become broader as they go through the transition region into the heavily deformed region. The broadening occurs at the down-hill side of the cluster on each side of the crystal. This one sided cluster broadening is thought to be caused by one of the effects illustrated in fig. 4.26. Case (a) shows a single slip cluster in the gauge length (the proportions of this diagram are grossly exaggerated in the interests of clarity). Because of the cluster's presence, the undeformed gauge length, when unloaded, is no longer parallel to the tensile axis. Hence on re-applying a stress, to give an incremental strain, the gauge length in trying to become parallel to the axis, will experience a greater tensile force at the down-hill sides of the cluster (indicated on the diagram), than on the opposite side. Hence dislocation sources in the shaded regions will tend to activate more readily than in any other part of the crystal, thus causing the cluster to propagate in the way that is observed. The alternative explanation is shown in fig. 4.26b.
Here the effect of the cluster is to cause a local reduction in the cross-sectional area of the gauge length. This can be seen to give a higher stress concentration on the downhill side of the stop, as indicated by the stress vs. distance graph accompanying the figure. In compression, however, it should be noted that the fig. 4.26a model will not give slip cluster broadening at the downhill side of the cluster, whereas the model in fig. 4.26b predicts this. It will be seen in section 5.7 and fig. 5.14 that compressive slip also broadens clusters on the downhill side and thus the second explanation postulated is thought to be the correct one.

Figs. 4.5 and 4.13 indicate that short and long range cross-slip play a part in the broadening of a slip cluster. Dislocations emanate on the cross-slip plane from the clusters and either cross-slip back onto a new primary slip plane (such as at A in fig. 4.13) or may even activate new sources on the primary slip plane. Fig. 4.14 shows an example of how a new cluster might be initiated by long range cross-slip. It is clear in this diagram that any dislocations on the cross-slip plane have cross-slipped onto the new primary slip planes, or have been annihilated by dislocations of the opposite sign on the new primary slip planes. As the slip clusters broaden and approach each other, their stress fields are such as to activate intense cross-slip (figs. 4.5 - 4.7) which relieves the stress between the clusters.

The mechanism whereby cross-slip activates new lines is not clear. A Koehler source could be a means of starting slip on new planes, but would not give rise to such large slip steps
as are observed on the cross-slip systems. The height of these, often several hundreds of angstroms, suggests the continuous passage of dislocations along the cross-slip plane. A possible explanation for short range cross-slip could be that primary system dislocations are held up at a sessile barrier. Evidence of this is shown in figs. 4.11 and 4.16 where Lomer-Cottrell barriers could be formed by the interaction of dislocations of the primary and secondary systems. The dislocations would cross-slip until they are out of the influence of the stress-field of the barrier; they would then return to a new primary slip plane. The constriction of stacking faults which are necessary on the Seeger model for cross-slip would be effected at Stage I stresses by the large pile-ups which are known to occur in this type of material (33,30). This mechanism could not satisfactorily account for long range cross-slip since the distance traveled by dislocations on the cross-slip plane in this case are an order of magnitude higher than for short range cross-slip. It is possible that dislocation sources on the cross-slip plane are activated by the total stress field of a nearby slip cluster, and that dislocations thus generated travel to a cluster and annihilate the primary dislocations. The absence of any long-range cross-slip in the lightly deformed region, where the spacing of the slip clusters is wide, supports this model.

The cross-slip seen in the vicinity of etch-pits could arise from the local stress gradient introduced by the pit. The local stress would be concentrated around the base of the pit.
and inhomogeneous stresses thus caused could give rise to local movement of dislocations on the secondary slip systems, which in turn would form Lomer-Cottrell barriers with dislocations on the primary slip system. Cross-slip would then ensue as a means of stress release. Fig. 4.11 strongly supports this explanation. The etch pits themselves could arise from two possibilities:

a) Local impurities or Al concentrations could cause a preferential polish attack.

b) A high local dislocation density which may give rise to preferred polishing.

The second alternative is considered unlikely in view of the anneal undergone by the crystal before the electropolishing.

The results of section 4.9 show that the slip lines on the surface adequately account for the macroscopic strain, and that there is no need to invoke fine slip on other parts of the gauge length. The discrepancy of Koppenaal and Fine was due to their limit of resolution of 540Å (represented by the dashed line in fig. 4.17). Since they could not resolve lines of height below this limit it was calculated that they could only have measured ~50% of the deformation within the slip clusters.

Slip line growth takes place in CuAl as shown by the results of sections 4.9, 4.10 and 4.11. The nature of the growth can be made to show that the deformation of CuAl is by means of inhomogeneous slip on individual [111] planes and not by small amounts of slip on many closely spaced planes. The fact that growth always takes place at the base of the step
on both sides of the gauge length makes this evident. If the deformation was of the homogeneous type then some growth from near the top of the step would be expected, but is never observed. Fournie and Wilsdorf (21) have shown by five different methods that α-brass exhibits inhomogeneous deformation; this is shown very clearly by these experiments for CuAl.

The occurrence of a maximum in the density of incremental slip lines at the transition point is a curious occurrence, and shows that once the slip line density increases to a certain value, the growth and formation of new lines becomes rapidly suppressed. When the slip line density reaches higher values the slip line growth ceases altogether and the formation of new lines becomes negligible. This is to be expected since once the neck has formed the crystal into a work-hardened state the stress here is constant since the load is constant. The constant value of \( N \) below the transition point reflects a constant rate of growth of the slip clusters, which shows that little interaction occurs between them. When \( N \) is equal to the transition value the slip clusters are close enough together for their stress fields to interact and for their propagation to be slowed down by the proximity of each other. It is possible that the transition point marks the onset of the intense cluster connecting cross-slip that is observed (figs. 4.5 - 4.7). Thus the stresses between the clusters would be such as to damp out primary slip by the motion of secondary system dislocations and to cause cross-slip to be the mode of stress release. An alternative way of looking at the relationship between \( N_n \) and \( N_0 \).
above the transition point is that when the slip clusters approach close enough to one another the formation of new slip lines reduces significantly the amount of gauge length left for slip to occur on. This alone does not explain the observed variation of $N_n$ with $N_0$ since it demands that $N_n$ drops to zero at some value of $N_0$, which is not what the results show.

4.13 The deformation equivalence of α-brass and CuAl.

It is interesting to note that the results of this chapter indicate that α-brass and 12 at. pct. CuAl have qualitatively identical deformation characteristics. Slip on α-brass is inhomogeneous (21); cross-slip at etch pits is observed (18,91) cross-slip is observed during Stage I (21); deformation proceeds by means of broadening slip clusters (18); slip line widths have similar distributions (21); slip line growth occurs (21) and no fine slip occurs between the slip clusters (21).

4.14 Summary and Conclusions.

Barriers are formed by the interaction of dislocations of the primary and secondary systems during unidirectional deformation of CuAl. Cross-slip occurs and is probably the cause of cluster broadening until the gauge length is covered with slip. During this propagation many slip lines grow in height the density of these being a maximum in the transition region. The slip lines represent inhomogeneous deformation on individual $\{111\}$ planes. Work-hardening takes place in Stage I and this determines the extent of necking. The slip lines on the surface account for all the macroscopic strain.
<table>
<thead>
<tr>
<th>Test</th>
<th>Macroscopic Tensile Strain</th>
<th>Ave. Tensile Strain in Slipped Region</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0051</td>
<td>0.31</td>
<td>0.41 ± 0.3</td>
</tr>
<tr>
<td>2</td>
<td>0.019</td>
<td>0.36</td>
<td>0.41 ± 0.3</td>
</tr>
<tr>
<td>3</td>
<td>0.045</td>
<td>0.34</td>
<td>0.46 ± 0.3</td>
</tr>
<tr>
<td>4</td>
<td>0.068</td>
<td>0.50</td>
<td>0.27 ± 0.3</td>
</tr>
<tr>
<td>5</td>
<td>0.087</td>
<td>0.27</td>
<td>0.39 ± 0.3</td>
</tr>
</tbody>
</table>
Fig. 5.1. Diagrammatic Load-Strain Curve for Crystal CA 5.
FIG. 4.2.
FIG 4.3 Lightly deformed region Crystal CA 5 x192

FIG 4.4 Transition region Crystal CA 6 x300
FIG 4.7  Heavily deformed region  Crystal CA 5  x230

FIG 4.8  Cross-slip in the repolished region  Crystal CA 6  x675
FIG 4.9  Cross-slip near polishing pits  Crystal CA 6
Repolished region  x300

FIG 4.10  Cross-slip near polishing pits  Crystal CA 6
Repolished region  x300
FIG 4.11 Electron micrograph of cross-slip and secondary slip near to a small polishing pit. Crystal CA 9 x30,000

FIG 4.12 Electron micrograph of cross-slip. Crystal CA7 x33,000
FIG 4.13  New primary slip being initiated from a small cross-slip cluster.  Crystal CA7 x8,800
FIG 4.15  Short range cross-slip  Crystal CA 5  x48,200

FIG 4.16  Secondary slip near to cross-slip. Crystal CA 9  x27,000
FIG. 4.17. Slip Line Width Distribution Curve from Expt. CA5, test 4
FIG. 4.18. "Universal" distribution

Experiment CA5

- Test 1 0.31 Str
- Test 2 0.34 Str
- Test 3 0.36 Str
- Test 4 0.37 Str
- Test 5 0.51 Str
FIG 4.20  New and incremental slip lines at the electropolish boundary. Crystal CA 7  x 15,000
FIG. 4.22.

+ 0.0044 total incr. strain.

O 0.0155
FIG. 4.23. + 0.0044 total incr. strain

- o.0155 " " " "

\[ \frac{N_i}{N_0} \]

Graph showing data points and lines with labels.
+ 0.004 total incremental strain

○ 0.0155 total incremental strain

FIG. 4.24
FIG. 4.25. Diagrammatic Variation of $N_n$ and $N_i$ with $N_o$. 

Transition Point

$N_o$

$N_i$

$N_n$
FIG. 4.26.

Downhill Side

Downhill Side
CHAPTER V

The mechanical behaviour of CuAl under reversed stresses.

5.1 Introduction.

No replica studies of slip lines formed on the surface of CuAl deformed under reversed stresses have been reported in the literature; only the optical micrographs of Avery and Backofen (53) have been published. This chapter will deal with the Bauschinger effect and its relation to slip line observations. A short account of some slip line observations over more than one cycle will then be given.

Since CuAl has a strain gradient along the gauge length, it was only necessary to make observations on two crystals (CA6 and CA9) using the technique described in section 3.11. Some of the other CuAl crystals were compressed after the tension experiments had been completed, to obtain values of the Bauschinger effect, and their results are presented in section 5.2. At larger reversed stresses some important preliminary observations were made and these will be presented in section 5.3 before the detailed results.

5.2 The Bauschinger effect in CuAl.

Bauschinger effect results taken from various crystals are shown in fig. 5.1. The ratio $\frac{\sigma_1}{\sigma_0} = 0.88$ was chosen so that the effect would be large enough to measure with reasonable accuracy. The points from crystals CA4, CA7 and CA9 lie on a straight line, but those of CA6 and CA5 lie well off this line. The ageing time at room temperature or the time which elapsed between the prestrain and the compression is shown also on the figure. There are three points which belong to CA7, the first
(i) being taken after an ageing time of five days. After the Bauschinger effect measurement had been taken the crystal was immediately reformed in tension and a sharp yield point was observed. It was then recompressed for a second Bauschinger strain measurement. The ageing time here was only five minutes, and point CAT (ii) is the result. After a further incremental pull in tension, which showed a small yield point, the pt. (iii) was obtained and the ageing time was in this case two hours.

5.3 Preliminary experiments which established the occurrence of reverse slip.

Crystal CA4 was pulled 0.008 in tension and, after repolishing a boundary, was compressed to a strain of 0.012. Replicas taken from the boundary on the gauge length revealed that new lines had formed in compression in the vicinity of the tensile slip clusters. In addition some compressive slip lines joined onto tensile lines as shown in fig 5.2. The white compressive line joins on at the base of the tensile slip step, just as the incremental lines (described in section 3.11) did. Hence reverse slip had taken place since no corresponding white slip can be seen beside the tensile slip line. In two clusters it was found that 70% of the tensile slip lines had partially reversed, but no total reversal had taken place. This crystal was then reformed to a strain of 0.015 in tension and a fresh boundary was polished over the original one to wipe off the compressive and tensile lines formed in 1st and 3rd half cyles
respectively. The crystal was compressed to 0.034 and fig. 5.3 is a typical region of the boundary. It can be seen that three tensile slip lines have totally reversed since no tensile slip is left in the old region, but compressive lines appear in the repolished region at the polishing traces of the tensile slip lines. Fig. 5.4 shows a singularly good example of this effect. No ever-reversal was ever seen. In fig. 5.3 the line marked A-A leaves a very faint trace in the unrepolished region (This can be seen best by looking at the photograph at grazing incidence and parallel to tensile slip lines). It is possible that this is due to a slight component of slip left in this region due to the possibility that exact total reversal did not take place. It could be argued however, that this is due to compressive slip on a slip plane near to the original tensile slip line thus forming an intrusion, which would give an extrusion on the replica. This would give a broad black and white line close together, but this is not observed. It could be objected that the intrusion in the metal is so narrow that the replicating formvar could not penetrate. To do this the slip plane separation would have to be less than ~30Å which would mean that compressive slip would occur on a slip plane which was only 12 atomic distances away. This does not appear likely in view of Strutt's observation in thin foils (58) which show dislocations lying on slip planes which are well separated by at least ~100Å after 50 cycles of deformation. This same argument applies to the case of an extrusion on the metal surface. The most reasonable explanation is that reverse slip does take place and that the
trace can be due to very slight over- or under-reversal (in the case of line A a slight over-reversal is appropriate.) Alternatively some surface film (e.g. oxide, sulphide etc.) which had formed on the face of the tensile step was 'scraped' off, as the slip line shrank, and remained on the crystal surface.

5.4 Cross-slip and reverse slip.

Figs 5.5 and 5.6 are micrographs taken from the unrepolished region on CA4 after the second cycle of deformation. They show examples of how cross-slip could either cause or stop reverse slip. Consider fig 5.6 which is the clearer example of the two. Since it is unlikely that the black tensile slip would stop in the middle of the gauge length, then either:

(a) dislocations travelling along line $P_1$ under compression to the left, say, (causing the tensile line $P_1$ to totally reverse) were diverted by a barrier and cross-sliped onto the new plane causing a compressive slip line;

or (b) dislocations travelling along $P_2$ (and of the opposite sign as those which caused $P_1$) cross-sliped onto $P_1$ and caused it to totally reverse slip.

It cannot be decided here which took place, but reverse slip is certain to have occurred from either alternative, especially in view of the fact that both slip lines are about the same height. Many examples of this reaction were found and it is strikingly convincing evidence for reverse slip.
5.5 The variation of reverse slip with prestrain.

Crystal CaS was pulled in tension to form a neck, and then repolished to form a boundary along the gauge length. The crystal was compressed to \( \frac{\sigma_0}{E} = 0.33 \) after ageing at room temperature for 2 hours. Replicas taken from the boundary then showed fine compressive slip lines joined to some of the tensile ones at the boundary. No new compressive slip was visible in the un-repolished region. The number, \( N_c \), of compressive lines in the repolished region and the number, \( N_0 \), of tensile lines in the unrepolished region were counted over 2\( \pi \)m portions of the boundary and their average values determined. Since no total reversal could be seen over the boundary, \( N_0 \), was then taken as an accurate value of the initial tensile slip line density i.e. before compression. \( \frac{N_c}{N_0} \) was plotted vs. \( N_0 \), and Fig. 5.7 shows the result. The crystal was then repulled in tension up to 0.88 of the previous tensile unloading stress and replicas were again taken from the boundary. The same counting procedure as before yielded the dotted line in Fig. 5.7. The relationship between \( \frac{N_c}{N_0} \) and \( N_0 \) for both curves is of a first degree equation form. The second count after the pull in tension gives a straight line of the same slope as the full one but displaced downwards so as to give a different intercept. The crystal exhibited a Bauschinger strain of 0.0030 during this third half cycle. The Bauschinger strain during the first compressive half cycle was 0.0072. The ratio of these was 0.42. After the third half cycle many fine black lines could be seen in the region which had previously only contained white compressive
slip lines (see fig. 5.8). These had the same appearance as the white lines being small in height, short in comparison with the original tensile lines (ie. \( N_0 \)), and sometimes being connected with cross-slip. The ratio of black to white lines in this region was 0.26.

The gauge length of CA6 was then completely repolished to remove all the slip lines, and was then cycled at constant stress amplitude of \( \sigma = 0.30 \sigma_0 \). Replicas taken from the surface showed fine black and white slip (see fig. 5.9). There were no long, coarse lines on the gauge length as found in the slip clusters formed during the tensile prestrain half cycle.

5.6 The variation of reverse slip with the Bauschinger effect.

Crystal CA9 was prestrained 0.03 in tension, and after repoling the boundary, it was compressed to increasingly higher stresses within the Bauschinger strain region (values of stress-strain measurements are given in table 5.2). After each compression the Bauschinger strain was measured and the number of compressive slip lines \( (N_c) \) in the same 2mm. region of the re-electropolished part of the specimen was counted. \( N_c \) was plotted against strain, \( \varepsilon_B \) (fig. 5.10) and Bauschinger stress fraction \( \frac{\sigma_B}{\sigma_0} \) (fig. 5.11). The slip line length, \( L \), was measured for three stress values and was found to increase as follows.

\[
\begin{align*}
\frac{\sigma_B}{\sigma_0} &: 0.625 \quad 0.75 \quad 0.84 \\
L &: 4.8 \mu \quad 6.0 \mu \quad 9.0 \mu
\end{align*}
\]
The values of $L$ were obtained by counting the number of slip line ends in a given field of view and by using the formula

$$L = 2 \frac{N_c}{D}$$

where $D$ is the average density of slip line ends and includes those slip lines which end in cross-slip. $N_c$ has already been defined, and as pointed out in appendix C the values of $L$ given by this formula are probably only qualitatively correct. The most important feature of the above figures is that $L$ increases with stress. Also the depth of the slip lines increases with stress as is shown in figs. 5.12 and 5.13, which show typical slip lines from the repolished area after $\frac{\sigma}{\sigma_0}=0.75$ and $\frac{\sigma}{\sigma_0}=0.92$ respectively. Fig 5.12 also shows cross-slip occurring, which was quite common at $\frac{\sigma}{\sigma_0}=0.625$, the lowest stress level at which slip lines were observed on the surface. Many line endings did not have any visible cross-slip, but just appeared to fade out.

5.7 Optical microscope observations.

The deformation history of CA9 is given in table 5.1; the note in the second row refers to the sub-table 5.2, which contains the information during the progressive Bauschinger compression tests numbered 2 - 7. After the compression indicated in row 3 a yield point appeared with dimensions given in the 4th and 5th columns of table 5.1. Fig. 5.14 shows the appearance of the slip clusters under the optical microscope after compression beyond the yield point. The top photograph shows two clusters in the unrepolished region and the lower one shows the same two clusters in the repolished region. The
diffuse slip in the clusters of the lower photograph is the reverse slip which took place in the old tensile slip clusters during compression. On either side of this diffuse slip there is coarse slip formed during yield in compression. This is most abundant on the downhill side of the cluster; the same result as was found during tensile increments of strain.

5.8 Yield point observations in CuAl during stress-reversals.

After the results of the last section were obtained a brief investigation of the appearance of yield points under reversed stresses was carried out on crystal CA9. Table 5.1 gives all the data (h and W have already been defined in Section 4.2). The second column refers to the results of table 5.2; the nine day period during which the results in section 5.6 were obtained. A few features are apparent. Firstly, immediate restressing in the same direction as the previous stress caused no yield drop (rows 10 and 12). A 3 hr lapse before restressing in the same direction as the previous stress was sufficient to produce a small yield drop (row 9). After a compressive prestrain of 0.015 (row 13) immediate restressing in tension produced a small yield drop (row 14), but after a small prestress in tension (row 14) no compressive yield drop appears (row 15), although a yield drop does appear in compression after a large tensile prestrain (rows 11, 12 and 13).
- 111 -

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of test</th>
<th>Strain</th>
<th>Ageing time since previous def'm.</th>
<th>$h$ (kgf/cm²)</th>
<th>$W$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tens</td>
<td>0.030</td>
<td>------</td>
<td>0.160</td>
<td>~0.03</td>
</tr>
<tr>
<td>2 - 7</td>
<td>Bauschinger strain measurements; see table 5.2.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Comp.</td>
<td>0.014</td>
<td>9 days</td>
<td>0.035</td>
<td>0.003</td>
</tr>
<tr>
<td>9</td>
<td>Comp.</td>
<td>0.004</td>
<td>3 hrs</td>
<td>0.019</td>
<td>0.003</td>
</tr>
<tr>
<td>10</td>
<td>Comp.</td>
<td>0.004</td>
<td>2 mins</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>11</td>
<td>Tens</td>
<td>0.020</td>
<td>5 mins</td>
<td>0.012</td>
<td>0.012</td>
</tr>
<tr>
<td>12</td>
<td>Tens</td>
<td>0.004</td>
<td>1 min</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>13</td>
<td>Comp.</td>
<td>0.015</td>
<td>5 mins</td>
<td>0.003</td>
<td>0.003</td>
</tr>
<tr>
<td>14</td>
<td>Tens</td>
<td>0.005</td>
<td>5 mins</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>15</td>
<td>Comp.</td>
<td>0.024</td>
<td>3 mins</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>16</td>
<td>Tens</td>
<td>0.059</td>
<td>3 mins</td>
<td>0.013</td>
<td>0.015</td>
</tr>
</tbody>
</table>

**TABLE 5.1** Yield drop data for Experiment CA 9.

<table>
<thead>
<tr>
<th>Compression test No.</th>
<th>$\sigma_B/\sigma_0$</th>
<th>$\varepsilon_B$</th>
<th>Ageing time since previous deformation.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.490</td>
<td>0.0000</td>
<td>3 hours</td>
</tr>
<tr>
<td>3</td>
<td>0.625</td>
<td>0.0007</td>
<td>1 minute</td>
</tr>
<tr>
<td>4</td>
<td>0.750</td>
<td>0.0019</td>
<td>~6 days</td>
</tr>
<tr>
<td>5</td>
<td>0.840</td>
<td>0.0041</td>
<td>1 day</td>
</tr>
<tr>
<td>6</td>
<td>0.920</td>
<td>0.0059</td>
<td>1 day</td>
</tr>
<tr>
<td>7</td>
<td>0.930</td>
<td>0.0081</td>
<td>1 day</td>
</tr>
</tbody>
</table>

**TABLE 5.2** Bauschinger strain data for Experiment CA 9. Tests took place during the interval indicated in the 2nd row of table 5.1.
\[
\frac{\sigma_B}{\sigma_0} = 0.88
\]

**Ageing Time**

- CA4: 2 hrs.
- CA6: 2 hrs.
- CA7: i) 5 days, ii) 10 mins., iii) 2 hrs.
- CA5: 14 days
- CA9: 6 days

**Fig. 5.1**

*Bauschinger strain*

*Tensile prestrain*
FIG 5.2
Partial reverse slip
Crystal CA4: After first cycle of deformation.

x30,000

FIG 5.3 Total reverse slip. Crystal CA4: After second cycle of deformation.

x 13,500
FIG 5.4 Total reverse slip in crystal CA 4 after second cycle of deformation.  x 30,000
FIG 5.5
Crystal Ca 4 deformed through 2 cycles.

x 24,000

FIG 5.6
Crystal Ca 4 deformed through 2 cycles.

x 36,000
FIG. 5.7
FIG 5.8 Crystal CA 6 Repolished region. Pulled in tension after a Bauschinger compression. x 21,000

FIG 5.9 Crystal CA 6, repolished region. Deformed through 5 cycles of const. stress amp. 0.33 o x 30,000
FIG. 5.10. $N_0 = 2.9$
FIG. 5.11 \( N_0 = 2.9 \frac{\sigma \omega}{\mu} \)
FIG 5.12 Slip during the Beuschinger compression region $\sigma_y/\sigma_0 = 0.75$. Crystal CA 9 x 24,000

FIG 5.13 As above $\sigma_y/\sigma_0 = 0.92$ x 24,000
FIG 5.14  Top:  slip clusters in unpolished region
Bottom:  same clusters in repolished region
x 200
CHAPTER VI

The results obtained on copper single crystals deformed in unidirectional tension and under reversed stresses.

PART I Unidirectional tension results.

6.1 Introduction.

In chapter III the nature of the measurements on copper deformed under reversed stresses has been considered; since this involves the counting of tensile and compressive slip lines, it was necessary to start the investigation by determining how \( n_t \) varied with prestrain, in order to find out how the deformation conditions affect the slip line densities. From the values of \( n_t \) vs. tensile prestrain given in this section, specific experiments were planned for the reversed stress measurements given in the second part of this chapter.

6.2 Flow stresses of copper crystals.

The results of other workers (13, 47, 31, 35) have shown that the critical resolved shear stress (c.r.s.s.) of annealed copper single crystals varies between 35 - 100 \( \mu \)N/\( \mu \)m\(^2\); the variation being due to crystal impurity, orientation and annealing conditions. In the experiments described here, 17 crystals were pulled, either to obtain c.r.s.s. values or simply to act as a prestrain prior to reversed stress measurements. The values of the c.r.s.s. for the crystals are given in table 2.2, together with relevant data on annealing and length of stage I. All the crystals were of single glide orientations as indicated in the penultimate column (see also Fig. 2.3). Initially the standard treatment was to anneal the crystals for \(~16\) hrs. at \~1000\(^\circ\)C (Table 2.2 for details), but this did not give
reproducible c.r.s.s. values. Group F8, F8'', and F9 exhibited a high c.r.s.s. and showed excessive, fine pitting as a result of electropolishing which other crystals did not. Little if any easy glide was detected in this group, and since this was an important parameter in these experiments it was necessary to locate the trouble. Both the annealing procedure and impurity content were suspected.

6.3 Annealing Procedure.

Livingston (78) has reported that cyclic annealing is more effective than an isothermal anneal in lowering the dislocation density below that of the as-grown crystal. A cyclic annealing procedure similar to that of Livingston (13) was carried out on some of the crystals and the results shown in table 2.2 indicate a lowering of the c.r.s.s. values with this kind of treatment. F15 was the only exception, but since this crystal showed double slip after only 1% resolved shear strain, its orientation is suspected of having drifted onto the [01̅] $\langle 11̅0 \rangle$ axis. This would account for its anomalous behaviour.

6.4 Effect of impurities on the c.r.s.s. values.

Although the as-received material had a specified impurity content of 8 p.p.m., the as-grown crystals F8, F8'', F9 were suspected of a higher impurity content since their c.r.s.s. values were high despite a long anneal. F9, and B1 (which had more normal behaviour) were sent to Johnson, Matthey & Co. Ltd. for analysis, and table 2.4 shows the results, and comparison with table 2.1b shows that both crystals contain more impurities than the as-received material. Furthermore the impurity content of F9 is about twice that of B1.
The cause of the impurities was traced to contaminated furnace tube, but the crystals used for the results in this section are known not to be affected by this. These crystals have a maximum impurity content of ~25 p.p.m.

6.5 Slip line results from unidirectional tension experiments.

Crystals EG1 and EG2 were pulled in tensile strain increments. Their resolved shear stress–glide strain curves are shown in Fig. 6.1. The resolved shear strain was calculated from the tensile strain using the formula given in Schmidt and Boas (79). After each strain, replicas were taken from the same region near the centre of the gauge length, and were shadowed in such a way as to give positive contrast to the slip lines. Slip line densities were counted from randomly selected regions of the replicas, and the variation of slip line density (in lines per micron) vs. glide strain, is shown in Fig. 6.2 for these and other crystals from which tensile prestrain slip line densities were measured. On crystal F11, three replicas were taken from different regions of the gauge length and the slip line densities agreed to within ±10%. The error indicated is the R.M.S. deviation of the counts taken for each point. Each point is taken from about 30 screen counts. The R.M.S. deviation gives an estimate of the uniformity of the slip line density. Crystal EG2 was slightly bent before straining, the amount of surface glide strain being estimated at 0.002 and could account for the higher flow stress, larger slip line density at high strains, and shorter easy glide found for this crystal compared with EG1, which had the same annealing time. For the sake of comparison, the values of $n_c^b$ for the two compressive strains of F11 have been plotted on this graph.
They will be discussed in Chapter VIII, part 2. It can be seen from these graphs that up to glide strains of \( \sim 0.025 \) the slip line density is independent of the crystal used. For this reason the reverse stressing experiments of 1 cycle were carried out at prestrains which did not exceed this value.

The appearance of the slip lines varied with strain. At strains \( <0.005 \) many slip lines were fragmented and short (Fig. 6.4), whilst at higher strains they were generally long and more well-defined. At low strains the slip line density was non-uniform; groups of lines appeared on the gauge length, and the regions between these groups did not, to within the resolution of the electron microscope technique, contain any slip lines.

6.6 Discussion.

The results show that the c.r.s.s. of copper crystals is dependent on the impurity content and the annealing procedure. Crystals containing Sulphur and Nickel impurities exhibited high flow stresses \(-250 \text{ gN/cm}^2\). Honeycombe (14) has reviewed the effects of impurities on the c.r.s.s. of single crystals and the results presented here agree with the generally observed trend.

The annealing procedure has a definite effect on the c.r.s.s. of copper. Crystals 01 and 02, which had the longest cyclic anneal (Table 2.2), had the lowest flow stresses, whereas crystals like EG2, F1 and F2, which were isothermally annealed, had higher flow stresses; F11 and F12 which were cyclically annealed for a shorter time than 01 and 02 had intermediate flow stresses. Easy glide can be seen to be dependent on flow stress. In general, it can be seen from Table 3.1 that the higher the flow stress, the shorter the easy glide region.
The variation of slip line density with strain follows a similar variation to that of slip bands in Al vs. strain, increasing rapidly at first and then becoming proportional to strain. Brown (30) shows graphs of this variation, which are originally due to Yamaguchi (31), but whereas Yamaguchi's results give a linear variation of slip band density with shear stress, no such variation can be found for elementary structure in copper. Seeger et al. find that the slip line density on copper remains constant during Stage 1. However, this is only true for strains larger than about 0.02 (tensile strain). No attempt has been made to fit an equation to the observed variation of slip line density with strain in copper, as Brown (30) has done for aluminium, since this was not the purpose of the investigation. It is left as a suggestion for further work.

The observed slip line width from replicas is about 120\(\AA\), but from calculations such as that of section 3.8 the real width of the majority of the slip lines is about 640\(\AA\). The observation on crystal F11, that the slip line density is uniform along the gauge length, excludes the possibility of the freak observation that all the slip had occurred in the observation area. The fragmented appearance of slip lines at low strains is probably a real effect, since the slip line resolution is good. The short slip line length could be attributed to the cross-slip and annihilation of screw dislocations, which is reasonable in view of the results of thin foil experiments (7, 8, 9).
Observations of slip lines on copper deformed under reversed stresses.

The method described in section 3.9 was used to determine the values of $n^t_1$, $n^c_1$, $n^c_1$ and $n^b_c$ after stress reversals in Stage I, where the dislocation arrangements are relatively simple, and consistent values of $n^t_1$ were obtained during the prestrain. The three crystals examined were F11, 01 and 02, and their resolved shear stress-cumulative glide strain curves are shown in Fig. 6.3. The tensile stress-strain curve for Crystal F7 is also given (dotted curve) as a comparison, and it can be seen that hardening is lower for the cyclically deformed crystals.

F11 was reverse stressed after a glide strain of 0.606 corresponding to about the middle of stage I. The first compressive strain studied was -0.006, after which the crystal was further compressed to a strain of -0.012. After each strain several replicas were taken from the observation regions a and b, and shadowed at $\sim 12^\circ$ so as to give positive or negative contrast to the tensile slip lines. The results are shown in table 6.3. A notation is used in what follows to denote the type of test carried out. $T_n$ represents the $n$th tensile test and $C_n$ is the $n$th compressive test irrespective of the order in which the tests were carried out. The end points of these tests are indicated in Fig. 6.3 to show where replicas were taken during the straining. The strain corresponding to $C_n$ and $T_n$ is given with the test notation in table 6.3a. The magnification quoted in the last column is the magnification of the image on the electron microscope screen. All the slip lines were counted on the screen since it was found to give more consistent results which could
be obtained faster. The values of slip line density shown in table 8.1a give the average of between 20-40 slip line counts taken at random from the replicas. The (±) or (−) sign after the slip line density value indicates positive or negative contrast of the slip lines whose density value it follows.

In experiment 01 the crystal was pre-strained in tension by 0.024±0.001 and the value of \( n^b \) was determined. It was then compressed, by a Bauschinger strain of 0.003±0.001, to a value of \( \varepsilon_B/\varepsilon_0 = 0.33 \). Two replicas of slip lines shadowed with opposite contrasts were made and values of \( n^a, n^b, n^c \) were determined. A further compression to a total compressive strain of 0.025±0.001 was carried out and again two oppositely shadowed replicas were made. The results of the slip line counts are given in table 6.2a. Experiment 02 was performed without repolishing the area b on the gauge length, to see if the slip line values of \( n^b, n^a, n^c \) were consistent; no value of \( n^b \) was therefore obtained. The straining part of the experiment was performed in a few hours, to prevent any serious oxide formation on the surface, replicas being taken after each strain and shadowed later. In the previous two experiments, the crystals had undergone the sequence of: deformation, replication, determination of slip line densities, redeformation, replication, etc. The time between successive deformations was one or two days, and although the Formvar covering was kept on the crystal surface at all times, except during the straining and replication processes, there was uncertainty as to the amount of oxide that may have formed during these intervals. For this reason 02 was performed quickly, and if the slip line counts had the same values as in 01, then clearly the oxide formed
during crystal storage would be negligible. The crystal was subsequently cycled as indicated in table 6.3a and Fig. 6.3, and at the end of each half-cycle the ratio of \( \frac{n_c}{n_t} \) was determined to obtain further information on slip line behaviour after more than one cycle. The values of the slip line densities are given in table 6.3b and the ratios \( \frac{n_c}{n_t} \) are given in table 3.3c.

### 6.8 Discussion of slip line density values.

Consider now the slip line density values in table 6.3a. The value of \( n_t = 3.5 \) line per micron, is taken as it stands, since it agrees well with the graphs in Fig. 6.2. If the values of the slip line densities after test C are studied, it can be seen that in general the slip lines of either type, which are shadowed with positive contrast, have higher densities than when shadowed with negative contrast. With respect to observation magnification, slip line counts taken at 10,000x had smaller values than those measured at 20,000x. For these reasons, only the line counts at 20,000x positive contrast lines are taken into account during the averaging of the slip line density values, since this will then be consistent with the conditions under which \( n_t \) was measured. The finally accepted values are given in table 6.3b.

Similar arguments are applied to the results of D1. The value of \( n_t \) ignores the negative contrast value since it is also taken at a magnification of 10,000x, and is probably low. However, the values for test E2 indicate that both types of contrast give reliable results. This is probably because, in this experiment, slip step heights were larger than those of F11 and the visibility of lines was not critically dependent on the type of contrast used. For E2, then, averages of all
values were taken. The magnification change to 20,000x was checked by taking slip line counts at both 18,000x and 20,000x, no difference in slip line numbers could be detected; again this is probably due to the larger and therefore more visible slip steps. The value of \( n_c \) after test \( C_2 \) has a large error since the few lines which were seen in this region were non-uniformly distributed. Table 6.4b summarizes the finally accepted values of slip line densities for crystal 01.

In experiment 02, the same reasoning applies as to 01. The high value of \( n_t \) after \( T_3 \) is probably due to the fact that the strain in \( T_3 \) was slightly larger than that of the other tests (see table 6.3a). From test \( T_2 \) it can be seen that the type of contrast on a line was unimportant. Electron micrographs of slip lines taken after each test are shown in Figs. 6.4-6.8. The 'spottiness' is thought to be a slight surface oxidation but does not seem to affect the slip lines. It can be seen by comparison of table 6.2b (last row) with table 6.3b test \( C_5 \) that the slip line values agree quite well, and the two types of experimental procedure do not lead to very different results.
<table>
<thead>
<tr>
<th>Test</th>
<th>$n_t^l$</th>
<th>$n_c$</th>
<th>$n_t^a$</th>
<th>$n_c$</th>
<th>Magn.</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_1$</td>
<td>3.5±0.6(+)</td>
<td></td>
<td></td>
<td></td>
<td>20,000</td>
</tr>
<tr>
<td>0.003*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$C_1$</td>
<td></td>
<td>4.6±0.7(+)</td>
<td>3.4±0.7(+)</td>
<td>3.5±0.9(+)</td>
<td>20,000</td>
</tr>
<tr>
<td>0.003*</td>
<td></td>
<td>2.3±0.8(+)</td>
<td>3.4±0.7(+)</td>
<td>2.5±0.5(+)</td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3.5±1.0(+)</td>
<td>3.2±0.6(+)</td>
<td>2.1±0.7(-)</td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.3±1.3(-)</td>
<td>3.2±0.6(-)</td>
<td>2.1±0.7(-)</td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.4±1.2(-)</td>
<td></td>
<td></td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.7±1.3(+)</td>
<td></td>
<td></td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2.8±1.2(+)</td>
<td>2.1±0.5(+)</td>
<td>10,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2.5±0.6(-)</td>
<td></td>
<td>10,000</td>
</tr>
<tr>
<td>$C_2$</td>
<td></td>
<td>6.3±1.7(+)</td>
<td>3.3±0.8(+)</td>
<td>5.5±1.6(+)</td>
<td>20,000</td>
</tr>
<tr>
<td>0.003*</td>
<td></td>
<td>10.0±0.6(+)</td>
<td></td>
<td></td>
<td>30,000</td>
</tr>
</tbody>
</table>

*Magnitude of the tensile strain during the deformation interval. All values are accurate to ±0.0007.

**TABLE 6.1a: Slip line density values for crystal F 11**
TABLE 6.1b: Average values of slip line densities used in experiment on crystal F 11
<table>
<thead>
<tr>
<th>Test</th>
<th>( n^b )</th>
<th>( n^c )</th>
<th>( n^a )</th>
<th>( n^g )</th>
<th>magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>3.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16,000</td>
</tr>
<tr>
<td></td>
<td>( \pm 1.3(+) )</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.02</td>
<td>7.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10,000</td>
</tr>
<tr>
<td>( \pm 0.001 )</td>
<td>( \pm 1.4(-) )</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>8.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16,000</td>
</tr>
<tr>
<td></td>
<td>( \pm 1.5(+) )</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>7.6</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16,000</td>
</tr>
<tr>
<td></td>
<td>( \pm 1.4(+) )</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C1</td>
<td>-</td>
<td>0.8</td>
<td>6.9</td>
<td>0.0(-)</td>
<td>16,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( \pm 1.0(+) )</td>
<td>( \pm 2.4(-) )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.003</td>
<td>-</td>
<td>-</td>
<td>8.0</td>
<td>-</td>
<td>16,000</td>
</tr>
<tr>
<td>( \pm 0.001 )</td>
<td></td>
<td></td>
<td>( \pm 1.2(+) )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C2</td>
<td>-</td>
<td>10.8</td>
<td>7.6</td>
<td>8.0</td>
<td>20,000</td>
</tr>
<tr>
<td>0.025</td>
<td>( \pm 2.9(-) )</td>
<td>( \pm 1.5(+) )</td>
<td>( \pm 2.5(-) )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \pm 0.001 )</td>
<td>-</td>
<td>10.2</td>
<td>( 7.4 )</td>
<td>( 6.2 )</td>
<td>20,000</td>
</tr>
<tr>
<td></td>
<td>( \pm 3.0(+) )</td>
<td>( \pm 2.1(-) )</td>
<td>( \approx 2.4(+) )</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TABLE 6.2a: Slip line density values for crystal 01**
**TABLE 6.2b:** Final slip line density values for crystal 01

<table>
<thead>
<tr>
<th>Test</th>
<th>$n_t^i$</th>
<th>$n_c^b$</th>
<th>$n_t^a$</th>
<th>$n_c^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_1$</td>
<td>8.0±1.4</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>$C_1$</td>
<td>---</td>
<td>0.8±1.0</td>
<td>7.5±1.2</td>
<td>0.000</td>
</tr>
<tr>
<td>$C_2$</td>
<td>---</td>
<td>10.5±3.0</td>
<td>7.5±1.8</td>
<td>7.1±2.5</td>
</tr>
</tbody>
</table>
### TABLE 6.3a: Strain measurements in tension and compression for crystal 02

<table>
<thead>
<tr>
<th>Test</th>
<th>$n_t^1$</th>
<th>$n_t^a$</th>
<th>$n_c^a$</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_1$</td>
<td>7.8±1.5(+)</td>
<td>-----</td>
<td>-----</td>
<td>16,000</td>
</tr>
<tr>
<td>$C_1$</td>
<td>-----</td>
<td>8.5±2.4(+)</td>
<td>6.0±2.1(+)</td>
<td>20,000</td>
</tr>
<tr>
<td>$T_2$</td>
<td>-----</td>
<td>6.1±1.9(+)</td>
<td>3.0±1.7(+)</td>
<td>16,000</td>
</tr>
<tr>
<td>$C_2$</td>
<td>-----</td>
<td>4.7±2.3(+)</td>
<td>5.6±2.5(+)</td>
<td>16,000</td>
</tr>
<tr>
<td>$T_3$</td>
<td>-----</td>
<td>9.0±2.1(+)</td>
<td>3.0±1.9(+)</td>
<td>16,000</td>
</tr>
</tbody>
</table>

*Not shown in fig. 6.3*

### TABLE 6.3b: Slip line densities on crystal 02

<table>
<thead>
<tr>
<th>Test</th>
<th>$n_c^a/n_t^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_1$</td>
<td>-----</td>
</tr>
<tr>
<td>$C_1$</td>
<td>0.69</td>
</tr>
<tr>
<td>$T_2$</td>
<td>0.52</td>
</tr>
<tr>
<td>$C_2$</td>
<td>0.83</td>
</tr>
<tr>
<td>$T_3$</td>
<td>0.33</td>
</tr>
</tbody>
</table>

### TABLE 6.3c: Ratio of $n_c^a/n_t^a$ for crystal 02
FIG. 6.1.

Tensile Stress-Strain Curves for Copper Crystals

Resolved Shear Stress kN/m$^2$

Resolved Shear Strain

X  F 11.
△  EG1.
□  EG2.
FIG 6.2

Density of Slip Lines (lines per micron)

Resolved Shear Strain

- \( \triangle \) EG 1
- \( \circ \) O 1
- \( \times \) O 2
- \( \square \) EG 2
- + FII tens.
- O FII comp.
Cyclic Stress-Strain Curves for Copper Crystals

**Fig. 6.3**

Cyclic Stress-Strain Curves for Copper Crystals
FIG 6.4 Fragmented slip lines as seen in Stage I. x10,000

FIG 6.5 Crystal 02. Glide strain in tension 0.028 after test $T_1$. Tension slip lines shadowed black. x24,000
FIG 6.6 Crystal 02. Cum. glide strain = 0.056 after test C1
Tensile slip lines shadowed black. x24,000

FIG 6.7 Crystal 02. Cum. glide strain = 0.080 after test T2
Tensile slip lines shadowed black. x24,000
FIG 6.8 Crystal 02. Cum. glide strain = 0.103 after test C2
Tensile slip lines shadowed black. x24,000

FIG 6.9 Crystal 02. Cum. glide strain = 0.136 after test T3
Tensile slip lines shadowed black. x24,000
CHAPTER VII

Theories to account for cyclic hardening and the
Bauschinger effect: A short review of the literature.

7.1 Introduction.

Before proceeding to the discussion, it is considered relevant to include here an account of the theories proposed by other workers to explain the Bauschinger effect, stability of deformation on unloading and finally cyclic hardening. Chapter I has already covered the principal experimental features of these phenomena. The review will be divided into three parts; the stability during unloading, the Bauschinger effect which has only been studied over a few cycles of deformation, and cyclic hardening which has been investigated up to several thousand cycles of deformation. Fatigue saturation will not be considered since it is not within the scope of this work. Indeed, cyclic hardening up to 5 cycles of deformation is the extent of the literature that should be covered, but since most of the work hardening experiments performed have exceeded this limit, it will be necessary to include them.

7.2 Stability of deformation during unloading.

The lack of any large reverse plastic deformation during the unloading of deformed crystals has been considered by Seeger (6), Buckley and Entwistle (44) and Galligan (35). Seeger in his theory of work-hardening of Stage II considers that dislocations become piled-up against barriers in the crystal. These barriers are Lomer-Cottrell dislocations and form in front of and behind dislocation pile-ups. When the applied stress is
removed the pile-ups cannot then relax in such a way as to give a large plastic strain during the unloading. Galligan explains his unloading effects in Stages I and III (but not in II) by considering long-range stresses due to dislocation arrangements. In Stage I such stresses are absent due to the fact that many dislocations leave the crystal. Hence only a small amount of rearrangement occurs on unloading. The onset of Stage II brings with it trapped dislocations which set up long-range stresses in the crystal and rearrangement is prevented when the applied stress is removed. In Stage III dynamic recovery is treated by Galligan as being responsible for long-range stress relaxation which accounts for the increase in the unloading effect observed by him. He does not suggest what process is responsible for dynamic recovery, but presumably this could be Seeger's cross-slip mechanism.

Buckley and Entwistle base their argument on Mott's (32) work-hardening theory, and use the concept of opposite sign dislocation pile-ups on adjacent slip planes which lock each other in position by means of their stress fields. In addition, both pile-ups are blocked at both ends by obstacles, which they suggest could be Lomer-Cottrell locks. They argue that unloading will not be accompanied by any appreciable dislocation rearrangement, but that when the stress is reversed, a stress asymmetry exists in such a way as to cause the piled-up dislocations to move more readily under a reversed stress than under a corresponding tensile stress. This constitutes a Bauschinger effect which is what their model primarily tries to explain. Their argument is difficult to follow, and hinges on the fact that the back-stress of the
pile-ups helps the dislocations to move backwards, but prevents them from moving forwards. However, according to Seeger's work-hardening theory, pile-ups do not form in Stage I. Hence if Seeger's theory is correct, Buckley and Entwistle's argument is only valid in Stage II and does not account for the Stage I Bauschinger effect. It is also known from thin foil studies of copper (7, 8, 9) and aluminium (54) that pile-ups are not seen in Stage I. Chen, et al. (33) have studied theoretically the properties of dislocation tripoles (i.e., the presence of a free dislocation in the presence of the stress field of a dipole) and conclude that they can account satisfactorily for the stability during unloading. Their explanation sounds reasonable in view of the many dipoles which are observed in Stage I in pure metals (7, 3, 9).

7.3 The Bauschinger Effect

Early theories concerning the Bauschinger effect have been reviewed by Woolley (45). These could only account for a Bauschinger effect in polycrystals, and Woolley demonstrates that the theories are only in qualitative agreement with experimental measurements on polycrystals: he shows that textural stresses only account for 1/10th of the Bauschinger strains which he measured in thin-walled copper tubes. Broom and Ham (85), however, suggest that the quantitative difference between the Bauschinger effects in single crystals and polycrystals at high stresses could be accounted for by the textural stresses.

In place of textural stress theories, Woolley postulates that the reverse motion of a fraction of dislocations generated during prestrain is responsible for the Bauschinger strain. Also he reasons from his results that when the strain
was again reversed (after the Bauschinger strain in compression), 2/3 of the above fraction of dislocation reversed a second time and the remaining 1/3 were trapped. He does not specify the method of trapping.

Mott (84), in a theoretical discussion, proposes two mechanisms to account for the Bauschinger effect. One is similar to Woolley's theory, and the other suggests that dislocation sources, active during the tensile prestrain, generate dislocations of opposite sign when the stress is reversed. These annhilate some of the dislocations generated in tension and hence reduce the flow stress. Mott rejects the second alternative on the grounds that a Bauschinger effect is observed in Stage I where, he assumes, most of the dislocations leave the crystal. Thus the back stress necessary for the reversal of the dislocation sources at low reversed stresses, would not be present. He concludes that the Bauschinger effect is due to the backward movement to the source of untrapped dislocations.

Van Bueren (51) claims that the results of Buckley and Entwistle and others indicate clearly that the reverse motion of dislocations is responsible for the Bauschinger effect. However, to prove this he assumes that during Stage I deformation, dislocations do not leave the crystal but are piled-up against obstacles on the slip planes. This is unlikely in view of the absence of pile-ups and Lomer-Cottrell locking and of the presence of long slip lines observed during this deformation interval (24).

From the optical microscope observations that new slip bands form in compression in preference to the reversal of old ones, Phillips (36) reasons that "it is evidently easier to start
new sources than to overcome the barriers; thus, only those dislocations that are not locked in by barriers can contribute to the Bauschinger strain. The barriers he refers to are of the Lomer-Cottrell type and are formed during the prestrain on the active glide planes. His theory must, therefore, assume that these Lomer-Cottrell barriers do not break down under compression.

Phillips's argument is difficult to follow since he himself admits that no slip lines form during the Bauschinger strain region, an observation endorsed by Charsley and Thompson (62) and Buckley and Entwistle (44). However, if one assumes that during the Bauschinger strain, few if any dislocations reach the crystal surface then his model accounts quite well for the observed Bauschinger effect behavior during Stage I. If a few Lomer-Cottrell dislocations are formed and many primary dislocations are free to reverse, then a rapidly-increasing Bauschinger effect up until the end of Stage I will be obtained, providing a stress asymmetry of the Chen et al. type is assumed.

His model in essence explains the Bauschinger effect by means of the reverse motion of free dislocations, but to account for the small magnitude of the effect in Stage I, he assumes the formation of Lomer-Cottrell locking which is not in agreement with the later thin-foil observation of Steeds and Hazzledine (7) and others (8, 9).

7.4 Cyclic hardening.

Cyclic straining at constant stress amplitude results in a decrease of the hysteresis loop width. To account for this result, Thompson et al. consider the two possibilities mentioned in the last section, i.e.: source reversal and the reverse motion of dislocations. They argue that dislocations moving to and fro against a friction stress in the crystal gradually
become trapped, by some unspecified means, and cause a corresponding reduction in strain amplitude. Their friction stress is caused by the intersection of dislocations on the primary glide system with forest screw dislocations. The loss of energy associated with the hysteresis loop area is taken into account by postulating the non-conservative motion of jogged primary system dislocations; the jogs being produced by the intersection of a dislocation forest by primary system dislocations. The point defects produced by the dragging jogs diffuse rapidly away so that when the jogged dislocations reverse, they cannot annihilate their own defects. Energy is therefore being continuously lost, as is indicated by the area of the hysteresis loop. However as the dislocations become trapped then the seriousness of the energy loss decreases, and the loop area and strain amplitude diminish.

Charsley and Thompson (42) use the same ideas as in the last paragraph to explain their results on loop width variation with cycling in Al, but in addition the type of trapping is specified as Lomer-Cottrell locking. This has the effect of not only reducing the number of mobile dislocations but also the distance through which the active dislocations can move. This assumes that Lomer-Cottrell dislocations are stable under a reversed stress and do not dissociate back into component dislocations. This model accounts for the orientation dependence of cyclic hardening since the rate of formation of Lomer-Cottrell locks is orientation dependent.

The experiments of Patterson (52) and Kemsley and Patterson (47) were carried out at constant strain amplitude, and the resulting hardening is given by an increase in the peak resolved shear stress. They explain their findings
(reviewed in Chapter I) by postulating that the reversibility of dislocation motion is more frequent at low strain amplitudes. At higher strain amplitudes their observations suggest that slip has a "ratchet-like property" and that "slip occurring during the reverse half-cycles is not entirely a reversal of that occurring in forward half-cycles".

Wedsworth (31) discusses his results in terms of the processes of reverse dislocation motion, and of the generation of dislocations in one half-cycle which annihilate with those of opposite sign generated during the previous half cycle. The same processes as have been mentioned above have been considered by many other workers, who have used these mechanisms, in whole or in part, to discuss their results. Theories on fatigue have elaborated on the point defect production theory to account for saturation.

7.5 Summary.

Two processes have been considered in the past to explain the behaviour of metals under reversed stresses. These are:

(1) That dislocations generated during the forward half-cycle reverse their motion during the stress reversal and return towards the source.

(2) Dislocations of one sign are generated by a source during tension, and under compression the same source generates dislocations of opposite sign. These two types of dislocation being on the same slip plane annihilate each other and thus reduce the rate of increase of the dislocation density in the crystal.

Most workers favour (1) to explain the Bauschinger effect but both (1) and (2) are used to account for cyclic strain hardening.
8.1 Discussion of CuAl results under reversed stresses.

The results of sections 5.3 and 5.4 show that many of the slip lines formed during the first cycle of tensile prestrain in CuAl partially reverse during a subsequent compressive strain. Since this is first observed at a compressive stress level of \( \sigma_c = 0.5 \sigma_0 \) (Fig. 5.11) then it is unlikely that the dislocations, responsible for the partial slip line reversal are generated during the compressive strain; sources responsible for these dislocations should require, to a first approximation, the same stress to activate them in compression as in tension. Hence the compressive slip must be caused by the reverse motion of those dislocations generated during the prestrain which did not leave the crystal. A calculation in Appendix B shows that ~80\% of the dislocations formed during the tensile prestrain leave the crystal. The remaining 20\% must be those responsible for the partial reversal of the tensile slip lines, if no new sources are activated during the Bauschinger strain interval of the stress-strain curve. To give the observed magnitude of the Bauschinger effect (~1/10 of the magnitude of the prestrain) these would have to travel on average half the distance, in compression, that the original dislocations travelled in tension. This could be possible, since at \( \sigma_c/\sigma_0 = 0.33 \) the estimated slip line length of 12\( \mu \) does not take into account cross-slip which certainly occurs and is known to increase the slip distance.

Partial reverse slip \( (N_c) \) varies in density with prestrain. This is shown clearly by Fig. 5.7. The variation of \( N_c/N_0 \) with \( N_0 \) is qualitatively the same as \( N_1/N_0 \) in
Chapter IV. The equation of the graph shown in Fig. 5.7 is:

\[ N_c = 0.68N_o - 0.068N_o^2 \]

The maximum value of \( N_c \) occurs at \( N_o = 5.33 \) lines/\( \mu \) and \( N_c = 0 \) when \( N_o = 10.1 \) lines/\( \mu \). Fig. 5.7 also shows that in the heavily deformed region the slip line density is \( \sim 6 \) lines/\( \mu \) since these are the highest \( N_c \) values for which \( N_c/N_o \) was measured. Hence the maximum value of \( N_c \) occurs in the heavily deformed region, whereas in tension the corresponding peak occurs in the transition region. This illustrates that partial slip reversal is greatest where the tensile hardening is greatest.

Since no long coarse slip lines form until \( \sigma_o = -\sigma_o \), the Bauschinger strain in CuAl manifests itself on the surface by means of compressive slip which takes place on those slip planes which were originally active in tension (Fig. 5.14). After the stress \( \sigma_o = -\sigma_o \) has been reached some new compressive slip occurs within the clusters, but the major contribution occurs in parts of the gauge length which were previously undeformed, the larger amount of compressive slip activity occurring on the downhill side of the slip clusters. The density of \( N_c \) increases as the Bauschinger strain increases (Fig. 5.16). This increase is linear above \( \xi_o = 0.40 \). At lower values of \( \xi_o \), \( N_c/N_o \) increases rapidly and then remains approximately constant. The relationship shown in Fig. 5.11 shows that no \( N_c \) type slip lines appear below \( \sigma_o = -0.8\sigma_o \). The upper dotted curve gives the experimentally found relationship between \( \sigma_o = -0.49\sigma_o \) and \( -0.625\sigma_o \). If the Bauschinger strain had set in as soon as stress reversal had started then the dotted line going to the origin would have been expected; this does not appear to be the case. Load-elongation
curves for CuAl are sensibly elastic in the region from $\sigma_0' = 0$ to $\sigma_D' = -0.5\sigma_0'$. This is also the case in Fig. 5.11. Referring again to Fig. 5.10, it is seen that at lower values of $\varepsilon_N$ $N_c/N_0$ increases rapidly then remains, to a first approximation, constant. This indicates that as the amount of strain in the specimen steadily increases the value of $N_c$ remains constant, so that the length and/or height of the slip lines must be increasing more rapidly than at strain values higher than $\varepsilon_0 = 0.40$. $N_c = \text{Constant}$ occurs at a stress of $\sigma_c = 0.75 \sigma_0' = 2.23 \text{ kg/mm}^2$. The foregoing suggests that dislocations start to reverse at $\sigma_0' = 0.5$ and that they are held up at barriers until the stress reaches $\sigma_0' = 0.75$. During this interval the density of slip lines increases rapidly but the length of the lines remains short. At $\sigma_0' = 0.75$ the barriers break down and the piled-up dislocations continue on their slip planes, the density of the lines remaining constant. After this the reverse slip line density rises steadily with strain, as does the slip distance $L$ and the slip line height. It is possible of course that the last two quantities remain constant but this is not suggested by the micrographs of the slip lines in Chapter V, which show that the height and length of slip lines increases between $-0.75 \sigma_0'$ and $0.92 \sigma_0'$. Meakin and Wilsdorf's (22, 23) micrographs of etchpits in $\alpha$-brass show the dislocation arrangement in $\alpha$-brass after a tensile strain. (Since the deformation equivalence of $\alpha$-brass and $\alpha$-CuAl has already been discussed, it will be assumed that the results of Meakin and Wilsdorf are applicable to $\alpha$-CuAl.) Dislocations are arranged in piled-up groups spaced $\sim 10\mu$ apart. These groups are in slip clusters and have an arrangement which
strongly suggests that the type of slip pattern that would be seen under stress reversal would be short slip lines of length \(\sim 10 \mu\), i.e. the type of pattern observed in these experiments.

The results also indicate that ageing influences the Bauschinger effect and the value of \(N^c\). In Figs. 5.7 and 5.11 the values of \(N^c/N^o\) are different when the other parameters are kept constant. Taking the case of \(\sigma_0 = 6.33 \sigma^c\) and \(N^o = 2.9\) lines per micron, it is found that for crystal CA6 (Fig. 5.7) the value of \(N^c/N^o\) was 0.5 but for crystal CA9 (Fig. 5.11) it was only 0.22. CA6 was allowed to age at room temperature after the prestrain for only two hours, while CA9 aged four days being compressed at intervals within this time. If ageing has the effect of locking in dislocations or increasing the barrier strengths, then the above discrepancy is explained, since fewer dislocations would be capable of reversing in CA9, thereby accounting for the low slip line density. Measurements of the Bauschinger strain also demonstrate the way in which this quantity is influenced by ageing. Specimens which are aged longer before stress reversal give a smaller Bauschinger effect. (Fig. 5.1, compare Bauschinger effects of crystals CA6, CA7 (i) and CA5.) Since the Bauschinger effect is caused by the reverse motion of dislocations, it is concluded that the effect of ageing is to lock in a fraction of those dislocations which would otherwise freely reverse in compression. The yield point observations in reversed stressing (section 5.9) support this conclusion. The theory of Hahn (87) attributes the appearance of a yield point to the motion of unlocked dislocations and to the generation of new dislocations. The fewer mobile dislocations in a crystal, the larger is the yield drop. Thus
when the stress is reversed, the reversing dislocations would tend to annihilate to a certain extent via cross-slip and partially reduce the free dislocation density; a small yield point would thus be observed as is in fact the case (Table 5.1, row 13). If the ageing time is appreciable before this reversal, then the yield point would be expected to increase. This too is observed (Table 5.2, row 3).

After compressing beyond the $\sigma_a = -\sigma_0$ for two cycles (section 5.3) total reversal of some tensile slip lines was observed, whereas after only one cycle no such effect was seen. This could be due to the fact that the crystal tends to become in a 'cyclic state' where the to and fro motion of dislocations could be easier. More important, it indicates that once the crystal is stressed beyond $\sigma_a = -\sigma_0$ the sources operative in tension, also operate in compression. In view of the fact that many of the dislocations generated in tension have left the crystal, the total reversal of a slip line would not be possible with the reverse motion of the remaining 20% dislocations by themselves. The deficit must be made up by the generation of new dislocations on the same planes in compression.

**Part II.**

3.2 Discussion of the reversed stressing results of copper.

The slip line results of crystals F11, 01 and 02 show that after a stress reversal the number of tensile slip lines in the surface are sensibly the same as before the reversal. However the results of F11 and 01 show that the number of compressive lines in the repolished region b are greater than in the unrepolished region a.
This is the condition expressed by equation 3.11, and it was shown that this gives values for \( \alpha \) and \( N \) as given by equations 3.20 and 3.21. The values of \( \alpha \) and \( N \) are given in table 3.1. It can be seen that increasing the compressive strain to a value of twice the magnitude of the tensile prestrain (F11, table 3.1, test C2) increases \( N \) but not \( \alpha \). Hence the difference between \( n^c \) and \( n^b \) stays constant after the initial compressive strain, and slip only occurs on new planes. Doubling the compressive strain does not double the value of \( N \); thus, since \( \alpha \) is constant, some of the slip lines formed during test C2 (F11) must grow in height (assuming that experimental error is not the cause of the difference).

Since the slip lines in Cu are so small it is very difficult to determine the exact meaning of the slip line figures which are displayed in tables 6.1b, 6.2b and 6.3b. However let us consider what happens in CuAl in, for example, the experiment on crystal CA4. Here we have shown that after a tensile prestrain of 0.008, tensile slip lines form on the crystal surface (section 5.3). When the crystal was compressed back by a strain of 0.012 no tensile slip lines had disappeared but many compressive slip lines appeared in the repolished region. These were associated with tensile slip lines in the unrepolished region. In addition a number of compressive slip lines went through both regions. In terms of slip line densities this tells us (using the Cu notation for CuAl) that \( n^t = n^a \) and \( n^b > n^c \) after a stress reversal. This is precisely what is observed in the case of copper. In CuAl
where the slip lines were large the condition expressed above was shown to be a partial reversal of the slip lines formed in tension. Thus the simplest interpretation of the Cu results is that a fraction $\alpha$ of the $n^t$ lines undergo a partial slip reversal.

Table 8.1 shows that $\alpha$ increases with prestrain and that the reversal probably occurs during the Bauschinger strain, since the strain interval (F11) containing the Bauschinger strain is the one where $\alpha$ becomes established. The magnitude of partial slip reversal in both Cu and CuAl is about the same. No comment can be made on any differences between $\alpha$ and $N_{gb}/N_{e}$ in view of the ageing effect in CuAl and the lack of quantitative data concerning these fractions. However since the Bauschinger strains in both Cu and CuAl are approximately the same ($\sim \varepsilon_{c}/10$) then the penultimate statement is reasonable.

The effect of repolishing the surface in view of the work of Kramer and Demer (7v) and Barratt (58) must be considered. When the surface of Cu is electropolished, dislocations in the surface layer are removed. If they slip out of the surface during the polishing, then their slip traces will not affect the value of $n^b$. Furthermore the values of $n^b$ after compressive glide strains of 0.006 and 0.072 (crystal F11) fall on the same curve (Fig. 6.2) as the values of $n^t$ when plotted vs. glide strain. There appears to be no reason why the slip line density formed in a compressive strain interval, should be different from the slip line density formed in a corresponding tensile strain interval. However, from the few results available it is difficult to draw dogmatic conclusions on this point.

Since, during the first cycle, slip occurs on new
planes in region a, and no slip lines totally reverse (this is because \( \phi = 0 \); see section 3.10), then new sources operating in compression contribute to the compressive strain. The compressive slip lines are about the same height as the lines formed during tension, i.e.: \( \sim 11 \, \text{\AA} \); this follows from a calculation based on section 3.8.

With continued cycling, the variation of \( \frac{n_c}{n_t} \) with strain given in Table 6.3c shows that this fraction increases with a compressive strain and decreases with a tensile strain. Table 6.3b shows that the largest contribution to the change of \( \frac{n_c}{n_t} \) comes from the numerator. The sum of \( n_c = n_t \) does not increase considerably with cumulative glide strain. After a cumulative glide of 0.136, \( n_c + n_t = 12.0 \pm 4.0 \text{ lines/\( \mu \)} \) (Tables 3.3a and b, test T3) and after test C1 a strain of only 0.056 it is 9.1 \( \pm 3.6 \text{ lines/\( \mu \)} \). Thus after 2\( \frac{1}{2} \) cycles of cyclic straining, the total slip line density does not increase significantly above the value attained after one cycle.

The observed increase and decrease of \( n_c \) and \( n_t \) with cyclic straining indicates that during cycling, some of the slip lines present in one half cycle disappear during the subsequent half cycle. One anomalous result appears in Table 6.3b. This is that after a tensile half cycle (T2) the value of \( n_t \) decreases. This value was checked with a second replica, shadowed with opposite contrast to the first, and the results of the two replicas were found to agree. It is not understood why this is so even in view of the large r.m.s. deviations. It is possible but unlikely that both the replicas used to count the values of \( n_t \) were of poor quality.

In the light of the data presented in Chapter VI the
only processes which can account satisfactorily for the ob-
servations are that of the reverse motion, during compression,
of a fraction of the dislocations which were generated during,
tension, together with the operation of new sources

Part III: General Discussion.

8.3. Summary of the principal experimental findings of Cu and
CuAl under reversed stresses.

The main findings of this thesis, concerning the
behaviour of both Cu and CuAl under reversed stresses are:

(1) Compressive slip occurs on the same
planes as the original tensile slip when the
stress is reversed after a tensile prestrain.

(2) During the first cycle of reverse stressing, the
magnitude of this compressive slip is always less than
that of the original tensile slip (i.e.: partial
reverse slip occurs).

(3) During subsequent cycling the magnitude of this
compressive slip sometimes equals that of the tensile
slip (i.e.: total reverse slip takes place).

(4) In CuAl, the Bauschinger effect is correlated
solely with the occurrence of reverse slip. The
same is probably true for Cu but the evidence is not
conclusive.

(5) The type of reverse slip involved in (1), (2) and
(4) above is due to the reverse motion of dislocations,
which were generated during the prestrain.

(6) If \( \sigma_0 \) exceeds \(-\sigma_0\) then new sources are
activated, and some of the sources active during the
previous half cycle reverse their operation.

(7) The Bauschinger effect has approximately the same magnitude in Cu and CuAl.

Furthermore:

(8) In CuAl the Bauschinger effect is influenced by ageing.

(9) Barriers to the dislocation motion which are established during the first tensile half cycle, break down during the subsequent compressive half cycle before $\sigma_C = -\sigma_C$.

(10) In Cu, cross-slip occurs during Stage I in tension.

3.4. Cyclic work hardening in the light of the results of this thesis.

Any theory which attempts to explain the cyclic work hardening of f.c.c. metals must account for:

(a) The Bauschinger effect.

(b) The lower rate of hardening observed under reversed stresses, compared with that observed in unidirectional tension.

(c) The orientation and plastic strain amplitude dependence of (b).

(d) The lowering of the cyclic hardening rate with the decrease in stacking fault energy.

Furthermore it must take into account the points listed in section 3.3. It must also account for the variation of the Bauschinger effect with the number of cycles as found by Snowden (5A) for Al. The main feature of this variation is that after a few cycles the Bauschinger strain becomes constant with further cycling.
This variation was confirmed for Cu by measuring the Bauschinger strain after each cycle in one of Wadsworth's cyclic stress-strain curves (ref. 31, Fig. 3). The resulting plot of $e_6$ vs. cumulative glide strain is shown in Fig. 3.1.

Some ideas will now be put forward in an attempt to account for the observed phenomena. To formulate a qualitative theory of cyclic hardening, let us first of all consider the case of CuAl. During the tensile prestain dislocations are generated from sources, and those that do not leave the crystal are arranged in long pile-ups on {111} planes of the primary system in Stage I. A small amount of cross-slip occurs and dislocation dipoles are formed. When the stress is reversed the Bauschinger strain is caused by the reverse motion of the dislocations which remain in the crystal. Due to the back stress of the pile-ups, the dislocations find it easier to move backwards under the reversed stress than to progress under a forward stress. After overcoming the stresses which prevented their rearrangement when the forward load was removed, the dislocations move back until more serious barriers, such as Lomer-Cottrell locks, are encountered. (The stresses preventing rearrangements could be of frictional nature arising from short range order or the intersection of the dislocation forest by the primary dislocations.) Due to the size of the pile-ups (~40 dislocations) the stress magnification on the leading dislocations, in the reverse sense, is large enough to break down, or to by-pass by cross-slip; these barriers when $e_6 \approx 0.5e_0$. The stress continues to increase since the supply of reversing dislocations becomes exhausted. As $e_6 \approx e_0$, the reversing dislocations start to activate, by means of their stress fields,
both new sources and in some cases their parent sources. In the latter case dislocations of opposite sign are generated and annihilation of the reversing dislocations follows. The new sources, however, start at the edge of the slip clusters where they generate new dislocations into unhardened virgin material. The reverse motion of the dislocations can thereby be made to account for the Bauschinger strain. Furthermore, the annihilation processes can explain the fact that hardening is lower during cycling than in unidirectional tension.

The breaking down of barriers in CuAl under compression suggests another explanation for the lower hardening rate observed under cyclic stressing than that observed in tension (53§). Some of the barriers set up in the tensile half cycle collapse during the compressive half cycle. Hence the number of barriers at the end of one cycle or twice the tensile strain is less than after the first half cycle. Hence the rate of increase of barriers with strain is less during cycling than in tension only. It follows that the rate of hardening during cycling must also be less than that in unidirectional tension. At large cyclic strain amplitudes, in addition to tensile barriers collapsing, new barriers would form during compression, and the effective rate of barrier formation would increase with strain. The hardening rate during cyclic straining could be expected, on this model, to increase with cyclic strain amplitude.

Now let us consider the case of Cu. During the tensile prestrain the slip line pattern and dislocation arrangements which are set up are quite different from CuAl. Instead of a very homogeneous slip line pattern, a uniform set of slip lines is observed. The height of these tells
us that, 6 - 10 dislocations leave the crystal on each active slip plane. Also, some screw dislocation annihilation occurs which helps to keep hardening low in Stage I. Some of the dislocations are left in the crystal when the prestrain is complete. These, which were generated against a friction stress were either travelling along their \{111\} planes when deformation stopped, or became stuck. Thin foil experiments reveal that many dipoles are at this stage present in the crystal, and by using a mechanism, such as that of dipole locking proposed by Chen et al. (33), the Bauschinger effect can be qualitatively accounted for. The Bauschinger strain will then be due to the reverse motion these dislocations present in the crystal after the prestrain. Again their number diminishes during the Bauschinger strain interval, and as \(\sigma_B \rightarrow \sigma_0\) new sources are activated which send out more dislocations as the resolved shear stress increases. In addition, some of the sources active during tension operate under compression to generate dislocations of opposite sign to the 'reversing' dislocations, which are subsequently annihilated. In this way edge dislocations can be annihilated. This was not possible during the prestrain where only screws could annihilate by cross-slip. This extra process of the annihilation of edge dislocations prevents the dislocation density from rising as rapidly as during the unidirectional tension half cycle, so that the overall hardening rate during cycling will be lower than during unidirectional straining. The orientation dependence of the cyclic hardening rate is satisfactorily accounted for by the arguments of Charsley and Thompson (42), who evoke the increased activity of dislocations on secondary systems as \(\langle110\rangle, \langle11\rangle, \text{ and } \langle100\rangle\) orientations are approached. This
increases the probability of Lomer-Cottrell lock formation which in turn increases the hardening rate for these orientations. At the end of one cycle of deformation the dislocation density is higher than after the prestrain. Further, the number of active slip planes has increased. This is shown in experiments F11, 01 and 02. Hence more dislocations are available for reversal, so that the magnitude of the Bauschinger effect increases. During constant plastic strain amplitude experiments this process continues until the Bauschinger strain becomes synonymous with the plastic strain amplitude. As the to-and-fro moving dislocations become trapped, new ones must be generated by sources to keep the plastic strain amplitude constant.

Different trapping mechanisms are suggested by the various theories of work hardening. Following Hirsch's unidirectional work hardening ideas, it could be postulated that dislocations become continually jogged by forest screw dislocations during their back and forth movement. The jogs thus cause, as suggested by Thompson et al. (41), would cause trails of point defects to be produced. The rapid diffusion of these from their creation sites would prevent recovery of the energy expended during their creation. However, it is unlikely that the forest 'trees' will remain stationary during cycling, so that a dislocation will not necessarily be jogged in the same place during adjacent half cycles. Thus the number of jogs on a given primary dislocation will increase with cycling. If this becomes serious then the dislocation would cease motion and be effectively trapped. However, jogs of opposite sign on the same dislocation can annihilate. Hence two dynamic processes of jog production and annihilation are going on simultaneously and the relative rates of these will decide the fate of the dislocation.
The presence of dislocation loops in cyclically deformed material has led to jog and cross-slip mechanisms of creating sessile barriers on the slip plane which make it more difficult for the dislocation to move. Feltner (39) has based a theory of cyclic strain hardening on a 'flip-flop' action of dislocation loops but this is not likely to account for the cyclic hardening reported here, since it is hardly possible that enough loops would be created to give to correct strain amplitude.

Trapping could also take place by means of Lomer-Cottrell lock formation, which would, as has been shown (43†), account for the orientation dependence of cyclic hardening.

All the trapping mechanisms will account for the plastic strain amplitude dependence of cyclic hardening. The smaller the amplitude of strain then the smaller is the total distance travelled by the dislocations and thus the lower would be the probability of trapping events. This leads to a lower hardening rate for small strain amplitudes as is observed. Calculations based on probability theory and a detailed knowledge of the dislocation arrangements during cycling would be necessary to determine which trapping mechanism gives an amplitude dependence of the correct order of magnitude.

The lowering of the cyclic hardening rate with the decrease in stacking fault energy can be deduced from the arguments laid out above. We have seen that in both Cu and CuAl the cyclic hardening rate is lower than in unidirectional tension because of the process of the annihilation of opposite sign dislocations. In CuAl the additional process of the breaking down in compression of barriers formed during tension, could also account for a lowering in the hardening rate. The barrier destruction is a consequence of the low stacking fault
energy of this material. Dislocations cannot cross-slip very easily and large pile-ups form. If these reverse they impose a large stress on the barriers causing them to yield. The lower the stacking fault energy, the larger will be the pile-ups, causing the barriers to be broken down at lower applied stresses. Hence the hardening rate will be lower for lower stacking fault energy materials.

8.5. Suggestions for further work.

Two suggestions have already been made in the text and are, briefly, that:

(1) In Cu, the variation of slip line density with stress and with strain in Stage I should be investigated, and an attempt to fit an equation to this variation should be made. This could lead to a deeper understanding of the dislocation processes taking part in this early stage of the deformation.

(2) In CuAl, an equation should be fitted to the complete slip line width distribution curve. Furthermore the possibility of a 'universal' slip line width distribution curve could be investigated more thoroughly.

The results on copper and copper aluminium suggest that total reverse slip may occur more frequently with cycling. In CuAl an experiment designed to measure the magnitude and frequency of reverse slip with cycling by use of the polish boundary technique would show how important this process becomes with further cycling. The same experiment could be performed for copper. The specimen would be cycled for n cycles, and the gauge length lightly re-electropolished. The
procedure described in this thesis could then be used to
determine the values of $\alpha$ and $N$. Plots of $\alpha$ and $N$ vs. $n$ would
show how the process of reverse slip varied with cycling.

Experiments concerning the influence of ageing on the
Bauschinger effect should also be carried out to determine how
slip line reversal is affected by this parameter.

The cyclic behaviour of CuAl is still in need of
more investigation. For example, it is not known what effect
the yield point has on the cyclic hardening of this material.
If the cyclic strain amplitude coincided with different points
on the yield drop, different hardening rates may be observed.
Further the Kemsley and Patterson type of investigation on CuAl
single crystals would be of interest especially in view of the
neck formation on this type of crystal.
<table>
<thead>
<tr>
<th>Crystal</th>
<th>Test</th>
<th>$\alpha$</th>
<th>$N$</th>
<th>Glide Strain ($\varepsilon_c$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F11</td>
<td>$C_1$</td>
<td>0.23</td>
<td>3.5</td>
<td>$-\varepsilon_t(0.006)$</td>
</tr>
<tr>
<td></td>
<td>$C_2$</td>
<td>0.23</td>
<td>5.5</td>
<td>$-2\varepsilon_t(0.012)$</td>
</tr>
<tr>
<td>01</td>
<td>$C_2$</td>
<td>0.42</td>
<td>7.1</td>
<td>$-\varepsilon_t(0.024)$</td>
</tr>
</tbody>
</table>

$\varepsilon_t$ = Tensile Strain
\[ \frac{\sigma_B}{\sigma_0} = 0.75 \]

**FIG. 8.1**

Cumulative Glide Strain
APPENDIX A

Calculation of the variation of resolved shear stress in the neck
of a deformed CuAl single crystal.

Consider the crystal shown in fig. A1. ABCD is the primary
slip plane in the undeformed crystal. \( \chi^u_0 \) is the angle between the
Burgers vector and the tensile axis. \( \chi^u_o \) is the angle between the
normal to ABCD and the tensile axis. \( F \) is the applied force on the
crystal and \( \sigma^u \) is the applied stress acting on ABCD. If \( A_u \) is the
cross-sectional area of the undeformed crystal, then by Schmidt's
law,

\[
\sigma^u = \frac{F}{A_u} \cos \chi^u_0 \cos \chi^u_o
\]

Similarly in the heavily deformed region,

\[
\sigma^d = \frac{F}{A_d} \cos \chi^d_0 \cos \chi^d_o
\]

where the \( u \) has been replaced by a \( d \) to indicate the parameter
change when the deformed part of the crystal is considered.
Consider \( l_u \) cm. of undeformed gauge length; if during deformation
it increases in length to \( l_d \), then since volume must be conserved;

\[
A_u \cdot l_u = A_d \cdot l_d
\]

Since the tensile strain is given by \( \varepsilon = \frac{l_d}{l_u} - 1 \), then,

\[
l_d = l_u (1 + \varepsilon)
\]

or:

\[
A_d = \frac{A_u}{1 + \varepsilon}
\]

using eqn. A3.

Using eqns. A2 and A4 to eliminate \( A_d \):

\[
\sigma^d = \frac{F(l + \varepsilon)}{A_u} \cos \chi^d_0 \cos \chi^d_o
\]
The ratio of the flow stresses before and after deformation is:

\[
\frac{\sigma_u}{\sigma_d} = \frac{\cos \lambda_o^u \cdot \cos \chi_o^u}{(1 + \varepsilon) \cdot \cos \lambda_o^d \cdot \cos \chi_o^d}
\]

It is a well known crystallographic relationship that

\[
l_u \cdot \cos \chi_o^u = l_d \cdot \cos \chi_o^d
\]

From eqns. A5 and A6

\[
\frac{\sigma_u}{\sigma_d} = \frac{1_d}{1_u(1+\varepsilon)} \cdot \frac{\cos \lambda_o^u}{\cos \lambda_o^d}
\]

But \( \frac{1_d}{1_u(1+\varepsilon)} = 1 \), so that the final result is:

\[
\frac{\sigma_u}{\sigma_d} = \frac{\cos \lambda_o^u}{\cos \lambda_o^d}
\]

Equation A7 expresses the resolved shear stresses in the undeformed and uniformly deformed parts of the crystal in terms of the ratio of the Burgers vector to the tensile axis in the two regions.
FIG. A1.
APPENDIX B

The percentage of dislocations which leave a single crystal of CuAl during deformation can be estimated from a knowledge of the slip line pattern and crystallographic data in the following manner. The estimate will be made for crystal CA5 to which it is relevant.

Consider a unit length of undeformed crystal, as shown in Fig. Bl, bounded by two (111) planes. The dislocation density ($\rho_u$) is assumed to be $5 \times 10^6$ per cm$^3$. Further, it is assumed on the strength of experimental results (13) that:

$$\sigma \propto \rho^\frac{1}{2}$$

where $\sigma$ = resolved shear flow stress

and $\rho$ = dislocation density

In section 4.12 it has been shown for crystal CA5 that:

$$\sigma_d = 1.16\sigma_u$$

Hence the dislocation density in the heavily deformed region is

$$\rho_d = 1.34\rho_u = 6.7 \times 10^6 / \text{cm}^3$$

The average slip step height is taken as ~ 600 $\AA$, (Fig. 4.17) and the active slip plane density as ~ 5 lines/μ measured on the crystal surface, (Fig. 4.23). The latter is taken from the heavily deformed region of crystal CA6, but is a reasonable estimate for CA5 since both crystals have the same dimensions and orientations. If the active slip plane is measured parallel to the (111) then a value of ~ 7.5 lines/μ will be obtained, when a slip plane angle of ~ 45° is taken into account. The volume of the section shown in Fig. Bl is 1.6 cc. Hence 1 cc corresponds to a gauge length section of 0.625 cm. The total slip per
micron length of crystal (measured perpendicular to (111) )

\[ = 7.5 \times 600 = 4,500 \mu \]

i.e. ~ 2000 dislocations have left the crystal in this micron length.

Assuming that each dislocation which leaves the crystal has a length in the order of magnitude of the crystal diameter, then the total length of dislocation taking part in the deformation of the micron slab is

\[ 2,000 \times 0.6 = 1,200 \text{ cm}. \]

Hence the number of dislocations which is generated and leaves lcc of crystal during deformation is:

\[ 0.625 \times 10^4 \times 1,200 = 10^7/\text{cm}^3. \]

Hence \(10^7/\text{cm}^3\) dislocations were generated and moved out of the crystal to establish the slip line pattern observed in the heavily deformed region. However, the increase in dislocation density of the crystal is

\[ (6.7 - 5) \times 10^6 = 1.7 \times 10^6/\text{cm}^3. \]

It follows that the total dislocation density which was generated during the deformation is the sum of

\[ (1.7 + 10) \times 10^6 = 11.7 \times 10^6/\text{cm}^3. \]

Therefore the percentage of the dislocations density, generated during deformation, which has left the crystal is

\[ \frac{11.7 - 1.7}{11.7} \times 100 = 80.5\%. \]
Although Mader (p6) mentions that the average length of slip lines can be obtained from a knowledge of the density of slip line endings and the slip line density, no formula can be found, by the author, in the literature which gives the relationship between the relevant quantities. An attempt will therefore be made here to derive a simple working formula for the average length of slip lines, L, in terms of the slip line density, N, and the density, D, of line endings. Consider, as in Fig. C1, a rectangular area of surface containing slip lines of finite length. Assume that the slip lines are all longer than the side $l_2$ of the observation rectangle so that both ends of the same line are not in the field of view at the same time. (This can be realized on the electron microscope image screen by adjusting the magnification.) If the line length is constant the number of slip line endings is directly proportional to the slip line density N. If the slip line density is kept constant then the number of ends observed will be inversely proportional to the slip line length. Therefore:

$$D = \frac{kN}{L}$$

where k is a constant.
However, each slip line has two ends so that the density of slip lines must be divided by 2, so that
\[ L = \frac{2kN}{N} \quad \ldots \quad \ldots \quad \ldots \quad \text{Cl} \]

It is difficult in such a simple treatment to determine the value of \( k \) since it will be affected by the slip line length distribution which may or may not be random. Also the fact that a circular and not a square observation area was used will alter \( k \).

In the particular application of equation \( \text{Cl} \) we shall put \( k = 1 \), since only a qualitative variation of \( L \) with strain is required, and \( N = N_c \) (see section 5.6).

So that \( L = \frac{2N_c}{D} \)

where \( D \) and \( kN_c \) are average values taken over ~ 20 readings.
REFERENCES

8. Z.S. Bazinski, ibid, p.93.
42. P. Charsley and U. Thompson, Phil. Mag., 3, 1098, 1950.
82. N.F. Mott, Phil. Mag., 43, 1151, 1952.
89. C.E. Feltner, Phil. Mag., 12, 1229, 1965.
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