The surface markings and dislocation microstructure in fatigued copper crystals.

Makram M. Chochol

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UNIVERSITY OF WINDSOR

THE SURFACE MARKINGS AND DISLOCATION MICROSTRUCTURE
IN
FATIGUED COPPER CRYSTALS

A THESIS
SUBMITTED TO THE FACULTY OF GRADUATE STUDIES
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF MASTER OF APPLIED SCIENCE
IN THE DEPARTMENT OF ENGINEERING MATERIALS

FACULTY OF APPLIED SCIENCE

BY

MAKRAM M. CHOCHOL

WINDSOR, ONTARIO
1972
ACKNOWLEDGEMENTS

The author wishes to express his sincere appreciation and gratitude to Dr. D.F. Watt for his invaluable guidance, encouragement and helpful discussions during all phases of this work. The co-operation and assistance of other faculty and staff of the Department of Engineering Materials are also gratefully acknowledged.

Acknowledgement is due to the National Research Council of Canada for the financial support given for the project in the form of a Research Grant (NRC Grant 746).
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ABSTRACT

The main purpose of the current investigation is to correlate the internal dislocation microstructure with the surface markings produced on O.F.H.C copper single crystals fatigued at constant plastic strain amplitude. Four crystals with two different orientations were fatigued at two different ranges of strain amplitude.

The surface markings of all crystals were mainly in the form of persistent slip bands grouped in bundles. The crystallographic plane of these bundles was either roughly parallel or normal to the primary slip plane.

Shadowed replicas from etched sections were the main technique used in the current work to reveal the dislocation microstructure; they provide a large area that can be studied compared to the very small area provided by a thin foil. However, the electron transmission technique was used on a limited scale to check the fidelity of the dislocation microstructure revealed by the etching technique.

At low amplitude the dislocation microstructure was mainly in the form of long narrow walls parallel to (101) plane and mats laying parallel to the primary slip plane (111). At the higher strain amplitudes the wall-mat structure was observed but also a more equiaxed cellular microstructure was introduced.

Although the dislocation microstructure of each crystal differed markedly from those of the other three crystals, strong correlation did exist between the observed external slip bands and the internal dislocation microstructure for each individual crystal. At the low amplitude the microstructure associated with the interbundle matrix regions was very similar to that associated with the persistent slip bands. At the high
amplitude the microstructure associated with the interbundle regions was equiaxed and cellular, while the microstructure associated with the persistent slip bands was dominated by walls and mats.

A new dislocation model is proposed to explain how the plastic shear strain is accommodated during the saturation period of the test.
I INTRODUCTION

The simplest concept of safe design is to arrange that no part in a structure or machine component carry a stress higher than some fraction of the stress that would cause it to fail in a single loading. Very seldom do parts fail because of the single application of a static load. On the other hand the repeated application of a load of much smaller magnitude may ultimately cause unexpected and catastrophic failure. Such failures are called fatigue failures. While the final failure itself may occur suddenly, the fatigue process is one of gradual deterioration of the metal when subjected to fluctuating stresses. The development of fatigue damage can be summarized in the following stages:

Stage 1. During the early cycles of the test, thin faint slip lines appear on the surface along certain crystallographic directions. As the number of cycles increases, the slip lines become more intense and concentrated in localized regions forming very pronounced slip bands, usually called persistent slip bands (P.S.B.). Within these bands, notches (intrusions), and ridges (extrusions) will eventually form.

Stage 2. A crack will nucleate generally at the surface in the region of a stress concentration. This could be the root of an intrusion. The crack will propagate along the crystallographic slip plane with the maximum resolved shear stress. When the stress concentration factor due to the growing crack becomes large enough, the crack will turn to follow the maximum tensile principal stress plane. When the amount of uncracked area becomes too small to support the tensile stress it will fail with a fracture surface normal to the direction of the principal stress axis.
Generally, the more cycles a material can last before failure, the higher its fatigue strength. There are many different factors that can affect the fatigue strength of a certain component, as listed in figure 1.1.

The most noticeable difference between unidirectional and cyclic straining is the difference in surface behavior these tests produce. While the slip lines in unidirectional straining are kept uniformly distributed as strain continues, the slip lines in cyclic strain concentrate into certain regions forming persistent slip bands, (P.S.B).

The P.S.B. were found to be reversible and reappeared after repolishing on the same areas where they originated before repolishing. These results obviously prove that the P.S.B. are not just a surface phenomenon, but they do extend through the bulk of the specimen. If the dislocation structure of the P.S.B. is markedly different from that of the inactive matrix, then any plane that intersects the plane of the P.S.B., should show discontinuities that correspond to those of the damaged surface.

Conflicting evidence exists about the dislocation structure of the P.S.B., and the depth that these bands penetrate below the surface. To date, most of the detailed structural evidence has been obtained by thin foil transmission electron microscopy. The most extensive studies report a structure for the P.S.B. ranging from a ladder-type of structure to a well defined cellular structure.

The main purpose of the current investigation is to find the relation between the internal dislocation structure and the surface damage of fatigued crystals. An important disadvantage of using the thin foil transmission electron microscopy technique is that only a limited area can be investigated. As well, the thin foil is subject to dislocation loss and distortion during handling and preparation. Therefore, the current investigation was carried out mainly by the etching technique. This technique reveals the dislocation structure over
Applied stress

Stress field (magnitude - axiality - concentration)

Spectrum (stress ratio - residual stresses - stress wave - frequency)

Stressed body

Composition (ferrous - nonferrous)

Substructure (point defects - dislocation - sub-boundaries)

Microstructure (heat treatment - particle distribution - cold work)

Macrostructure (size - shape - notches - surface condition)

Framed structure (simple joints - welds - bolts)

Environment

Temperature

Surroundings (vacuum - radiation - corrosion)

Figure 1.1 Factors affecting the fatigue strength of a machine component (after Yen 1968)
the entire section of the fatigued specimen, so that any gross discontinuity could be detected at low magnification. Replicating the etched section for the electron microscope allowed study of the detailed structure at high magnification without causing any distortion to the true structure. Transmission electron microscopy has been used on limited scale to ensure the validity of the dislocation structure revealed by the etching technique.
II LITERATURE REVIEW

2.1 Historical

In the last hundred years, the attention of engineers and metallurgists has been drawn to the failure of many mechanical components made of high quality ductile metals which were subjected to cyclic stresses. The matter was frustrating because the stress was below the ultimate tensile strength and sometimes even below the stress necessary to initiate plastic deformation.

Experience has shown that most failures in metallic members are of the fatigue type. Extensive research has been done and still continues to find out why a metallic member subjected to cyclic stresses fails after a certain number of cycles even though the applied stress is well below the ultimate tensile strength.

The first systematic study of metal fatigue was undertaken by the German engineer Vohler (1819-1914) in order to understand and prevent the fatigue failure of railway axles. Since that time metal fatigue has been studied extensively by Bauchinger, Jenkins, Cough, Crowan and many others.

In the case of polycrystalline alloys the problem was very difficult to understand. So much of the research has been carried out on polished single crystals of a pure metal. Thus cumulative fatigue damage could be studied without having the complication caused by the presence of the following factors:-

1- Grain boundaries
2- Impurities
3- Second phase
4- Surface roughness

Many comprehensive reviews have been published on the fatigue phenomenon: Thompson and Wadsworth (1958), Han (1967A-B), Plumbridge and Ryder (1969),
Manson (1965), and Feltner and Beardsmore (1969). The main purpose of this chapter is to give a brief review of previous work that relates to the current study.

2-2 Fatigue Hardening

Special experimental procedures designed to control the important fatigue test variables have been developed. Two such tests are:

1- Fatigue between two constant plastic strain limits.

2- Fatigue between two constant stress limits.

In the first procedure, as shown in figure 2.1, the test is usually started by pulling the specimen until it undergoes the amount of plastic strain ($\Delta \gamma_p$) at A. The specimen is then compressed until it undergoes the same amount of plastic strain on the compression side at point B. Notice that the compressive stress necessary to start the deformation is less than the tensile stress. This phenomenon is called the Bauschinger effect. Also notice that the tensile stress (peak stress) to achieve the same plastic strain is increased as the number of cycles increases. This is called cyclic hardening. The cyclic hardening will continue until saturation is reached, beyond which point the peak stress and the shape of the hysteresis loop will remain constant.

The second procedure is to fatigue the specimen between two constant stresses. In this case the width of the loop decreases as the number of cycles increases showing that the specimen is undergoing fatigue hardening. Fatigue hardening will continue until saturation is reached.

Results from either of the above two procedures can be presented in graphical form. The S-N curve shows the relationship between the stress amplitude and the corresponding number of cycles that the specimen can stand before failure. As shown in figure 2.2, there are primarily two different types of S-N curves. Curve A, which is characteristic for ferrous alloys,
Figure 2.1 Fatigue between two constant plastic shear strains.

Figure 2.2 The S-N curves.

Figure 2.3 The fatigue hardening curves.
shows a fatigue limit which is the stress amplitude below which the specimen will last for an infinite number of cycles without failure. Curve B, which is a characteristic of most non-ferrous alloys, does not show the fatigue limit phenomenon, i.e., no matter how low the stress amplitude is, the material will eventually fail. The fatigue limit in this case is replaced for design purpose by the endurance limit which is defined as the cycle amplitude required to cause failure after a specified number of cycles.

Fatigue hardening behavior can be presented as shown in figure 2.3 by plotting the peak stress \( \tau_p \) (for the loop of width \( 2\gamma_p \)) and the corresponding number of cycles. It is well established that the cyclic hardening curve of a single crystal is highly dependent on the following parameters:

- Plastic strain amplitude
- Stacking fault energy
- Orientation
- Temperature

Kemsley and Paterson (1960), showed that the cyclic hardening rate is strongly dependent on the strain amplitude. On aluminum crystals, Snowden (1963) showed that for high cyclic stresses the specimen saturated after 50 cycles, while for low cyclic stresses, it saturated after 800 cycles. Similar results have been obtained by Eibner and Backofen (1959), and Alden and Backofen (1961) by testing copper crystals in bending. For copper polycrystals tested in push-pull fatigue, Fellner and Laird (1967) showed that in the high amplitude range the cumulative strain at saturation did not change. Wood and Segall (1957), by fatiguing copper crystals in reverse twist showed that for the higher amplitude test, saturation was reached at slightly larger cumulative strain but in far fewer cycles. By fatiguing two copper crystals, both oriented for single slip, at two different strain amplitudes, \( \pm 0.012, \pm 0.003 \) Watt (1967) showed that in the lower amplitude case, the cyclic hardening rate was slower and saturation
was reached at about 26 cumulative shear strain. In the higher amplitude case, the cyclic hardening rate was higher, and the specimen saturated at about 3 cumulative shear strain. He also reported that the initial hardening rate was higher for specimens oriented for multiple slip.

Feltner (1965) showed that the initial cyclic hardening rate is increased by increasing the stacking fault energy and or the temperature.

2.3 Detailed Observations of Fatigue Surface Damage

During the early stages of cycling, the deformation is uniformly distributed over the specimen in the form of fine slip lines. Eventually in the saturation region the deformation was found to be concentrated in certain areas which were called persistent slip bands. They were originally called persistent because parts of them did not disappear after electropolishing 2 microns off the specimen's surface (Thompson, Wadsworth and Lout, 1956). Today we would guess that Thompson et al. actually saw only intrusions or microcracks after electropolishing. However since cycling reproduces the bands in some detail, the name persistent slip bands is appropriate for the bands of intense slip.

Within these persistent slip bands intrusions and extrusions are developed. The crack usually is nucleated at the surface where there is a stress concentration (root of an intrusion). The crack will propagate in a transgranular manner following the slip plane with the maximum resolved shear stress. At a certain length the crack will propagate normal to the tensile axis until complete failure, when the material becomes too weak to withstand the normal tensile stress. The observation of the P.S.B. has been reported repeatedly since Thompson et al.'s work.
Alden and Backofen (1961), in their experiment on aluminum crystals fatigued in plane bending, showed that during the fatigue hardening period, the slip lines were fine and uniformly distributed. In the saturation region, they found that those slip lines were broken into intense slip line bundles. Watt (1969), reported similar behavior under similar conditions to the current work.

The persistent slip bands are almost never a single intense slip line. Rather they are composed of clusters of short intense slip lines. In general, these clusters tend to align along a direction roughly parallel (i.e. within 20°) to the primary slip plane. Some exceptions have been observed which are highly pertinent to the present work. Gastelow (1971), found that as cycling proceeded in his copper crystals, the slip concentrated in discrete regions normal to the primary slip plane as shown in figure 2.4. The same type of surface pattern was reported by Woo, (1972). Gastelow related the behavior to the development in the crystal interior of alternating hard and soft regions normal to the primary slip plane, with the slip process concentrated in the soft ones.

Most of the previous work reported the nucleation of the crack in persistent slip bands. King and Teer (1969), fatiguing aluminum polycrystals at 50 Hz and 1950 lb/in², reported a new surface phenomenon (rumples) in which the crack was nucleated. These rumples, shown in figure 2.5 were found to be short surface undulations in regions where the slip markings appeared very uniform over the whole grain. As cycling continued they increased in length. Arnell and Teer, (1969) studied in detail the effect of grain orientation on the surface behavior. They fatigued aluminum polycrystalline specimens with the same specifications used by King and Teer (1969). They identified the orientation of the individual grains by the standard Laue back-reflection technique, but using an electron microscope aperture to produce a very fine X-ray micro beam. They reported that for grains oriented around the center of the standard triangle of the stereographic projection, the only feature observed on the grain surface
2.4 Persistent slip bands on fatigued copper crystal (Gostelow, 1971).

Fig. 4 The development of cramped bands. (a) After 25,000 cycles; (b) after 31,000 cycles.

2.5 Rumples observed on the surface of fatigued Al polycrystal. (King and Teer, 1969).
was the usual P.S.B. As the orientation of the stress axis moved toward any of the triangle corners, other surface features (rumples) were formed besides the usual P.S.B. The persistent slip bands were totally absent in grains with their stress axis coinciding on any of three corners of the standard triangles. By using the trace analysis on the stereographic projection they showed that for grains oriented near the [001]-[111] and [001]-[011] boundaries, two sets of slip bands were observed parallel to the primary and conjugate slip plane. The crystallographic plane of the rumples could not be identified, but they found that the individual rumples were parallel to the trace of cross slip plane while the rows of rumples were nearly parallel to the trace of the primary slip plane.

Extensive research was done to find out whether the P.S.B. are restricted to the surface or extended through the whole specimen. Many observations suggest strongly that they do extend through the whole crystal. Watt (1967), electropolished 55 microns off the surface of a fatigued crystal after 3000 cycles. When fatiguing was resumed, it was found that the P.S.B. were reversible and increased in roughness as cycling proceeded. As shown in figure 2.6 after 7000 cycles, the original pattern of the P.S.B. was reproduced. The same behavior happened again even after electropolishing 310 microns off the surface at 7000 cycles. This P.S.B. reproduction was seen by Greenough and Roberts (1965) after 50 microns were polished off fatigued copper polycrystals. King and Yeer (1959), on aluminum polycrystals, electropolished the surface to remove the surface markings and annealed the specimen for 1 hr at 500 c. Upon refatiguing, the original surface markings were eventually reproduced. In another attempt to prove the bulk behavior of the P.S.B., Watt (1969) electropolished the surface deep enough to remove any characteristic surface structure. He refatigued the specimen, but it did not show any change in the mechanical response. This showed that the surface structure is not grossly
Figure 2.6 A single strip of surface of a fatigue specimen showing the slip-unslip process that occurs at the site of the p.s. bands. (Watt, 1967)

(a) Before electropolishing
(b) ½ cycle after electropolishing
(c) 1 cycle
(d) 1½ cycles
(e) 10½ cycles
(f) 11 cycles
(g) 100½ cycles
(h) 101 cycles
(i) 2500 cycles
different from that of the crystal's interior.

2.4 Plastic Strain in Fatigued Metals

A major portion of this chapter deals with the dislocation structure of fatigued copper. This dislocation structure was found to be mainly composed of dislocations, dipoles and prismatic loops. Clarification follows on the nature of a dislocation, and how dipoles and prismatic loops can be generated.

2.4.1 Dislocations

To explain the discrepancy between the theoretical shear strength of a perfect crystal to that of the actual one, many investigators independently proposed the existence of line defects (dislocations) in the crystal. With the existence of this kind of defect, the slip process could take place at the observed applied shear stresses.

There are two extreme types of dislocations. The edge type shown in figure 2.7, has the Burgers vector $\mathbf{b}$ normal to the dislocation line. An applied shear stress $\tau$ will keep pushing the dislocation until it emerges from the surface producing a slip step equal to $\mathbf{b}$. The screw type, shown in figure 2.8, has the Burgers vector $\mathbf{b}$ parallel to the dislocation line. An applied shear stress $\tau$ will push the dislocation line in a direction normal to $\mathbf{b}$ until it reaches the surface parallel to $\mathbf{b}$. It will also produce a slip step equal to $\mathbf{b}$ on the plane which lies normal to $\mathbf{b}$. The main difference between the two types is that the screw dislocation can slip along any close-packed plane which contains its Burgers vector, while the slip of the edge type is restricted to the plane that contains the dislocation line and its Burgers vector.

It is well known that deformation increases the number of dislocations in the crystal. Many mechanisms have been proposed to explain this phenomenon. The most important is the Frank-Read mechanism (1953) and which is
Figure 2.7 The edge-type dislocation.

Figure 2.8 The screw-type dislocation.
Fig. 6.6 The plane of the figure is the slip plane of a section of dislocation $DD'$; the dislocation leaves the plane of the figure at the fixed points $D$ and $D'$. An applied stress produces a normal force $rb$ on the dislocation and makes the dislocation bulge. The initially straight dislocation $a$ acquires a curvature proportional to $r$. If $r$ is increased beyond a critical value corresponding to position $b$, where the curvature is a maximum, the dislocation becomes unstable and expands indefinitely. The expanding loop doubles back on itself, $c$ and $d$. Unit slip occurs in the (shaded) area swept out by the bulging loop. In $e$ the two parts of the slipped area have joined; now there is a closed loop of dislocation and the section $DD'$ is ready to bulge again and give off another closed loop.

2.9 Frank-Read source. (Read, 1953)
explained in figure 2.9. When a single crystal is pulled in tension, it is found that an increasingly higher stress is required to keep the deformation progressing (work hardening). The main reason for this kind of hardening was found to be due to the creation of dislocations, dipoles, and prismatic loops that act as obstacles to the dislocations which produce the imposed strain. More stress is required to allow the moving dislocations to bypass these obstacles as the density of the obstacles increases.

2.4.2 Dipoles and Prismatic Dislocation Loops

Dipoles are pairs of mutually attracting edge dislocations of opposite sign. Prismatic loops are closed loops of edge dislocation which can slip in a direction other than the plane of the loop and so leave a prismatic shaped slip path. Dipoles, multipoles and prismatic loops are commonly observed in fatigued microstructures.

Many mechanisms for the generation of dipoles and prismatic loops have been proposed. In Gilman's model (1962), as shown in figure 2.10 part of the moving screw dislocation will cross slip from plane I to plane II. The cross glide motion may cause two things:-

1. Regenerative multiplication and production of a prismatic loop, figure 2.10 c.

2. Production of an edge dislocation dipole, figure 2.10 d.

Another model has been proposed by Segall et al (1961). In figure 2.11 the advancing screw dislocation leaves behind a dipole which can be terminated by cross slip. Many papers have been published on the interaction between dislocations and dipoles: Sharp and Makin (1964), Chen (1964), and Kroupa (1965). There is no evidence that the dipoles may act as strong barriers for the advancing dislocations. However, they may cause some torque
Figure 2.10 Gilman's mechanism to form dipoles and prismatic loops (Gilman 1962).

Figure 2.11 Segall's mechanism to form prismatic loop (Segall 1961).
on the screw portion of a dislocation line, forcing it to cross slip, (Kroupa, 1965).

2.5 The Bulk Microstructure of Fatigued metals

It is strongly believed that the dislocation structure produced by cyclic fatigue is highly dependent on the following parameters:-

- Strain or stress amplitude
- Stacking fault energy (S.F.E.)
- Temperature

One of the earliest investigations of the fatigue microstructure using the electron microscope was carried out by Segall and Ibartridge (1959). In their work they tried to compare the dislocation arrangement produced by fatigue with that produced by unidirectional straining. On aluminum polycrystals, with an average grain size of 10 microns, they showed that the dislocation arrangement for high stress fatigue ($N_f = 5 \times 10^4$ cycles), was similar to that of unidirectional straining but with a larger cell size. For low stress fatigue ($N_f = 3 \times 10^6$ cycles), the dislocation structure was mainly composed of prismatic loops heterogeneously distributed. Also Segall et al. (1961) on copper crystals fatigued in plane bending for $5 \times 10^4$ cycles ($10\%$ of $N_f$), found areas with high dislocation density mainly composed of dipoles and loops elongated along the primary slip plane and normal to the Burgers vector.

Basinski, Basinski and Howie (1969), made a detailed study of the dislocation microstructure developed during the hardening of copper single crystals. They showed that during the early cycles the etched dislocation microstructure on the cross slip plane was mainly in the form of dense dislocation rows parallel to the trace of the primary slip plane. On the primary slip plane patches with high dislocation density were observed, but most of the area was nearly empty. As cycling proceeded the microstructure on
the cross slip plane transformed to long walls normal to the trace of the primary slip plane. Along the primary slip plane the microstructure consisted mainly of tidy regions (bundles of dislocation parallel to [121]) and untidy regions (a vaguely cellular appearance). The majority of dislocations were primary edge-type multipoles. At saturation the cross slip plane microstructure was broken into spotlike structure, while along the primary slip plane the microstructure remained unchanged but with thicker walls and higher dislocation density.

In summary, the main structure observed by Businski et al. was essentially one-dimensional veins elongated in the [121] direction, which is the direction along which a primary edge dislocation lies. They also saw loose cellular areas and some multipoles standing perpendicular to the Burgers vector.

Hancock and Grosskreutz (1967) and Shinozaki and Embury (1969) independently examined (101) foils and (111) foils from copper single crystals fatigued during the hardening period. Both groups reported that during hardening complex braids of dipoles and monopoles are created and accumulated in the region of forest dislocations. As cycling proceeded, the braids become aligned along the [121] direction and the trace of the conjugate plane. These braids mainly consisted of large numbers of Lomer-Cottrell dislocations, Frank dipoles, and dislocations of the primary and coplanar systems. As saturation was reached the separation of the braids became constant. The "braid" structure seems to be basically the same as Businski's "vein" structure.

In the saturated structure, both Watt et al. (1970) and Gostelow (1971) found structures on the primary slip plane similar to that shown in Figure 2.12. But along the (121) plane, Gostelow showed a cellular structure, Figure 2.13. From his observations along the primary slip plane and the (101) plane, Gostelow suggested that the nature of the dislocation distribution that gave
2.12 Dislocation microstructure along the primary slip plane ($\gamma=\pm 0.003$) (Watt et al., 1970).

2.13 Dislocation microstructure along the (121) plane ($\gamma=0.01$) (Gostelow, 1971).
rise to the cell-like appearance of figure 2.13 could be walls on the (111) and (101) planes and planes at 45° to these. On the (121) plane Watt reported long walls normal to the Burgers vector.

Both Lukas et al (1968) and Laufer and Roberts (1969) tested copper single crystals at high frequency. They reported distinctly different structures for the persistent slip bands (section 2.6) but both groups observed Basinski's vein structure in the inactive matrix regions.

Waldron (1965) testing Al-1% Mg crystals found that the structure transformed from fairly uniform distribution of elongated loops and jogs to a sharp well-defined cellular structure as the amplitude is increased. By fatiguing polycrystalline copper tubes in high strain amplitude torsion, Pratt (1966) reported a cellular structure as shown in figure 2.14 with the cell size inversely proportional to the saturation stress. To calculate the cell size, Pratt divided the area of the micrograph by the number of cells to get the average area of each cell, and consequently the average cell diameter. This is an accurate technique only if the cells are about equiaxed, but it does not give a representative value of the sub-boundary spacing shown in figure 2.14.

It has been shown that fatigued copper (S.F.E. = 40 erg/cm²) has cellular structure at high amplitudes. Feltner and Laird (1967), did not find any cells in fatigued Cu-7.5% Al (S.F.E. = 2 erg/cm²). Lukas and Klesnil (1967) reported that an increase in the zinc content of brass (i.e. a decrease in S.F.E.) results in a gradual tendency to homogenize the dislocation distribution.

The effect of temperature is not as clear as that of amplitude and stacking fault energy. However, Feltner (1963) showed that in polycrystalline aluminum fatigued at 78°K with a normal strain amplitude of ±0.002, loops constitute the vast majority of the sub-grain walls. Similarly, for copper fatigued at room temperature, the predominant structure is loops, dipoles and multipole tangles. Pratt (1967) showed that the cell size was

16 Schematic summary of dislocation structures in f.c.c. metals as a function of amplitude, temperature, and stacking-fault energy. (Feltner and Laird.44)

2.15 Schematic summary of dislocation structures in f.c.c. metals as a function of strain amplitude, temperature, and stacking fault energy. (Feltner and Laird, 1965).
decreased by decreasing the temperature from room temperature to 76°C.

The general effects of strain amplitude, stacking fault energy and temperature are best summed up in a diagram, figure 2.15 created by Feltner and Laird (1967). The diagram is out of date in that the section labelled prismatic loops should be multipole veins and walls, when more recent findings are included.

2.6 Dislocation Structure Associated with P.S.B.

As shown in section 2.3, many reported surface observations indicate that the P.S.B. are bulk phenomena. Many attempts to show this have been made by investigating the internal dislocation structure using transmission electron microscopy. The wide variation in results may in part be attributed to the difficulty in preparing thin foils large enough to reveal the successive change in structure from the P.S.B. to the matrix.

McGrath and Waldron (1964), cut thin specimens from the surface of an Al-1%Mg specimen. By only polishing the inside surface, they preserved the external structure of the P.S.B. They found poorly defined cells associated with the P.S.B. The cells were composed mainly of loops and tangled dislocations, while the matrix was found to be mainly dipoles, loops, and dislocations. Laufer and Roberts (1964), fatigued copper single crystals in bending (surface stress = 3.16 kg/mm²). They found the ladder-type structure figure 2.16 for specimens fatigued below 1000 cycles. Above 1000 cycles, the structure of the P.S.B.'s sometimes developed into well-defined cells. They also found that the cellular structure disappeared at 1/20 of the thickness below the surface. Lukas et al. (1966), fatigued copper polycrystals (push-pull, σ = 9 kg/mm² at 105 Hz). They showed that extrusions were associated with soft regions that act as channels for the dislocation motion, figure 2.17. Later Lukas and his co-workers (1968), fatigued copper single crystals (push-pull, σ = 6.5 kg/mm², 105 Hz). They showed that the matrix
General arrangement of dislocations at a fatigue striation. The area of the specimen examined in the electron microscope is indicated by the arrow in the insert.

2.16 The ladder-type structure (Laufer and Roberts, 1964)

2.17 Dislocation free channels producing persistent slip bands. (Lukas et al. 1966)
2.18a Dislocation microstructure along the (121) plane and within a persistent slip band in a high frequency fatigued copper crystal. (Lukas et al, 1968).

2.18b Space model of microstructure associated with persistent slip bands. (Lukas et al, 1968)
consisted mainly of veins in the [121] direction. The P.S.B. were composed mainly of short irregular cylinders, the axis of which is normal to the primary slip plane, figure 2.18 a and b.

Watt (1967), showed a differential etching that the P.S.B. extend throughout the specimen at shear strain amplitudes of ±0.012 and ±0.006, while at ±0.003 the whole section responded the same to the etchant (FeCl₃) dissolved in HCl). It may be concluded from this, that at high amplitudes the dislocation structure of a P.S.B. is different from that of the matrix, while at low amplitudes the difference is not enough to be detected by a crude etching technique.

2.7 Dislocation Models of the Fatigue Hardened State

Many models and mechanisms have been proposed to explain the fatigue problem. Yet none of these has fully explained the nature of the hardening process, the reason for the formation of P.S.B., nor how the enforced plastic shear strain is accommodated under different testing conditions. In this section some of these models will be discussed briefly.

2.7.1 Mechanisms Involving Cross Slip

Broom and Ham (1962), concluded that the cross slip process is necessary for the formation of P.S.B. The minimum stress for P.S.B. formation had a temperature dependence similar to which is the stress at which cross slip begins in a unidirectional test. Due to the difference in flow stress between the P.S.B. and matrix they concluded that each has a different structure. The difference in flow stress was found to be 20%. To explain the formation of P.S.B., Greenough and Roberts (1965), proposed the existence of dislocation sources (formed by cross slip) acting co-operatively on different parallel planes having a total thickness equal to the thickness of the P.S.B. However, Roberts and Greenough did not explain how a set of sources could work co-operatively in
such a way that the regions which carry the most strain are the zones which remain softest.

2.7.2 Avery and Backofen (1963)

Avery and Backofen suggested the existence of two main categories of internal stress:

1. Short range stresses which result from friction
2. Long range stresses which result from dislocation pile-ups, dislocation tangles, and grain boundaries.

As shown in figure 2.19 during low amplitude fatigue, the dislocations carrying the enforced plastic strain oscillates within the short range stresses. At high amplitude fatigue, dislocations interact with the long range stresses. This explained why their reported hardening rate was not sensitive to the orientation or grain size of the specimens at low amplitude. They also proposed two possible reasons for zero hardening. The first is a balance between hardening and softening caused either by dynamic recovery or by a balance between the rate of creation and sweeping up of obstacles by moving dislocations. Secondly, the reversible portion of strain is increased as a portion of the total enforced strain until they are equal to each other, resulting in zero hardening. Feltner (1965) objected to this reversible mechanism on the basis that the hysteresis loop should be closed if the enforced strain is totally reversible and is being carried out mainly by dislocations bowing back and forth. Watt (1969) showed that during saturation the damage keeps accumulating proving that a portion of the enforced plastic strain must be irreversible.

2.7.3 Feltner (1965)

Feltner developed the first exclusive model that describes the dislocation behavior during hardening and saturation periods. During the first few cycles, prismatic loops (section 2.4) are formed and act as obstacles to the moving
Fig. 6. Schematic view of internal stresses in a crystal. At "low" amplitudes the major contribution to the flow stress arises from the friction stress. At higher amplitudes the interaction of dislocations with the long range stress fields becomes controlling.

2.19 Long and short range stresses in fatigued copper (Avery and Backofen, 1963)
dislocations. As cycling proceeds, the rate of prismatic loop formation decreases because some of the screw dislocations become entangled in their own debris. For this reason, the fatigue hardening rate will slow down. At this stage a relatively high portion of the enforced plastic strain is carried by the flip-flop motion of the prismatic loops, figure 2.20. During saturation, the rate of loop formation is zero due to the absence of screw dislocations free to form the loops. The fatigue hardening rate becomes zero, and the enforced strain is totally carried by the flip-flop motion of the prismatic loops.

Using the electron microscopy results of Segall et al. (1961) and the following equation:

\[ \gamma = 2 \frac{b}{h} \frac{1}{l} \frac{p}{p} \]

Where

<table>
<thead>
<tr>
<th>( \gamma )</th>
<th>Plastic shear strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>( h )</td>
<td>Average height of loop</td>
</tr>
<tr>
<td>( l )</td>
<td>Average length of loop</td>
</tr>
<tr>
<td>( p )</td>
<td>Average density of loop</td>
</tr>
</tbody>
</table>

Feltner showed that for a loop density of \( 10^6 \) loops/cc the calculated shear strain had the same order of that of the applied shear strain. In objecting to this model, Watt (1967) showed that the minimum volume that can contain \( 10^{16} \) non-interacting prismatic loops is equal to 25.6 cm\(^3\) which shows the lack of validity in the density used by Feltner. Adding to the previous objection, most of the results reported in recent work shows that the dislocation structure at low amplitudes (\( \gamma \leq 0.003 \)) is regular walls of high density entangled dislocations. At high strain amplitudes (\( \gamma = 0.01 \)), the structure was cellular. In either case, this shows that the even distribution of prismatic dislocation loops reported by Feltner does not exist, and only about 15 to 20% of the crystal volume is occupied by dislocations while the balance is nearly dislocation free.
Figure 2.20 The flip-flop motion (Feltner 1965).
2.7.4 Shuttling Mechanism

Watt and Ham (1967), and Basinski et al. (1969) suggested that a portion of the enforced plastic strain could be carried by dislocations moving back and forth between the walls. As dislocations pile up on one side of the cell, they will increase the back stress. When the load is reversed, the back stresses help the leading dislocations to become disentangled from the walls and move back. Although this mechanism is more reasonable than the flip-flop mechanism, it does not discuss specifically any interaction between these dislocations and the sub-boundaries. The vagueness of this model makes it impossible to either prove or disprove it by experiment or observation.

2.7.5 Kuhlmann-Wilsdorf and Nine (1967).

Kuhlmann-Wilsdorf and Nine fatigued copper crystals in torsion. They reported that the P.S.B. were absent where the resolved shear stress on the cross slip plane was a maximum. The P.S.B. existed where the resolved shear stress along a co-planar system on the primary plane was a maximum. They proposed that during the early cycling, the dislocation density increased until it reached a critical value. At this value the applied resolved shear stress would be enough to form dislocation twist boundaries forming a network. Dislocations moving toward these boundaries sweep out obstacles, hence making regions in the vicinity of the boundaries easy slip regions. As cycling proceeded the network was transformed to a well defined cellular structure. From the proceeding results, Kuhlmann-Wilsdorf and Nine concluded that the P.S.B. were formed due to the formation of twist boundaries which transform to cellular structure as cycling proceeds.
Based on a single electron micrograph by Laufer (1965), they assumed that the P.S.B. would have a specific cellular structure. Other work by Lukas et al. (1968), Laufer and Roberts (1964), McGrath and Waldron (1964), Gostelow (1971) and the present work shows that, in general, P.S.B. do not have the structure proposed by Kuhlmann-Wilsdorf.
III EXPERIMENTAL TECHNIQUES

This chapter contains a detailed description of the techniques used to produce the single crystal specimens, to fatigue test these specimens, and finally the techniques used to reveal their dislocation microstructures. Specifications for standard commercial equipment used are listed in appendix C. The final part of this chapter contains an error analysis of the experimental data.

3.1 Growing Single Crystals

A furnace was built to grow single crystals of O.F.H.C. copper in a nitrogen atmosphere. Figure 3.1 shows a schematic diagram of the different parts of the furnace. The heating element was made of silicon carbide (Crusilite) in the form of a long cylindrical spiral. A mullite tube isolated the graphite mould from the ambient atmosphere. Measurements of the temperature gradients along the centerline of this tube are shown in figure 3.2. Crystal growth was accomplished by a modified Bridgman technique. The current input to the heating element was lowered at a controlled rate so that the solid liquid interface moved upward, from the seed crystal into the molten copper.

3.2 Determination of Orientation

The orientation of the single crystal was determined by back-reflection Laue technique, as shown in figure 3.3. The stage shown in this figure was designed to rotate the specimen about two perpendicular axes (specimen axis and vertical axis) to get a specified plane parallel to the plane of the cutting electrode in the electrical discharge machine. The stage could then be transferred to the EDM machine and the appropriate cut made.
Figure 3.1  Sectional view of the single crystal furnace.
Figure 3.2 Temperature gradients along the centerline of the single crystal furnace.
3.3 Back Reflection Laue Camera Arrangement.
3.3 Preparing the Fatigue Specimen

The as-grown crystals were 3/8 inch in diameter and about 3 inches in length. The fatigue specimens shown in figure 3.4, were cut to shape from the single crystals on the E D M. The two flat surfaces and the curved ones on the gage length were carefully wet polished with fine emery paper (grit 600), then washed in water and dried in a stream of warm air. Following this the specimen was annealed in a nitrogen atmosphere at 950°C for 10 hours. This was done to reduce any dislocation microstructure developed during preparation.

After annealing, the specimen was rinsed for 10 seconds in 50% nitric acid solution and washed thoroughly in distilled water. Then the specimen was electropolished according to the following specifications:

- **Electrolyte**: 50% Orthophosphoric acid
- **Voltage**: 10 V
- **Current density**: 1 Amp./cm²
- **Temperature**: -20°C
- **Time**: 10 Minutes

After electropolishing the specimen was rinsed in distilled water and then sprayed with alcohol. This washing technique was repeated several times and the specimen was finally dried in a stream of warm air.

3.4 Fatigue Apparatus and Fatigue Procedures

The specimens were fatigued at constant plastic strain using an adapter attached to the Instron machine as shown in figure 3.5. The load and the extension were plotted on the Instron X-Y chart recorder.

During the hardening period the reversal points were controlled manually to maintain the same plastic strain amplitude. After saturation, the machine was set to run automatically between the two fixed reversal points.
Figure 3.4  The fatigue specimen
Figure 3.5. Instron Universal Testing Machine
About 300 cycles after saturation, the strain rate was increased from 0.006 per minute. The test was continued for about 8000 cycles for all specimens of different orientations and at different strain amplitudes according to table 3.1

3.5 Optical Microscopy

The slip bands on the crystal surface and the etched surfaces were investigated using both optical and electron microscopy. Thin foils were also examined by transmission electron microscopy to check the fidelity of the etched dislocation patterns.

3.5.1 Polishing and Etching of Interior Planes

Before etching, the specimens were cut parallel to the specified plane on the E D M using a sheet of brass 0.02 inch thick as the cutting electrode. The surface was rinsed in 50% nitric acid for 10 seconds and washed in water to eliminate any traces of the oil left on the surface during the cutting. The surface was then chemically polished and etched according to the following steps:

1- The surface was rubbed very carefully over a piece of soft cotton sheet soaked in a saturated solution of cupric chloride in concentrated hydrochloric acid (Mitchell, 1967). The cotton was stretched over an empty beaker and was held in place by a rubber band.

2- The specimen was washed in distilled water.

3- The surface was rubbed very carefully in a circular manner using the same technique as in step 1, but with a different polishing solution having the following composition: (Mitchell, 1967)

<table>
<thead>
<tr>
<th></th>
<th>50 parts by volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orthophosphoric acid</td>
<td></td>
</tr>
<tr>
<td>Glacial acetic acid</td>
<td></td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td></td>
</tr>
<tr>
<td>Hydrochloric acid saturated with cupric chloride</td>
<td></td>
</tr>
<tr>
<td>Specimen</td>
<td>Orientation</td>
</tr>
<tr>
<td>----------</td>
<td>-------------</td>
</tr>
<tr>
<td>A</td>
<td></td>
</tr>
<tr>
<td>$\bar{A}$</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td></td>
</tr>
<tr>
<td>$\bar{B}$</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.1 Fatigue Program.
4- Again the specimen was rinsed in distilled water then sprayed with alcohol. This step was repeated several times and after the last spraying the surface was dried in a stream of warm air.

5- The surface was etched with a solution having the following composition: (Livingston, 1960)

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Parts by Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glacial acetic acid</td>
<td>25</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>15</td>
</tr>
<tr>
<td>Liquid bromine</td>
<td>1</td>
</tr>
<tr>
<td>Distilled water</td>
<td>90</td>
</tr>
</tbody>
</table>

The etching time ranged between 7 and 15 seconds depending on the freshness of the solution.

6- Step 3 was repeated very carefully to eliminate tarnishing caused by the etching. Final washing and drying were the same as in step 4.

3.5.2 Replication

Replicas were prepared from the original specimen exterior surface to examine the details of the slip bands, and from the etched interior planes to determine the dislocation microstructures. Two stage replicas were prepared by the following technique.

A dilute solution (2%) of the replicating material (parlodion), was made in an organic solvent (ethyl acetate). This solution was placed on the surface to be investigated. The solvent evaporated leaving a thin film of the replicating material over the surface. To strip off the replica, an electron microscope copper grid was placed on a piece of adhesive tape (Sellotape) with a small piece of lens tissue between the grid and the gum of the tape. This was placed so that about one quarter of the grid stuck lightly to the
Figure 3.6  

A Different layers of a replica. 
B Field on the microscope screen 
C Intensity along the screen
Figure 3.7 The vacuum evaporation unit

1- Gold 40% palladium loop
2- Carbon electrodes
3- Spring
4- Specimen
gum of the adhesive tape, and three quarters was over the lens tissue. The adhesive tape with the grid was pressed down over the replica and then the tape was pulled off, stripping the replica from the specimen. The adhesive tape was then stretched gum side up and was fixed to a clean glass slide as shown in figure 3.6. The replica was shadowed with both carbon and gold 40% palladium simultaneously as shown schematically in figure 3.7. After shadowing the replica of the surface, stretched over the glass slide, could be examined on the optical microscope.

For electron microscopy, several needle holes were perforated very close to the grid circumference which was in contact with the lens tissue. A scalpel was used to make a square cut around the grid and the lens tissue. The whole square was picked up with a pair of tweezers and placed over several layers of filter paper saturated with amyl acetate. The thin evaporated film was in the upper position while the non-adhesive side of the tape was in contact with the filter paper. The solvent was absorbed up through the holes so that it dissolved the gum of the tape and the replicating material leaving the thin evaporated carbon-gold-palladium film over the copper grid. After two minutes the grid was picked up with a pair of tweezers and placed over a new piece of filter paper to dry. Then the grid was transferred back to the filter paper saturated with the solvent for two minutes and again dried in the same way. This cycle was repeated several times to insure complete elimination of the parlodian replicating material.

The grid, to which the thin carbon-gold-palladium evaporated film adhered was finally ready to be inserted in the electron microscope holder.

3.5.3 Transmission Electron Microscopy

Thin sections were cut with the EDM with an initial thickness
Figure 3.8 Dishing apparatus

Figure 3.9 Final thinning apparatus
of about 0.02 inch. This thickness was reduced to a thickness ranging between 0.009 - 0.012 inch, by bright dipping in the following solution (Thomas, 1962)

<table>
<thead>
<tr>
<th>Acid</th>
<th>Parts by Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitric acid</td>
<td>50</td>
</tr>
<tr>
<td>Orthophosphoric acid</td>
<td>25 &quot;&quot;</td>
</tr>
<tr>
<td>Glacial acetic acid</td>
<td>25 &quot;&quot;</td>
</tr>
</tbody>
</table>

After the initial thinning, the specimens were washed in distilled water and dished using the jet technique shown in figure 3.8. The electrolyte solution was 20% orthophosphoric acid. After the dishing, the specimens were washed thoroughly in distilled water and dried with a piece of filter paper. The final thinning was done by electropolishing in a 50% orthophosphoric acid at 1.6 volts at -20°C. A strong light source was located behind the specimen and the specimen was watched carefully through a large magnifying glass as shown in figure 3.9. As soon as a hole was observed, the specimen was taken out of the solution and washed twice in distilled water and twice in ethanol. It was then dried with a piece of filter paper.

A very thin area around the hole with relatively thick edges made the specimen suitable for electron transmission and very easy to handle during insertion in the electron microscope holder.

3.6 Analysis of probable errors

In this investigation, three different functions have been calculated from data obtained by experiment. These functions are the resolved shear stress (τ), the resolved shear strain (γ), and the cyclic hardening rate (τ'). In this section, the probable error in each function is calculated from the probable errors encountered in the experimental measurements.
3.6.1 Resolved Shear Stress

The equation used to compute the resolved shear stress is as follows:

\[
\tau = \frac{F}{A} \cos \theta \cos \lambda \quad \ldots \ldots \ldots \ldots (3.1)
\]

\[
\tau = \frac{X.M}{A} \cos \theta \cos \lambda \quad \ldots \ldots \ldots \ldots (3.2)
\]

Where:
- \( \tau \) Resolved shear stress
- \( F \) Load
- \( A \) Cross sectional area
- \( \theta \) Angle between the slip plane normal and tensile axis
- \( \lambda \) Angle between the slip direction and tensile axis
- \( X \) Distance on the chart
- \( M \) Calibration constant

Taking the first derivative of 3.2 and dividing by \( \tau \)

\[
\frac{\delta \tau}{\tau} = \frac{\delta X}{X} + \frac{\delta N}{N} + \frac{\delta A}{A} + \frac{\delta \cos \theta}{\cos \theta} + \frac{\delta \cos \lambda}{\cos \lambda} \quad (3.3)
\]

All terms of the R.H.S. of equation 3.3 are listed in table 3.2.

Notice that the value of \( \frac{\delta X}{X} \) listed in the table is an average value since it increases as the stress is decreased. By adding these terms, the probable error in the calculated resolved shear stress is equal to 9.24%. This value is the percentage error in resolved shear stresses of specimens A and B. The probable error for specimens A and B is 9.43%.

3.6.2 Resolved Shear Strain

The equation used to compute the resolved shear strain is as follows

\[
\gamma = \frac{\epsilon}{\cos \theta \cos \lambda} = \frac{\Delta L}{L \cos \theta \cos \lambda} \quad \ldots \ldots \ldots \ldots (3.4)
\]

\[
\gamma = \frac{X.M}{L \cos \theta \cos \lambda} \quad \ldots \ldots \ldots \ldots (3.5)
\]

Where
- \( \gamma \) Resolved shear strain
- \( \epsilon \) Normal strain
- \( \Delta L \) Normal extension
- \( L \) Gage length
- \( X \) Distance on the chart
- \( M \) Calibration constant
<table>
<thead>
<tr>
<th>Error</th>
<th>$B$ and  $\overline{B}$</th>
<th>$A$ and  $\overline{A}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\frac{3x}{x}$</td>
<td>0.0143</td>
<td>0.0143</td>
</tr>
<tr>
<td>$\frac{3N}{N}$</td>
<td>0.005</td>
<td>0.005</td>
</tr>
<tr>
<td>$\frac{3A}{A}$</td>
<td>0.0013</td>
<td>0.0013</td>
</tr>
<tr>
<td>$\frac{3 \cos \theta}{\cos \beta}$</td>
<td>0.0465</td>
<td>0.055</td>
</tr>
<tr>
<td>$\frac{3 \cos \lambda}{\cos \lambda}$</td>
<td>0.0253</td>
<td>0.0187</td>
</tr>
<tr>
<td>$\frac{3 \tau}{\tau}$</td>
<td>0.0924</td>
<td>0.0943</td>
</tr>
</tbody>
</table>

Table 3.2 The Probable Errors in Calculating $\tau$ for Specimens B, B, A and A

*This is due to a probable error in measuring $\theta$ and $\lambda$ that is equal to $2^\circ$.

<table>
<thead>
<tr>
<th>Error</th>
<th>$B$</th>
<th>$\overline{B}$</th>
<th>$A$</th>
<th>$\overline{A}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\frac{3\tau}{\tau}$</td>
<td>0.0924</td>
<td>0.0924</td>
<td>0.0943</td>
<td>0.0943</td>
</tr>
<tr>
<td>$\frac{3\gamma}{\gamma}$</td>
<td>0.1025</td>
<td>0.0968</td>
<td>0.105</td>
<td>0.1000</td>
</tr>
<tr>
<td>$\frac{3(3\tau/3N)}{(3\tau/3N)}$</td>
<td>0.1949</td>
<td>0.1892</td>
<td>0.1995</td>
<td>0.1943</td>
</tr>
</tbody>
</table>

Table 3.3 The Minimum Probable Errors in Calculating $\frac{3x}{3N}$ for Specimens A, A, B and B
Taking the first derivative of equation 3.5 and dividing by $\gamma$

$$\frac{\partial \gamma}{\gamma} = \frac{\partial x}{x} + \frac{\partial M}{M} + \frac{\partial L_0}{L_0} + \frac{\partial \cos \theta}{\cos \theta} + \frac{\partial \cos \lambda}{\cos \lambda}$$  \hspace{1cm} (3.6)

The strain gage extensometer has been calibrated with a micrometer that is accurate to 0.00002 inch. The conversion factor was such that 2 inches on the chart corresponded to 0.001 inch extension. The probable error $\partial M/M$ can be reasonably assumed to be nil. The error $\partial X/X$ is 0.0107 and 0.005 for specimens B and $\bar{B}$ respectively. The error $\partial L_0/L_0$ is estimated to be 0.02. By taking the values of $\partial \cos \theta/\cos \theta$ and $\partial \cos \lambda/\cos \lambda$ from table 3.2, and adding them to the probable errors $\partial X/X$ and $\partial L_0/L_0$, the probable errors in the calculated resolved shear strain will be 0.1025 and 0.0968 for specimens B and $\bar{B}$ respectively. For specimens A and $\bar{A}$ the probable errors are 0.105 and 0.1 respectively.

3.6.3 Cyclic Hardening Rate $\tau = \frac{\partial \tau}{\partial 2\gamma}$

The cyclic hardening rate $\partial \tau/\partial H$ per unit cumulative shear strain has been calculated by dividing the slope of the hardening curve by twice the resolved shear strain ($2\gamma$). This includes two considerable sources of errors, $\partial \tau/\tau$ and $\partial \gamma/\gamma$. Table 3.3 shows these errors and the corresponding minimum probable errors in $\partial \tau/\partial H$ for crystals $A$, $\bar{A}$, $B$ and $\bar{B}$ calculated by simply adding equations (3.3) and (3.6). The slope was measured by drawing a tangent to the hardening curve. This involves a third source of error which is the exact position of the tangent. By measuring the slope of the two tangents drawn on the two possible extreme positions, the error was found to be less than 15%. This error would eventually decrease to zero when the slope reaches zero.
IV RESULTS AND OBSERVATIONS

4.1 Test Program

Four single crystals with two different orientations were fatigued at two different strain amplitudes. Specimens A and $\bar{A}$ had their stress axes at orientation A on the stereographic projections, figures 4.1A and B. The stress axes of specimens B and $\bar{B}$ is marked A, figures 4.2 A and B. Low strain amplitude specimens A and B were cyclically fatigued at $\pm 0.0037$ and 0.0039, respectively, while high strain amplitude specimens $\bar{A}$ and $\bar{B}$ were cyclically fatigued at $\pm 0.0051$ and $\pm 0.0070$ respectively. This test program provides a study of the effect of strain amplitude and secondary slip on the cyclic hardening rate, the type of surface damage and the saturation dislocation structure.

The cyclic hardening curves for all crystals were plotted individually, and for comparison the cyclic hardening rate curves for the different specimens were calculated.

Exterior markings were observed and photographed at planes F and C, figures 4.1 and 4.2. Interior planes P and Q of specimens B and $\bar{B}$, and planes P and X of specimens A and $\bar{A}$ were etched to reveal the dislocation structure. Thin foils parallel to planes P and X of specimen A and plane Q of specimen $\bar{B}$ were prepared and investigated by the transmission microscopy technique to ensure the validity of the etching technique.

4.2 Results of Cyclic Hardening

The cyclic hardening curves for the four crystals have been drawn by plotting the peak stress versus the corresponding number of cycles in figures 4.3, 4.4, 4.5 and 4.6.

From these four figures it is obvious that specimen A ($\gamma = 0.0037$) took
Figure 4.1a Standard projection of crystals $\Lambda$ and $\bar{\Lambda}$
Figure 4.1b  Axis projection of crystals A and A
Figure 4.2 a  Standard projection of crystals B and \( \bar{B} \)
Figure 4.2b  Axis projection of crystals B and B
Figure 4.4 Cyclic hardening curve of crystal $\bar{A}$
Figure 4.7 Cyclic hardening rate for crystals A, A', B, and B'.
about 660 cycles, and 4.82 cumulative shear strain before reaching saturation at a stress of 3 kg/mm². Specimen A (γ = ±0.006) took about 130 cycles, and 1.57 cumulative shear strain before reaching saturation, with a 3.45 kg/mm² saturation stress.

Specimen B (γ = ±0.009) which showed considerable conjugate slip, took about 220 cycles, and 1.70 cumulative shear strain before reaching saturation, at 3.7 kg/mm². Specimen B (γ = ±0.007) saturated after about 90 cycles and 1.25 cumulative shear strain, at a stress equal to 5 kg/mm².

To show the relation between the cyclic hardening rate and the cumulative shear strain, the slope of the cyclic hardening curve has been computed at different intervals.

Figure 4.7 shows that the cyclic hardening rate curve of specimen A was markedly different from specimens A, B, and B.

Specimen A had the lowest initial cyclic hardening rate (2.1 kg/mm²), while specimen B had the highest cyclic hardening rate (18 kg/mm²). The rate of decrease in cyclic hardening rate was low and smooth for specimen A, while for specimens A, B, and B the rate decreased sharply during the early cycles of the test. At certain points X₁, X₂, and X₃ the cyclic hardening rate of specimen A was equal to that of specimens B, A, and B respectively. Beyond these points the hardening rates of specimens B, A, and B were less than that of specimen A, which shows that they will reach saturation earlier.

The results for the four crystals are summarized in table 4.1.

4.3 Surface and Interior Structures

The surface slip line distribution and dislocation microstructures of each specimen differed markedly from those of the other three specimens. However, for each specimen considered individually, obvious correlations did exist between the observed external slip bands and the internal dislocation microstructure. For this reason, all of the visual observations
<table>
<thead>
<tr>
<th>Specimen</th>
<th>$\gamma$</th>
<th>Orientation</th>
<th>Initial $\tau$ kg/mm$^2$</th>
<th>Saturation $\tau$ kg/mm$^2$</th>
<th>Cycles to Saturation</th>
<th>Initial Hardening Rate kg/mm$^2$</th>
</tr>
</thead>
</table>
| A        | 0.37    | ![Diagram](image)
| A        | 0.61    | ![Diagram](image)
| B        | 0.39    | ![Diagram](image)
| B        | 0.7     | ![Diagram](image)

Table 4.1 Fatigue Hardening Results
for a given specimen are presented together in the following subsections.

4.3.1 Low Amplitude Cyclic Fatigue

Specimen A

Surface markings of specimen A have been recorded after about 8000 cycles. As shown in figure 4.8, the surface markings are in the form of persistent slip bands that are grouped into bundles that are roughly parallel to the primary slip plane. By following the trace of the bundles their crystallographic plane could be located. \( \text{A}_b \) is the pole of their plane plotted on the stereographic projections, figures 4.1 a and b. Figure 4.9 shows at high magnification a shadowed replica taken from the same surface. Most of the slip lines in the bundle shown have formed intrusions.

As shown in figure 4.10 a the dislocation structure along the primary slip plane \( (P) \) is mainly in the form of long walls of high density dislocations. In this section the walls are mainly parallel to the \([121]\) direction, which is normal to the Burgers vector. The average spacing as measured from figure 4.10 a is 1.3 u. Figure 4.10 b shows the same type of structure by transmission electron microscopy.

Figure 4.11 is a low magnification micrograph of the etched plane marked X in the stereographic projection, figure 4.1 a. The inset shows the corresponding surface markings of the same crystal at plane C in figure 4.1 a. The correlation between the surface and interior structure is obvious. As shown in figure 4.11, the structure is mainly composed of layers (average thickness 18 u) that are roughly parallel to the trace of the primary slip plane. The pole of the crystallographic plane parallel to the layer is marked \( \text{A}_b \) in figure 4.1 a. As shown in figure 4.12 the detailed structure of the layers is mainly in the form of short walls that are roughly parallel or normal to the trace of the primary slip plane. The boundaries between the layers are well defined by
4.8 Surface markings of surface C, crystal A, 200X

4.9 Shadowed replica showing intrusions, crystal A, 6000X
4.10 a  The etched primary slip plane, crystal A, 3600X

4.10 b  Dislocation microstructure along the primary slip plane crystal A, 4000X
4.11 The etched X plane (main figure, 480X) and surface markings (inset 200X) crystal A.
4.12 The etched X plane, at high magnification, crystal A, 800x
4.13 Dislocation microstructure along the plane X, crystal A, 14,500 X
elongated cells that extend parallel to the layers. Figures 4.13 a and b show the same type of structure in transmission electron microscopy.

By investigating the dislocation structure on two different planes \( \mathbf{P} \) and \( \mathbf{X} \), the overall structure of specimen A could be described as a mixture of long narrow walls and mats extending to the \([12\overline{1}]\) direction; with mats parallel to the primary slip plane and the walls parallel to the \((\overline{1}01)\) plane.

Only minor differences existed between the microstructure of the active persistent slip bands and the much less active matrix regions. Both were comprised of the wall-mat structure. The only indication of the strain inhomogeneity was that the relatively uniform structure was divided into slats roughly parallel to the primary slip plane by sharp boundaries in the form of elongated cells.

Specimen B

Figure 4.1b shows the surface markings taken from plane \( \mathbf{F} \) at the end of the cyclic hardening period of fatigue. As shown in the figure the slip lines were uniformly distributed over the specimen surface. At the end of the test (9000 cycles), and as seen in figure 4.15, the uniform distribution of slip lines has been broken into bundles that are parallel to the crystallographic plane marked \( B_p \) in figure 4.2. The average thickness of each bundle is 16 \( \mu \).

Besides the persistent slip bands, other phenomena have been observed. Figure 4.16 shows slip traces on the conjugate slip system. Kinked deformation bands have been observed frequently, figure 4.17. Intrusions have been observed more frequently than extrusions, and figure 4.18 shows the details of a single persistent slip band. In some regions where the slip bands are uniformly distributed, long ripples have been observed, figure 4.19. The pole of the plane of these ripples could not be located accurately on the stereographic projection because they were observed only on one side of the specimen.
4.14 Surface markings near saturation of surface F, crystal B, 100X

4.15 Surface markings of surface C, crystal B, 100X
4.16 Traces of slip along the conjugate system, crystal B, 5000X

4.17 Kinded deformation band, crystal B, 30,000X
4.18 The details of a persistent slip band, crystal B, 30,000X

4.19 Rows of ripples, crystal B, 200X
However, they are about 33° off the trace of the primary slip plane in figure 4.19.

Figure 4.20 is a shadowed replica of the etched primary slip plane P showing that, on this plane, the dislocation structure is mainly in the form of long walls of high density dislocation. These walls are mainly parallel to the [121] direction, which is normal to Burgers vector. The average spacing between these walls as measured from figure 4.20 is 1.6 µ. At the area marked A in figure 4.20, the pattern of walls is not as distinctively defined as it is in the rest of the figure.

Figure 4.21 is a micrograph of the etched cross slip plane Q. As seen from the figure, the structure is mainly in the form of layers that are roughly parallel to the trace of the primary slip plane. Notice the matching between the etched surface (main plate) and the surface markings (inset). The average thickness of the layers as measured from figure 4.21 is 17 µ.

At higher magnification the detailed structure is shown by a shadowed replica in figure 4.22. Two distinctive types of dislocation microstructure are evident. The first has the form of short walls that extend nearly normal to the trace of the primary slip plane, which will be referred to as region 1. The second structure is a mixture of short walls that are normal or parallel to the trace of the primary slip plane, region. In some areas the boundaries between the two types are sharply defined by cells elongated in the direction of the layers, (R.H.S. of figure 4.22), but they are poorly defined in other areas (L.H.S. of figure 4.22).

From the investigation along both the primary and the cross slip planes, the overall structures of specimen B is divided into two types of structure. The first has the form of long narrow walls, extending along the [121] direction and laying nearly parallel to the (101) plane. The second is a mixture of long
4.20 The etched primary slip plane, crystal B, 1400X
4.21 The etched cross slip plane (main figure 480X) and the surface markings (inset, 100X) crystal B.
4.22 The etched cross slip plane at high magnification, crystal B, 1000X
narrow walls that are extended parallel to the \( \overline{121} \) direction, and lay nearly parallel to the primary or to the \((101)\) plane.

From the relative width of the two types of regions in figure 4.20, one can conclude that the simple wall structure represents the interbundle matrix regions while the more complex wall-mat structure is to be associated with the persistent slip bands. Once again however, the two structures are more noteworthy for their similarity than for their differences.

4.3.2 High Amplitude Cyclic Fatigue

Specimen B

The surface markings have the same general appearance as those of specimen B. The dark areas in figures 4.23 a and b are persistent slip bands grouped into bundles that extend parallel to the crystallographic plane \( B \), shown on the stereographic projections in figure 4.2. Notice on figure 4.23 a that a considerable amount of conjugate slip is developed in regions between the concentration of the persistent slip bands into bundles. Intrusions and extrusions have been observed, figures 4.24 a and b, but no clear relation between the amplitude and their size or intensity could be found.

Figures 4.25 a and b represent the etched primary slip plane at two different magnifications. On this plane the structure is a mixture of long walls extending parallel to the \([\overline{121}]\) direction, and closed cells that are slightly elongated parallel to the walls. The average spacing between the walls is 1 \( \mu \). The average longitudinal dimension of the cells is 8 \( \mu \).

Figure 4.26 shows the etched surface of the cross slip plane, and the inset shows the surface markings. The layers have a distinct wavy form, and the overall direction of the layers is approximately 90° from the trace of the primary slip plane.
4.23 a Surface markings of surface F, crystal B, 200X

4.23 b Surface markings of surface C, crystal B, 200X
4.24 Intrusion and extrusion, crystal B, 30,000X
4.25 a The etched primary slip plane, crystal $\overline{B}$, 1100X

4.25 b The etched primary slip plane, crystal $\overline{B}$, 4000X
4.26 The etched cross slip plane (main figure 480X) and the surface markings (inset, 200X) crystal B.
In the detailed pattern of the dislocation structure shown in figure 4.27, two distinctive types of structure can be recognized. The first is a mixture of short walls that are parallel or normal to the trace of the primary slip plane. Figures 4.28 a and b are transmission electron micrographs, showing the same type of structure. The second type cannot be resolved by the etching technique, but the transmission electron technique reveals that it consists of the irregular closed cells shown in figure 4.29.

Due to the mixed microstructure observed along the primary slip plane, it would be difficult to describe unequivocally the nature of the microstructure in three dimensions. However, it possible that the microstructure is mainly in the form of wall-mat microstructure and three dimensional cellular structure.

Specimen A

In the previous three cases, the persistent slip bands were grouped into bundles nearly parallel to the primary slip plane. In specimen A the persistent slip bands are grouped into bundles, but in this case the bundles are nearly normal to the primary slip plane (Pole $h_0$ on the stereographic projection figure 1.1). Figures 4.30 a and b are the surface markings taken from the two surfaces C and F. The dark areas are the persistent slip bands. Figure 4.31 a shadowed replica from surface C, shows the difference between the regions where the persistent slip bands are concentrated and the inactive region.

Figure 4.32 shows the etched primary slip plane P and the inset represents the surface markings from surface F of the same specimen. Figure 4.33 is a shadowed replica of the etched primary slip plane. As seen from the figure, two different types of structure are recognizable. Region 1 is a clearly defined structure dominated by long walls extending parallel to the $\overline{12}1$ direction. Within these walls closed cells, regular or slightly elongated in $\overline{12}1$ direction, are frequently observed. The average spacing and the cell size
4.27 The etched cross slip plane at high magnification, crystal B, 1300X
4.28 Dislocation microstructure along the cross slip plane, crystal B
4.29 Dislocation microstructure along the cross slip plane, crystal $\overline{B}$, 16,000X
4.30 a Surface markings of surface F, crystal \( \overline{A} \), 200X

4.30 b Surface markings of surface C, crystal \( \overline{A} \), 100X
4.31 Shadowed replica of the surface markings, crystal $\bar{A}$.
4.32 The etched primary slip plane (main figure, 480X) and surface markings (inset, 200X) crystal $\overline{A}$. 
4.33 The etched primary slip plane at high magnification, crystal $\overline{A}$, 1200X

4.34 The etched X plane, crystal $\overline{A}$, 900X
measured from figure 4.33 is 1.4 microns. The other type of structure observed on the same plane is an indistinctive cellular structure, (region 2).

Figure 4.34 shows the etched X plane of the same specimen. The pattern is nearly the same as that of figure 4.33. Region 1 is again mainly composed of long walls that are normal to the trace of the primary slip plane. The unresolvable region 2 could have a cellular type structure.

From both the primary and the X planes of specimen A, it can be concluded that the crystal developed into layers that are nearly normal to the primary slip plane. The structure of the layers changes alternately from clear walls parallel to the (101) plane, to an indistinctive three dimensional structure. As in specimen B, equiaxed cells are found within the clearly defined walled regions.
4.4 Summary of Results

1- Crystal A which was fatigued at low amplitude and did not undergo any secondary slip had the lowest initial cyclic hardening rate, the lowest saturation stress, and the largest cumulative shear strain before reaching saturation. Crystal B which was fatigued at high amplitude and underwent considerable amount of secondary slip, had the highest initial cyclic hardening rate, the highest saturation stress, and the smallest cumulative shear strain before reaching saturation.

2- The surface markings of all crystals were mainly in the form of persistent slip bands grouped in bundles. These bundles were roughly parallel to the primary slip plane for crystals A, B, and B, but they were normal to it for crystal A.

3- The dislocation microstructure differed from one crystal to another, but a strong correlation was found between the microstructure and the surface manifestation for each individual crystal.

4- The low amplitude microstructure consisted mainly of long narrow walls roughly parallel to the (101) plane, and mats parallel to the primary slip plane. The difference in microstructure between the inactive and the active regions was not distinctive. At high amplitude the distinction between the two regions was clear, changing alternately from indistinctive equiaxed cells to the wall-mat microstructure.
V DISCUSSION

5.1 Introduction

The basic purpose of these experiments was to elucidate the difference in dislocation microstructure between the persistent slip band bundles and the interbundle matrix regions. The experiments were carried out very carefully and were conducted under the simplest possible test conditions; single crystals of a pure and simple metal tested uniaxially at constant plastic strain. The specimens all formed bands of concentrated strain in which intrusions were nucleated, but the microstructures of all four specimens were different and the microstructures of the current tests differed from those previously reported by other workers. One can only conclude that the tendency to go to an inhomogeneous strain mode is very basic to metals in a reversing stress field. The microstructure formed appears to be only a manifestation of the particular test conditions for each individual specimen.

The current results indicate that strain amplitude and the crystal orientation and the mode of instability will affect the microstructure produced. Other work shows that stacking fault energy and test frequency are also important factors. Unfortunately, the experimental procedure necessary to relate internal dislocation arrangements to their surface manifestation is slow, tedious and very involved so that cataloguing of fatigue microstructures in terms of the important parameters is not likely to be accomplished soon. Even in the present work we have only one specimen for each test condition, and we cannot assume that it represents a unique mode of fatigue for the prescribed test constraints. Again, the length of time required to get results makes it impractical to do duplicate tests. Therefore it is necessary to assume
that the current results, considered in the context of all the previously reported work, are representative of the prescribed test conditions. With these reservations, provisos and assumptions, the following generalizations and observations can be delineated.

5.2 Cyclic Hardening

5.2.1 Effect of Strain Amplitude

As shown in figure 4.7 and table 4.1, the initial cyclic hardening rate of crystals $\overline{A}$ and $\overline{B}$ which were fatigued at the high amplitude range, was higher than that of crystals $A$ and $B$ fatigued at the low amplitude range. The saturation stress for crystals $\overline{A}$ and $\overline{B}$ was higher than that the saturation stress of crystals $A$ and $B$ respectively. Crystals $\overline{A}$ and $\overline{B}$ reached saturation with a cumulative shear strain less than that of crystals $A$ and $B$ respectively.

These results are in agreement with previous results reported by a number of workers.

Wood and Segall (1957), reported that in the higher amplitude ranges the specimen saturated at slightly larger cumulative shear strain contradicting the current results. This discrepancy could be mainly due to difference in testing procedures. In their work they investigated copper specimens fatigued in reverse twist, so the strain varies from a maximum at the surface to zero in the center. Besides the strain gradient effect, Ronay (1966) showed that as cycling proceeds longitudinal compressive stresses build up in the specimen. This was proved by testing titanium tubes in torsion, (MacDonald and Wood, 1972).

From the current results, it can be concluded that higher amplitude cyclic

Kensley and Paterson (1960), Snowden (1963), Ebner and Backofen (1959), Alden and Backofen (1961), and Watt et al. (1967)
fatigue for crystals of the same orientation leads to:

1- higher initial cyclic hardening rate
2- higher saturation stress
3- less cumulative shear strain before reaching saturation.

5.2.2 Effect of Secondary Slip

As shown in figure 4.7 and table 4.1, the initial cyclic hardening rate of crystals B and $\overline{B}$, which underwent a considerable amount of conjugate slip, was higher than that of crystals A and $\overline{A}$. The saturation stress for crystals B and $\overline{B}$ was higher than that of crystals A and $\overline{A}$ respectively. Also crystals B and $\overline{B}$ reached saturation with a cumulative shear strain less than that of crystals A and $\overline{A}$.

These results are in agreement with previous results reported by Kemsley and Paterson (1960), Snowden (1963), and Wadsworth (1963).

From the current results, it can be concluded that crystals that undergo more slip on a secondary system but are fatigued at the same strain amplitude result in:

1- higher initial cyclic hardening rate
2- higher saturation stress
3- less cumulative shear strain before reaching saturation.

From the combined effect of amplitude and slip on a secondary system, it has been shown that crystals cyclically fatigued at higher amplitude and which have considerable slip on a secondary system result in the highest cyclic hardening rate, the highest saturation stress, and the lowest cumulative shear strain before reaching saturation. Also the introduction of more strain on secondary systems has the same effect on the hardening rate as introducing more strain on the primary system.
An explanation for this is that higher strain amplitude leads to a higher density of obstacles at the end of the compressive path, because the crystal undergoes more cumulative shear strain. This subsequently leads to a higher tensile peak stress to reach the same strain amplitude on the following tensile path. The presence of slip dislocations on a secondary system will interfere with the main slip process along the primary slip plane resulting in a higher resolved shear stress to reach the same strain.

5.3 Surface Damage

5.3.1 Development of Surface Damage

The surface damage observations reported in the current work are in agreement with many previously reported results. The uniform distribution of slip lines in the presaturation period, figure 4.14, has been reported before by Alden and Backofen (1963) in bending specimen, and Watt et al. (1967) and Basinski et al. (1969) for uniaxial tests. Basinski et al. (1969) showed that in the hardening region the uniform distribution of slip lines on the surface corresponded to a more-or-less homogeneous dislocation microstructure consisting of one dimensional braids of dipoles extended basically in the [\( \overline{121} \)] direction. At the end of the current tests (after 8000 cycles), the uniform distribution of slip lines had been broken into regions (bundles) where the deformation was heavily concentrated.

For specimens A, B and \( \overline{B} \) these regions were roughly parallel to the primary slip plane. This agrees with most previous reports. In specimen \( \overline{A} \), the regions were roughly normal to the primary slip plane. Similar behavior was reported by Gostelow (1971) and Woo (1972).

This difference in surface behavior shows that the uniform dislocation microstructure in the presaturation period can break up into hard and soft regions roughly normal to the primary slip plane as well as roughly parallel.
5.3.2 Effect of Strain Amplitude

For crystal B which has been cyclically fatigued at the higher strain amplitude, the spacing between the individual slip bands was smaller than that of crystal B which has been cyclically fatigued at the lower strain amplitude. The average thickness of the bundles was smaller for crystal B than for crystal B. This shows that higher amplitude fatigue tends to narrow down the regions where deformation is concentrated.

No clear relation was found between the nature of intrusions and the strain amplitude. Intrusions ranging in length from 1 to 30 μ were observed at all amplitudes.

5.3.3 Effect of Secondary Slip

The surface markings of crystals A, A, B and B, showed that crystals B and B underwent a considerable amount of conjugate slip, while crystals A and A did not.

For crystal A, the surface markings were mainly the usual slip bands that are parallel to the trace of the primary slip plane. For crystal B other phenomena have been observed. Beside slip traces parallel to the trace of the conjugate slip plane, kinked bands and ripples have been observed. The last phenomenon (ripples) has been reported for fatigued Al polycrystals by King and Teer. (1969). They also saw crack nuclei within these ripples. Similar behavior was reported by Arnell and Teer (1969). They investigated the effect of grain orientation on the nature of surface markings, (section 2.3). They showed that as the the stress axis of the grain moves away from the center of the standard triangle toward its corners ripples will increase* until they

* The same effect is shown in figure 4.19 of crystal B, which has its orientation 180° away from [100].
dominate the grain surface when the axis reaches the [100] orientation.

As seen in figure 4.30, the slip along the conjugate system is concentrated in regions where the persistent slip bands are absent. The reason for this could be the higher density of forest dislocations intersecting the conjugate plane due to the concentrated slip along the primary plane within the persistent slip bands regions.

5.4 Dislocation Microstructure

5.4.1 The low Amplitude Microstructure

For the two lower amplitude tests, the structure of the layers representing the active strain regions and those associated with the inactive matrix regions were basically the same. All layers were primarily composed of high dislocation density mats lying parallel to the primary slip plane and walls roughly perpendicular to the primary burgers vector. This similarity between the layers would explain why Watt (1967) could not reveal any difference between the matrix and the persistent slip band structures by a crude etching technique.

The existence of this structure both in the intense slip bands and in the inactive regions has not been previously reported, although similar structures have been observed. Pratt, figure 2.14, called this structure elongated cells. Sastri et al. (1972) have observed the structure throughout fatigued single crystals of copper containing Al₂O₃ particles. Watt et al. (1970) also observed a structure containing extensive areas of regularly spaced walls, figure 2.12 at low amplitude but other types of structure, namely dipole braids and equiaxed cells were also seen. Stobbs et al. (1971) have also seen these walls in copper single crystals containing a dispersion of SiO₂ particles.

It is somewhat surprising that the markedly different fatigue hardening rate of specimen A was not manifested in the microstructure developed. The
difference in hardening rate was presumably caused by the fact that only one
slip system was highly stressed, but the microstructure formed is very similar
to that of specimen B which had active slip on at least two systems.

The two most extensive studies of persistent slip bands microstructures
have been those of Laufer and Roberts* and by Klesnil, Lukas and coworkers**.
Laufer (1969) sees the sequence of persistent slip bands development as a
three stage process:

1- The matrix structure develops as a one dimensional braid structure.
2- At some points the matrix structure becomes unstable and the braids
collapse into multipole walls, which propagate into the matrix forming
a ladder structure.
3- The walls become unstable and break up into cells forming a
sandwich structure of a row of cells, bordered by rows of walls set in
a braid matrix.

The walls described by Laufer and Roberts are identical to the walls
seen in the present work. The most striking difference between their work and
the present work is the retention of the braid structure as the matrix. Lukas,
Klesnil, and Krejci (1968), fatiguing copper crystals of the same orientation
also formed a persistent slip bands structure consisting of walls and some cells
set in a matrix structure consisting of heavy braids. Both Laufer and Lukas
used high frequency test machines; 30 Hz and 105 Hz respectively. Watt (1967)
showed that the width of the hysteresis loop, that is the plastic strain per cycle
is orders of magnitude lower for high frequency tests than for a low
fatigue test. This difference can be explained in terms of the effect of strain

* Laufer and Roberts (1964), (1966), Laufer (1969)
et al. (1968)
rate on the thermal activation of dislocation motion. The lower plastic strain required in the high frequency test may explain why the braid structure did not develop into walls in the high frequency tests.

A weak point about the results obtained by Laufer and Roberts and Lukas et al. is that they only used the transmission electron microscopy on foils and so were only able to examine relatively small areas. With the etching technique used in the current work, whole cross-sections of the specimen could be investigated. A second weakness in their work is that they only fatigued specimens of one orientation. It is interesting that the model invented by Kuhlmann-Wilsdorf, Laufer and Nine to produce cells in fatigue required two highly stressed coplanar primary systems. This requirement was satisfied by the orientation chosen by Laufer and by Lukas but was not fulfilled in the current set of tests.

The most positive aspect about the work of Laufer and of Lukas was that they discovered well defined differences between the matrix and persistent slip bands structures. This provided considerable encouragement for those who hoped to explain why the strain concentrated into intense bands. The current work unfortunately shows that the results of Laufer and Lukas are not generally representative.

5.4.2 The High Amplitude Microstructure

It has been a general observation that as the strain amplitude increases the tendency to form a cellular structure increases; (section 2.5). The present results confirm this trend, but they also show that layers of the low amplitude microstructure are also present in these specimens, so the low amplitude structure appears to play an important role in concentrating the strain.

Specimen B retained slabs of the low amplitude microstructure which lay roughly parallel to the primary slip plane and which were separated by layers
of cells (figure 4.27). For this specimen it is impossible to state unequivocally which microstructure represents the more active regions. However, figure 2.16 (after Laufer and Roberts 1964) and other results reported by MaGrath and Waldron (1964), suggest that the microstructure associated with the more active regions is the wall-mat microstructure.

In specimen A, the alternate layers of low amplitude microstructure and cellular microstructure were roughly perpendicular to the Burgers vector. As discussed in chapter IV, the bands of intense slip are also continuous in the (101) plane. From the relative widths of the active and inactive regions, the low amplitude microstructure appears to carry the intense strain.

There is a striking similarity between the layer microstructure of specimen A and the alternating tidy and untidy regions seen by Gostelow (section 2.5). He used a high voltage electron microscope and was able to view large areas of thin foil and thereby getting a representative picture of the microstructure of his specimens. The tidy regions, consisting of (101) multipole walls and (111) mats similar to the wall-mat microstructure of specimens A and B, also contained walls containing the [21] direction at about 45° from the slip plane. His untidy regions were mainly small well-defined equiaxed cells. The coincidence of the similar alternating layers in these two independent studies shows that the breakdown of the fatigued structure into plastic slabs perpendicular to the Burgers vector is not anomalous, but rather represents an alternative mode of accommodating bands of intense plastic strain.

5.5 The Delineation of Three Dimensional Structures from Plane Sections.

No technique has yet been devised for viewing thick sections of dislocation microstructure, so one must rely on the largely intuitive matching of two dimensional sections to recreate the space pattern of the structures. As yet no one has succeeded in taking two sections through the same cell or
other dislocation array, so that one must always assume that the sections one views are representative. In the present work, complete cross-sections of the specimen were polished, etched and examined, so that one can be confident that they were representative. The problems inherent in matching the views were made easier in the high amplitude case by the very different etching characteristics of the two type of structure, and in the low amplitude case by the fact that only one basic type of structure existed. The apparent objection that the primary plane cut may have only sectioned one of the layers in a specimen like B is not valid. The polishing technique consists of rubbing the specimen over a cloth and this imparts a crown to the specimen so that by the time the surface is etched the originally plane section is in fact a gentle curve which would have intersected many layers in a specimen like B.

One thing the current study shows is how little information about fatigued structures obtained from foils cut parallel to the primary slip plane. The braid structure, the wall structure and Gostelow's tidy structure all have a very similar appearance on the primary plane. The [101] normal slice is also not very informative because the (101) multipole walls are not perfectly straight so that sections through these walls are similar in appearance to braids, as are sections through the other type of walls seen by Gostelow. Unfortunately a number of investigators* have done studies using only these two sections because they are the most convenient foils to do a detailed burgers vector analysis, and one can only get a limited number of slices from a given single crystal. The result of their choice of sectioning is that their efforts shed very little light on the patterns of dislocation arrangements developed in fatigue. The (121) or the cross-slip plane (111) slice is much more informative with regard to substructure development.

* Hancock and Grosskreutz (1968), Shinozaki and Embury (1969) and Woo (1972)
5.6 Effect of Amplitude on Microstructure Size

It is shown in table 5.1 for crystals B and $\overline{B}$ (both having the same orientation) that there is a tendency for the spacing between the sub-boundaries to decrease as the strain amplitude is increased. This was not the case for crystals $A$ and $\overline{A}$. The reason for this could be that crystal $A$ degenerated into a different slip mode, having the bands of intense slip perpendicular to rather than parallel to the primary slip plane. Costelow (1971), whose specimens slipped in a similar mode to $\overline{A}$, found that the sub-boundary spacing was not a function of strain amplitude. On the other hand Stobbs et al. (1970) found that the wall spacing did decrease as amplitude increased for specimens containing SiO$_2$ particles.

Figure 4.10 b, 4.13 a, and 4.13 represent the dislocation microstructure of crystal $A$. It can be seen from the figures that a high portion of the sub-boundaries have an average thickness nearly equal to the spacing between them. For specimen $B$ when the strain amplitude was nearly doubled, the dislocation sub-structure is shown in figures 4.28 a and b and 4.29. In this case the average thickness of the sub-boundaries was much smaller than the spacing between them.

From the previously mentioned results it appears that for specimens that slip in the type $\overline{A}$ mode, the wall spacing is independent of strain amplitude but for specimens which slip like specimen $A$, the wall spacing is a function of strain amplitude and orientation.

5.7 Plastic Flow During Fatigue

Section 2.7 showed that all the dislocation models so far proposed to account for strain during fatigue are wrong or are inadequate. The cell shuttling mechanism proposed by Watt and Ham (1966) was stated in a form that it could not be proved nor disproved by experiment and it was vague about
Table 5.1

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Plastic Strain Amplitude</th>
<th>Sub-boundary Spacing</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.0037</td>
<td>1.3 μ</td>
</tr>
<tr>
<td>A</td>
<td>0.0051</td>
<td>1.4 μ</td>
</tr>
<tr>
<td>B</td>
<td>0.0039</td>
<td>1.6 μ</td>
</tr>
<tr>
<td>B</td>
<td>0.007</td>
<td>1.0 μ</td>
</tr>
</tbody>
</table>

The Effect of strain amplitude on sub-boundary spacing
the detailed interactions of the dislocations and the cell walls. In this section it will be shown that some aspects of fatigue behavior can be understood by a more advanced version of the cell shuttling model.

The fatigued crystal is simplified to a series of relatively neutral multipole walls standing perpendicular to the Burgers vector and having a spacing of about 1.5 μ and a thickness of 0.15 μ. It is assumed that strain is accomplished by dislocations leaving the surfaces of the walls to move across the open cells to the opposite surface of the next wall in the array. Let us first examine these assumptions.

The walls observed in the present work are 1000 to 2000 A thick, and this agrees with previously reported observations. Because of the high dislocation density within the walls it is difficult to resolve individual dislocations, but the walls are estimated to have about 100 to 400 dislocation per micron of height. These have a net Burgers vector very close to zero. This is proven by the lack of any misorientation across the walls, as measured by misorientation contrast or Kikuchi line shift.

To produce a prescribed shear strain γ=0.006 uniformly in the crystal requires the shuttling of an average 24 dislocations per micron of height along the wall. However, the strain in the persistent slip bands is higher than the average. From slip line height measurement by Watt (1967) a reasonable average displacement for a 4 μ thick band would be 400 A or 40 dislocations per micron of height. This represents the sum of the number of dislocations leaving and arriving at the surface of a wall, so according to the density estimate a neutral band of inactive dislocations does exist in each wall.

* Laufer (1964), Lukas et al. (1968) Watt et al. (1970)
One salient feature of the walls not previously understood is that each side of the wall will have a stress field polarity which will reverse every cycle. Consider the wall marked A B in figure 5.1. When the piece of fatigued crystal represented in the figure is subjected to a positive shear stress, then positive mobile dislocations will be pushed to the right and the negative dislocations will move to the left until they reach the next multipole wall. Figure 5.2 shows what this means in reference to the wall itself. Down the left hand side of the wall will be an excess of positive dislocations. Negative dislocations will be predominant on the right hand side of the wall. The walls thus consist of two tilt boundaries of opposite sign with a neutral sandwich of dislocations between them. The difference in polarity could lead to relative displacement of positive and negative dislocations in opposite directions and shearing in the wall itself.

Figure 5.3 is a view of the slip plane as a positive edge dislocation moves from one wall across to the next wall in the persistent slip band. Figure 5.3 a shows a segment of the dislocation of length 1 bowing out into the free space between the walls. Figure 5.3 b illustrates that, once the dislocation has bowed beyond a semi-circle of diameter 1 it will continue to expand without an increase in stress. The dislocation in 5.3 b has been pinned at two points. On the right hand side the line tension force is acting through a very short radius of curvature to pull the dislocation out of the wall. On the left hand side the dislocation is firmly pinned at one point, but the expanding loop is about to annihilate the segment to the left of the pinning point, and so allow continued expansion. Figure 5.3 c shows the simple transfer of the edge dislocation from one wall to the other being accomplished by the running apart of two screw dislocations.

The mechanism that determines the flow stress is thus the stress
Figure 5.1 Movement of positive and negative edge dislocations between the sub-boundaries.

Figure 5.2 Movement of positive and negative edge dislocations within a sub-boundary
Figure 5.3 A possible mechanism for the movement of an edge dislocation from one sub-boundary to another.
required to bow a dislocation segment out of the wall in the presence of the stress field of the wall. The pronounced Baushinger effect means that there is a strong long range stress field aiding the segment bowing. The high density and sharp boundaries of the walls show that there is a short range force inhibiting the bowing. Unfortunately no theoretical analysis nor specific experimental data for these internal stress fields exists at present.
VI. CONCLUSIONS

1. Increasing the strain amplitude results in a higher initial cyclic hardening rate, higher saturation stress and lower cumulative shear strain.

2. Increasing the resolved shear stress on a secondary system has the same effect as increasing the strain amplitude.

3. The breakdown of the dislocation microstructure at the end of the hardening period can be roughly normal as well as parallel to the primary slip plane. This would lead to the concentration of the persistent slip bands in bundles that are roughly normal or parallel to the primary slip plane.

4. The low amplitude microstructure is characterized by the wall-mat microstructure, while the high amplitude microstructure is a combination between the wall-mat and the three dimensional cellular microstructure.

5. Although the general appearance of the microstructure for each individual crystal differed markedly from the other three crystals, strong correlation was found between the surface markings and the interior microstructure proving that the P.S.B. are a manifestation of crystal interior and not just a phenomenon that is restricted to the surface.

6. The etching technique is a very efficient technique for studying microstructures if the dislocations are grouped in high density regions separated by areas which are relatively dislocation free.
A.1 Determination of resolved shear stress

\[ \bar{A} = A/\cos \theta \]

Force in slip direction \[ F = P \cos \lambda \]

R.S.S. \[ \tau = F/\bar{A} = (P/A)\cos \theta \cos \lambda \]

\[ \tau = \sigma \cos \theta \cos \lambda \]

Where:

- \( P/A \) Normal stress
- \( \theta \) Angle between the tensile axis and the slip plane normal
- \( \lambda \) Angle between the tensile axis and the slip direction

Figure A.1. Resolved shear stress (R.S.S.)
A.2 Determination of resolved shear strain

For triangle \( \triangle \text{ABB} \)
\[
\frac{L_u}{L_u} = \frac{\sin \lambda_*}{\sin \lambda_t}
\]

For triangle \( \triangle \text{ABN} \) and \( \triangle \text{ABN} \)
\[
AN = L_u \sin \chi_* = L_t \sin \chi_t
\]

For triangle \( \triangle \text{ABB} \)
\[
\frac{BB}{\sin \lambda_0} = \frac{L_t \sin (\lambda - \lambda_t)}{\sin \lambda_0}
\]

The resolved shear strain
\[
\gamma = \frac{BB}{AN}
\]

\[
\gamma = \frac{L_t}{L_u \sin \chi_*} \left[ \frac{\left( \frac{L_t}{L_u} \right)^2 \sin^2 \lambda - \cos \lambda}{\sin \lambda_0} \right]
\]

(Honeycombe, 1968)

\[
\gamma = \frac{1}{\sin \chi_*} \left[ \sqrt{\frac{(L_u L_t)^2}{L_u} - \sin^2 \lambda - \cos \lambda t} \right]
\]
\[ \gamma = \frac{1}{\sin \lambda} \left[ \sqrt{\frac{L_1^2 + 2\alpha L_x + \alpha L^2}{L^2}} - \sin^2 \lambda - \cos \lambda \right] \]

Since \(4L^2 \to 0\), the term can be neglected

\[ \gamma = \frac{1}{\sin \lambda} \left[ \sqrt{1 + 2\alpha - \sin^2 \lambda} - \cos \lambda \right] \]

\[ (\gamma \sin \lambda + \cos \lambda)^2 = 1 + 2\alpha - \sin^2 \lambda \]

\[ \gamma^2 \sin^2 \lambda + \cos^2 \lambda + 2\gamma \sin \lambda \cos \lambda = 1 + 2\alpha \sin \lambda \]

Since \(\gamma \sin \lambda \to 0\), the term can be neglected. Applying the following trigonometric relation \(\cos \lambda = 1 - \sin^2 \lambda\), the last equation takes the following form

\[ 2\gamma \sin \lambda \cos \lambda = 2\alpha \]

Therefore,

\[ \gamma = \frac{\alpha}{\sin \lambda \cos \lambda} \]

where
- \(\gamma\): Resolved shear strain
- \(\alpha\): Normal strain
- \(\lambda_x\): Angle between slip plane and tensile axis
- \(\lambda\): Angle between slip direction and tensile axis
APPENDIX B

Plastic strain and dislocation displacement (Gilman, 1962)

If an edge dislocation moves a distance $L$, figure B.1 it will produce a surface displacement equal to $b$. Since $b \ll L$, any displacement $x_i$ results in a surface displacement $\delta_i$ equal to $\frac{x_i}{L}$.

The total surface displacement, can be assumed equal to

$$\Delta = \sum \delta_i$$

$$= \frac{b}{L} \sum x_i$$

$$= \frac{b}{L} N \bar{x}$$

$$\gamma = \frac{\Delta}{H}$$

$$= \frac{b}{HL} N \bar{x}$$

$$\gamma = b N \bar{x}$$

Where

- $\gamma$ Shear strain
- $b$ Burgers vector
- $N$ Number of edge dislocations per unit area parallel to $b$ and normal to the primary slip plane
- $\bar{x}$ Average distance travelled by each dislocation

The same argument can be applied if the deformation is carried out by dislocation loops expanding on the primary plane as shown in figure B.2.

$$\gamma = b N A$$

Where

- $N$ Loop density per unit volume
- $A$ Average area swept out by the loop.

In his analysis, Gilman did not take into account that each dislocation is surrounded by a cylindrical region $2D$ in diameter. Outside this
Figure B.1 Relation between plastic strain and dislocation displacement

Figure B.2 Slip by a dislocation loop
cylinder the crystal is assumed to be perfect. In order for any dislocation to contribute to the macroscopic strain, the strain field of the dislocation which is confined by the cylinder should reach the surface. If a dislocation is moved a distance $X < l - D$, this will result only in transferring the strain field surrounding the dislocation by the same distance without causing any net effect on the surface.

From the previous argument, it may be concluded that dislocations positioned within a distance $D$ from the surface are the only ones that can contribute to the macroscopic strain, and not the total number of dislocations.
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