THE FATIGUE BEHAVIOUR OF ALUMINIUM AND THE EFFECTS OF SURFACE DAMAGE

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Presented for the Degree of Doctor of Philosophy

Physics Department

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1988

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Both single and polycrystalline specimens of aluminium (Al) have been fatigued at a range of total strain amplitudes, both with and without prior surface indentation. Specimen surfaces have been studied by optical microscopy and scanning electron microscopy (SEM) for the formation of fatigue slip bands. Transmission electron microscopy (TEM) has been used to study the dislocation configurations resulting from fatigue and indentation. It is shown that close to unannealed indentations either a stable cell structure is formed or a very dense quasi-homogeneous dislocation distribution; PSBs do not form in these regions during fatigue in the range of amplitudes investigated. PSBs are formed in regions of moderate dislocation density which are found near annealed indentations, and at larger distances from both annealed and unannealed indentations.

In addition to room temperature studies of Al, the dislocation configurations produced by fatigue at 77 K have been studied by TEM. The observed configurations after low strain amplitude fatigue at low temperature show considerable similarity to those found in other f.c.c. metals, such as copper (Cu) and nickel (Ni), after room temperature low amplitude fatigue. These configurations are quite different from those observed in Al after room temperature fatigue. A type of lattice dislocation configuration has been observed for the first time in Al fatigued at 77 K, and this can be related to multiple slip.
Some of the work described in this thesis has previously been published as follows:


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ACKNOWLEDGEMENTS

I should like to acknowledge my thanks to the following: to my supervisor, Dr. Philip Charsley, for help and guidance throughout the course of this work; to the Procurement Executive of the Ministry of Defence for funding this project; to Dr. R. N. Wilson of R.A.E. Farnborough for useful discussions; to the Microstructural Studies Unit at Surrey University for the provision of the scanning and transmission electron microscope facilities; to Mr. F. Bristow, Mr E. Worpe and members of the Physics Department workshops for assistance with construction of experimental apparatus.
CHAPTER 1

INTRODUCTION

Earlier work on pure Cu (White, 1984; Charsley and White, 1987) has shown that localized surface damage on annealed surfaces enhanced the formation of persistent slip bands (PSBs) during subsequent fatigue. This suggests that fatigue properties are considerably degraded by such damage, and mechanical failure by fatigue remains a major problem in many industrial situations. In the work on Cu, surface damage was introduced by indentation. It was shown that internal long range stresses were a contributory factor but that microstructural effects were also important; in particular plastic deformation on specific slip systems led to early PSB formation. However, transmission electron microscopy (TEM) studies were not undertaken in the work on Cu, hence the suggestions remained tentative.

The work described in this thesis is an extension of the earlier work on Cu to aluminium. Fatigue failure in commercial aluminium materials is of considerable importance, for example, in aircraft the main structural materials are Al alloys which are susceptible to corrosion pitting as well as mechanical damage. A degradation of fatigue properties that can be traced to these causes is well documented (Weston and Wilson, 1981). The aim of the work described here is to study the effect of the surface damage produced by
indentations on fatigue performance of Al and to use TEM, in addition to surface observations, to elucidate the mechanisms involved. The work has concentrated on pure Al, both polycrystalline and monocrystalline, in order to provide a basic understanding of both fatigue and indentation mechanisms.

The literature on the fatigue microstructures in Al is not detailed and dates back to 1971 and earlier. The considerable advances made since that date based on studies of fatigued Cu have not therefore been applied to Al. The dislocation microstructures around indentations in Al have, similarly, been neglected. The present work has sought to investigate these areas and the results are presented here.
CHAPTER 2

FATIGUE IN F.C.C. MATERIALS

2.1: Introduction

The study of metal fatigue is important because of the technological significance of fatigue damage which arises from cyclic deformation. Cyclic straining can result in the formation of cracks in materials which are initially perfectly free from cracks. This event is the final stage in a complex sequence of events. The fatigue process may be divided into two main regimes (Mughrabi, 1985): the early stages of cyclic deformation, which are associated with microstructural changes in the bulk extend up to some form of cyclic strain localization; this leads to different types of fatigue damage; strain localization, crack initiation and propagation.

The most detailed fatigue studies have been concerned with copper, therefore reference will first be made to studies of this material, with observations made on other materials presented subsequently.

2.2: Persistent Slip Bands and Other Surface Deformation Markings

2.2.1: Initial Observations of Persistent Slip Bands
During the fatigue of annealed polycrystalline copper specimens, it was found by Thompson, Wadsworth and Louat (1956) that periodic surface removal by electropolishing could apparently indefinitely extend the specimen life. It was also observed that regions existed where the specimen had undergone "some modification of the surface which (caused) them to react differently to the electropolishing conditions". Such regions were able to withstand a short electropolish which was sufficient to remove normal slip bands, thus these regions were termed persistent slip bands (or PSBs). The PSBs were marked by severe rumpling of the specimen surface and by intrusions and extrusions.

PSBs were found to be reluctant to propagate across grain boundaries, with each surface grain acquiring its own set of bands.

It was suggested by Thompson et al (1956) that PSBs might be regions where there was an enhanced oxygen concentration. However it was evident that such bands had their origins in, and were associated with, regions where there was higher than average strain amplitude. PSBs were found to spread slowly across the specimen surface as the fatigue test continued. This was accompanied by a steady increase in the area of the stress-strain loop. The response of a material to cyclic deformation at a constant stress amplitude may be characterised by continuously monitoring the plastic strain amplitude. During the initial phase of constant stress amplitude deformation of annealed crystals rapid cycling hardening occurs, giving rise to a decrease in the plastic strain amplitude.
accompanied by a decrease in the stress–strain loop width). In many cases an extended regime of cyclic saturation then follows, in which the plastic strain amplitude assumes a steady state value for constant stress amplitude. Thompson et al (1956) found the spreading of PSBs across the surface of polycrystalline specimens to be accompanied by a steady increase in the area of the stress–strain loop.

2.2.2: Two-Phase Model for PSBs

The fractional coverage of the surface of a specimen by PSBs is found to be linearly related to the plastic strain amplitude in the stable state. According to Winters two–phase model (Winter, 1974), the specimen may be considered to be a composite material consisting of a soft phase, the PSBs, which are fatigued at an amplitude \( \gamma_{pl,psb} \), and a harder phase, the matrix, which is fatigued at a much lower strain amplitude \( \gamma_{pl,m} \). The volume fraction, \( f \), of PSBs varies according to the plastic strain amplitude of the test as given by the following law of mixtures;

\[
\gamma_{pl} = \gamma_{pl,psb} \cdot f_{psb} + \gamma_{pl,m} (1-f_{psb}).
\]

The most detailed work concerned with the characterisation of the cyclic stress–strain curve has been on copper. Consequently the features of this curve will initially be discussed with reference to copper crystals, prior to the presentation results available for
other f.c.c. materials. Mughrabi (1978) investigated the fatigue of Cu single crystals and found that below a shear strain amplitude of $\gamma_{pl}$ of $6 \times 10^{-5}$, the saturation resolved shear stress, $\tau_s$, increases with increasing $\gamma_{pl}$ (see Fig. 2.1, region A). At $\gamma_{pl} = 6 \times 10^{-5}$ the first discrete PSBs form in the cyclically hardened matrix. Above $\gamma_{pl} = 6 \times 10^{-5}$ the volume fraction of PSBs increases approximately linearly with $\gamma_{pl}$, with PSBs occupying the whole of the gauge length at $\gamma_{pl} = 7.5 \times 10^{-3}$ (see Fig 2.1, region B). Throughout this plateau region, the saturation stress ($\tau_s \approx 28$ MPa) remains approximately constant, and corresponds to the stress required for localized deformation in the relatively soft PSBs which are strained in series with the matrix (see above equation). Above $\gamma_{pl} = 7.5 \times 10^{-3}$, $\tau_s$ again increases with $\gamma_{pl}$ (see Fig. 2.1, region C).

In nickel single crystals oriented for single slip Blochwitz and Veit (1982) found that PSBs do not form below a plastic strain amplitude value of $\gamma_{pl} = 6.5 \times 10^{-5}$. The cyclic stress-strain curve presented showed that the plateau region extended from $\gamma_{pl} = 6.5 \times 10^{-5}$ to $> 10^{-3}$, and throughout this region the saturation shear stress has a constant value of 50 MPa. Results were not presented for region C of the cyclic stress-strain curve.

2.2.3: Properties of PSBs

Fatigue of single crystals at constant strain amplitude results in the initial formation of PSBs as the stress amplitude approaches
saturation, followed by rapid propagation (Roberts, 1969). The PSB structure soon stabilizes and in this state the coverage of the surface by PSBs may be much less than unity. Even if the fatigue test is continued to failure PSBs do not propagate to cover the entire specimen, unless the strain amplitude is high enough.

It appears that once PSBs are formed, most remain active until the end of fatigue testing, at which point due to macroscopic crack propagation the stress amplitude falls. This point is illustrated by the observation by Mughrabi (1978) that at a plastic strain amplitude of $3 \times 10^{-3}$ the formation of PSBs on the surface is essentially completed within $\sim 5 \times 10^3$ cycles at a surface coverage of about 40%. Furthermore, results obtained by Winter et al (1981) showed that the surface coverage of PSBs on a crystal fatigued to failure ($2 \times 10^5$ cycles) was measured as 37%, whilst in a second crystal which was fatigued for $1.2 \times 10^5$ cycles, repolished to remove PSBs, and fatigued to failure (total number of cycles = $3.3 \times 10^5$), the surface coverage was 36%.

The point that individual PSBs remain active is illustrated by their removal from the specimen surface by a deep electropolish, and their reappearance on re-testing (Watt, 1969). It has also been found that the new PSB structure corresponds to the old apart from the finest detail for both polycrystalline and single copper crystals (Rasmussen, 1980; Winter 1974).

It should however be noted that although the PSB pattern does not
apppear to change drastically throughout fatigue, most PSBs do not remain continuously active and the sites of current activity are found to shift. Basinski et al (1983) carried out in situ optical microscopy of the surface of crystals repolished after fatiguing well into saturation. It was found that on resuming the test the activity was confined to sharp narrow lines which were located predominantly but not exclusively near the borders of original wide bands, except for the continued operation of very narrow PSBs in the matrix. The inner band was found to be almost inactive with only a low level of uniform deformation. SEM investigations by Basinski and Basinski (1985 a) confirmed that active areas found at the beginning of saturation usually ended up deep within wide PSBs due to the method of widening by addition of extrusions in the matrix at both PSB borders and merging of neighbouring bands. Observation of the development of the PSBs in a number of areas, showed that after initial rapid PSB growth, the addition of new extrusions then continued at a lesser rate until the observed PSB areas were filled up with extrusions, at which point it became apparently inactive apart from the appearance of fine secondary slip markings. These results are in agreement with the interferometric observations on PSB development on repolished copper single crystals, which were of single slip orientation, by Finney and Laird (1975).

Thompson et al explained the beneficial effect of electropolishing on increasing fatigue life by suggesting that PSBs did not spread into interior grains. However, Winter et al (1981) suggest that the increase in the area of the stress-strain loop which accompanied
PSBs spreading would indicate that these might be spreading into the bulk of the specimen as well as across the surface. Furthermore, TEM studies of the dislocation structure in fatigued polycrystalline copper by Winter et al (1981), reveal structures characteristic of PSBs in the interior of the specimens.

The observations by Basinski and Basinski (1985a) of cracks located at narrow PSBs, isolated intrusions and inside PSBs, led to their suggestion that the beneficial effect of electropolishing at any stage of fatigue life is the removal of cracks which have accumulated, and thus the minimum of another life-span would be necessary to rebuild a similar array of cracks.

In aluminium, King and Teer (1969) observed PSBs in crystals which had their stress axes at the centre of the stereographic triangle and in those near the [100] - [110] boundary. Stress axes near the [100] - [111] boundary were seen to result in the formation of PSBs on two sets of slip planes thus creating a "herring-bone" appearance.

2.2.4: PSB Geometry

Mughrabi and co-workers (1983) found that PSB profiles were net protruberances whose surfaces become progressively roughened as the fatigue test continues. Neumann (1983) found PSBs to consist entirely of extrusions, with adjacent sides of neighbouring extrusions forming notches whose vertices are never below the
original surface, and thus intrusions as such are not observed.

Recent work by Basinski and Basinski (1985 a) on room temperature low amplitude ($\varepsilon_{pl} = \pm 2 \times 10^{-3}$) fatigue of copper single crystals with crystal axes [321], showed PSBs to consist of intrusions and extrusions of comparable abundance, and with similar geometry. This is in agreement with the observations of Cotrell and Hull (1957) on polycrystalline copper. Basinski and Basinski (1985 a, b) examined by SEM replicas of the surfaces of crystals, prepared by electropolishing away from a coating of lacomit. The first features observed anywhere were invariably tips of replicated intrusions. With continued electropolishing bands of intrusions became apparent over the whole of the specimen, while a layer of crystal still remained, indicating that intrusions had indeed penetrated the bulk. In addition, the existence of intrusions was shown by the common observation of isolated intrusions which were unaccompanied by extrusions. Clearly, such structures could not be confused with notches between extrusions.

It is suggested by Basinski and Basinski (1985 a) that Neumann's (1983) observation of PSBs which consist entirely of extrusions might be explained by the replicating technique used, in which the original surface reference level is removed and hence no distinction could be made between true intrusions and notches between extrusions. SEM observations by Charsley and Razzak (1987) give further support to the PSB observations of Basinski and Basinski. Thus available experimental evidence appears consistent with PSBs.
2.2.5: PSB Size

At the onset of saturation extrusions and intrusions form in large numbers with a limiting height of approximately 4 μm not exceeded by extrusions and most intrusions (Basinski and Basinski, 1984; 1985 a, b). However, a small number of intrusions are not found to be limited in this way. Such intrusions acquire rough surfaces, indicating that growth has occurred non-uniformly along their length and they advance into the crystal as a series of spearhead advances along an irregular front, with increasing cumulative plastic strain.

2.2.6: Effect of Environment on PSBs

Wang, Mughrabi, McGovern and Rapp (1984) examined the fatigue of copper crystals in high vacuum. Room temperature fatigue of single crystals oriented for single slip, at constant plastic resolved shear strain amplitude of 2 x 10^{-3} in high vacuum, revealed a prolonged fatigue life although the cyclic deformation behaviour in vacuum and air were similar up to the number of cycles for failure in air (N ~ 10^5). No significant differences were observed with regard to the distribution and surface features of the matrix and PSBs. Fatigue in high vacuum up to 2-3 x 10^6 cycles, revealed that a secondary hardening stage occurred after ~ 10^6 cycles and the
surfaces were found to be almost completely covered with PSB traces. Wang et al (1984) inferred that old PSBs gradually harden during the process of secondary cyclic hardening by conversion of their dislocation structure into cell structures, and are continuously replaced by new PSBs which form out of the matrix until the specimen is filled with both old and new PSBs. The homogeneous surface coverage with PSBs after extensive fatigue in high vacuum occurs because of the prolonged fatigue life.

2.2.7: Models to Describe PSB Geometry

Early models of PSB surface profiles were based mainly on surface observations of extrusions and intrusions, these have been reviewed by Laird and Duquette (1972). More recently some models have been based on conclusions from TEM observations of dislocation behaviour of PSBs in bulk material.

2.2.7 (i): Model Predictions

Essmann, Gösele and Mughrabi (1981) and Brown et al (1981, 1985; Antonopoulos, Brown and Winter, 1976) have each presented microstructural models for fatigue which make predictions about PSB surface geometry and crack initiation sites. These models are based on different dislocation mechanisms but both predict that the surface profile arises from two sources. The predicted PSB profile
has the following features: a PSB surface level raised above that of the adjacent matrix and superimposed on this protrusion is a statistical roughening attributable to microscopic slip events, which increases with increasing cumulative strain. Cracks nucleate predominantly at the PSB-matrix interface, initially at that interface where the primary plane externally makes an obtuse angle with the matrix surface and later also at the other interface.

2.2.7 (ii): Model Descriptions

Considering steady-state cyclic dislocation glide, as cyclic saturation is approached in a PSB, the dislocation arrangement is assumed by Essmann et al (1981) to consist essentially of two layers of PSB-matrix interface edge dislocations of opposite sign. In PSBs operating under single slip conditions, annihilation of edge dislocation dipoles is assumed to be a dominating source of point defect production. The atoms which belong to the extra half plane of the interface dislocations correspond in number to those atoms missing in vacancy-type defects present within the PSBs at saturation. Superficially the model described by Essmann et al appears similar to that described outlined by Brown et al, however the assumptions on which the latter is based are different. Brown et al assume a vacancy type dipole array derived from TEM studies. It is also assumed in the model described by Brown et al that the dislocation density in the PSB exceeds that in the matrix and continues to increase in saturation. Annihilation of edge
dislocations is not considered by Brown et al. Fig. 2.2 shows PSB formation due to the model by Brown et al, and the basic mechanisms of the model due to Essmann et al are illustrated in Figs. 2.3 a, b.

Interface dislocations are assumed to be less stable in the model of Essmann et al than in that of Brown et al, and under the action of applied and internal stresses, the interface dislocations are driven out of the crystal rather quickly. This leads to the rapid formation of extrusions at both top faces. On the other hand, dislocations do not glide out in the model of Brown et al, thus no specific surface feature apart from roughness due to random slip is predicted.

In the model of Brown et al, a residual tensile stress is predicted to prevail in the PSB in the direction of the active slip vector after fatigue (see Fig. 2.2). Essmann et al (1981) consider the applied stress to act additively to the stress arising from the dislocations at the PSB-matrix interface. The resulting stress tends to move interface dislocations towards the specimen surface. Consideration of the interface dislocation arrangement indicates an elastic compressive stress within the PSB in the direction of the active slip vector, and a tensile stress within the surrounding matrix. Residual stresses result in the favourable formation of PSBs next to existing ones. For this situation an optimum distance should exist between adjacent PSBs for which the elastic energy of the interface dislocations is minimized. Through the action of applied and internal stresses on the interface dislocations in PSBs stress concentrations are expected to form.
The order of magnitude of the elastic strain has been estimated by Antonopoulos et al from microscopic observations. Atoms which are missing from the vacancy dipole in the PSB appear as extrusions on the specimen surface. Since the extrusions are typically a few microns high for a specimen ~ 1 cm in diameter, the order of magnitude of misfit strain is ~ 10^{-4}. Tensile stresses produced by elastic misfit are about 26 MN m^{-2} for elastic strains of 2 \times 10^{-4}.

As noted previously, in the model developed by Essmann et al it is assumed implicitly that in PSBs operating under single slip conditions the annihilation of edge dislocation dipoles is the dominating source of point defect production. After initial rapid extrusion growth two situations are thus distinguished; depending on the testing temperature the vacancy type defects can be swept up by or can migrate to dislocations in the walls, or can diffuse out of the PSBs continuously. In the latter case the possibility of void formation at the PSB-matrix interface has been noted. At low temperatures, e.g. for copper at 77 K, the extrusions should cease growing and their surfaces should roughen gradually due to the randomness of irreversible slip processes. Although the slip process would become less random once severe stress raisers or cracks have formed. Vacancy-type point defects are considered to be immobile at temperatures well below the resistivity recovery stages III and IV.

With increasing temperature in the model developed by Essmann et al, some of the vacancy-type defects will become mobile and will diffuse
from the region of high vacancy concentration in the walls into the matrix. If their diffusivity is sufficiently high vacancies will reach sinks in the matrix, such as dislocations, before a high supersaturation of vacancies can be built up. This regime is termed the high mobility temperature regime and this model assumes the formation of a matrix and PSB structure, in which vacancies which are lost to the matrix will be replaced continuously by dipole annihilation processes in the PSB walls. Thus at intermediate temperatures, slow continuous growth of extrusions is predicted which can interfere with roughening. In very thin PSBs continuous extrusion growth over the whole thickness is predicted whereas in the thicker PSBs (> 1 μm), continuous extrusion growth would occur only at thin ribbons of extrusions at the PSB-matrix interface (e.g. as has been observed experimentally for Cu fatigued at 293-403 K). These conditions are found to hold up to about 0.4 T_m (where T_m is the material melting point temperature). At higher temperatures dipoles become unstable due to dislocation climb and thus PSBs are no longer expected to form in Cu (Shirai and Weertman, 1983).

2.2.8: Comparison Between Model Predictions and Experimental Observations

Experimental results obtained by Basinski and Basinski (1985 a, b) on copper fatigued at room temperature, after 2.5 x 10^4 to 3.15 x 10^5 cycles, may be compared with PSB model predictions. The predicted height of the PSB surface level above that of the adjacent
matrix calculated using the models by Mughrabi et al and Brown et al would be a maximum of 1 μm. Basinski and Basinski (1985 a) experimentally observed PSBs to have an irregular sawtooth profile with individual extrusion and intrusion heights of up to 4 μm. Thus Basinski and Basinski suggest that the height of the PSB surface level above that of the adjacent matrix would be difficult to detect, since this height is within the amplitude of the jagged profile. It should be noted that Basinski and Basinski often observed protrusions which were very much larger than those predicted by the theories, however they were not always found to be present for the strain amplitudes investigated which were in the range ± 1 x 10^-3 - 2 x 10^-3. Stronger surface protrusions were evident for crystals fatigued at much lower strain amplitudes, however no detailed studies have been made.

Due to the regularity of extrusions formed in fatigued copper Basinski and Basinski (1985 b) suggest that the characteristic extrusions and intrusions are a result of a systematic dislocation mechanism. Thus difficulty is experienced with regard to reconciling the the degree of regularity with the types of profiles generated using models based on random statistics. The development of PSB features is also found by Basinski and Basinski not to be compatible with a random walk model. Basinski and Basinski found that a given extrusion will appear and may grow for approximately 10^4 cycles to a limiting height and will then be unchanged by further cycling with no progressive roughening of the extrusion being observed.
Finally, both models require cracks to occur at the PSB-matrix interface where the primary plane externally makes an obtuse angle with the matrix surface and subsequently at the other interface also. Basinski and Basinski (1985 b) found that although cracks did form at these locations there was no preference for one particular interface. In addition crack sites which were well within wide PSBs were observed.

It is suggested by Mughrabi (1985) that model predictions are most specific for low temperature tests and for the first few thousand cycles of room temperature tests. Hence, Mughrabi suggests that low temperature SEM observations of fatigued aluminium and copper crystals (Mughrabi 1985) indicate support for the model predictions, whereas after a large number of cycles at room temperature the impression of a more complex system is conveyed.

2.2.9: Development of Surface Deformation Markings other than PSBs

During low amplitude fatigue testing of polycrystalline Al, King and Teer (1969) found that in grains other than those with their stress axes at the centre of the stereographic triangle, the surface features developed were rows of surface undulations or "rumpled" bands. A schematic diagram to illustrate rumpled bands is shown in Fig. 2.4. Such rumpled bands were found to develop much later during fatigue than persistent slip bands.
Arnell and Teer (1969) examined in detail the relationships between the type of surface structure developed, and the grain orientation for polycrystalline Al. For crystal orientations which had stress axes < 5° from [100] bands of cross-hatched ripples were observed. For orientations 5° to 10° from [100] the bands had an appearance intermediate between that of rumpled bands and persistent slip bands. As the stress axis moved from the centre of the stereographic triangle towards [111], the surface appearance of the PSBs became striated, whilst for axes ~ 5° from [111] the absence of bands was noted and the surface had a rumpled appearance. As the orientation of the stress axis moved close to [100] along the [100] - [110] boundary, a cross-hatched structure similar to that observed by Forsyth (1956) was observed.

It is interesting to note that King and Teer (1969) did not observe regions exhibiting only rumpled bands in fatigued copper specimens, and no rumpling of any kind was seen in α-brass.

2.3: Fatigue Crack Initiation

2.3.1: Crack Formation in Materials with PSBs

In copper, Thompson, Wadsworth and Louat (1956) observed fatigue cracks to nucleate in regions of specimen surface which exhibited PSBs. Hunsche and Neumann (1986), Neumann (1983), Mughrabi et al
(1983) found that cracks appeared predominantly at PSB-matrix interfaces. Basinski and Basinski (1985 b) observed cracks to form anywhere in PSB material. Cracks were found both deep within wide bands and at the borders of such bands. The most frequently observed crack site was at very narrow PSBs, including cracks forming at isolated intrusions.

It is suggested that the observed differences in apparent crack development lies in interpretation rather than in experimental data. The SEM micrographs of Mughrabi et al (1983) appear to be very similar to those presented by Basinski and Basinski (1985 a, b), however the former have based their observations on surface data only, whereas the latter have examined cross-sections which enable intrusions and extrusions to be distinguished. Basinski and Basinski (1985 b) suggest that surface studies are further restricted due to extrusion inclination to the crystal surface, which results in intrusion and crack sites being observed with certainty only at the PSB-matrix interface where extrusions make an obtuse angle with the matrix structure.

In addition, some of the apparent disagreement may be of a semantic nature. Basinski and Basinski (1985 b), for example, use the term extrusion to refer to long ribbons of material which constitute the usual external PSB appearance when grouped together. Thus, Basinski and Basinski would term the features described as new smooth PSBs by Mughrabi et al as an isolated extrusion; whilst the increased roughness described by the latter in an old extrusion, would be
referred to as a PSB which contained many similar extrusions by Basinski and Basinski. Further, it is suggested by Mughrabi (1985) that the intrusions described by Basinski and Basinski are similar to the notch like grooves discussed by the former.

Cracks have also been found to occur in regions which exhibit PSBs in aluminium (Arnell and Teer, 1969; King and Teer, 1969). The cracks which formed were observed to be parallel to the primary slip planes.

In copper, close to failure there is a large population of cracks in the 50 - 100 \( \mu \text{m} \) range, for the amplitude investigated by Basinski and Basinski (1985 b). On the basis of their data, Basinski and Basinski suggest that a reproducible fatigue life may be explained by the point that near to failure cracks are the result of steady continuous growth, and thus the fatigue life is a measure of how many cycles are required before the array of cracks reaches a configuration such that one or some of them could propagate catastrophically.

2.3.2: Crack Initiation in Regions other than PSBs

After low amplitude fatigue of polycrystalline Al, Arnell and Teer (1969) found that in grains which exhibited rows of rumpled bands, cracks initiated along the rows and were parallel to the primary slip planes. In the case of bands of intersecting rumples, the crack
was found to form perpendicular to the stress axis, with individual fracture segments meandering between the two rumple directions.

2.3.3: Effect of Environment on Fatigue Cracks

An additional important factor in the fatigue cracking process is the role of environment. For copper crystals oriented for single slip, stage I shear cracks also develop in high vacuum (Hunsche, 1982; Mughrabi et al., 1983; Neumann, 1983; Wang, 1982; Wang and Mughrabi, 1984). For single crystals oriented for multiple slip and tested in high vacuum, no stage I fatigue cracks are observed (Neumann et al., 1977). From the work of Hunsche (1982) and Neumann (1983) it is found that in air local cumulative shear strains as high as several thousand are required for stage I cracks in "intrusions" (v-shaped notches between adjacent extrusions) to reach a length of ~ 1 \( \mu \)m. Local cumulative shear strains an order of magnitude larger are found to be necessary in vacuum.

There is agreement between Hunsche, Mughrabi and Neumann that all features of PSB surface profiles which are relevant to crack initiation in fatigued copper crystals are found to be similar in air and vacuum for a given number of cycles (Hunsche, 1982; Mughrabi, 1985; Neumann, 1983). In addition, the local cumulative shear strains in the PSBs in air and in vacuo are found to be of similar magnitude at the stage before a crack can be defined. Thus environmental effects may be considered negligible until a crack of
finite length can be defined. Subsequently the rate of growth of stage I fatigue cracks is reduced in vacuum, and this is seen to be a major factor contributing to enhancement of fatigue life by an order of magnitude compared to tests in air (Wang and Mughrabi, 1984; Wang et al, 1984).

2.4: Dislocation Structures in f.c.c. Metals

2.4.1: Parameters Affecting Dislocation Structures Produced

The type of dislocation structure developed during fatigue is dependent on the stress or strain amplitude used, the stacking fault energy (SFE) of the material and the testing temperature. Low stress fatigue tests are generally defined to be tests in which the fatigue life (N_f) is greater than 10^6 cycles, and high stress fatigue tests as those in which failure occurs in < 10^5 cycles. A diagram showing the types of dislocation structure observed with various SFE and N_f values is shown in Fig. 2.5.

The most detailed work concerned with the development of fatigue dislocation structures has been on copper. Consequently, results obtained on this material will be considered prior to examination of structures produced in materials with higher and lower SFE.
2.4.2: Dislocation Structures in Medium SFE Materials

An example of a material with a medium stacking fault energy is copper (SFE = 5.5 x 10^{-2} Jm^{-2}; Gallagher, 1970). At low plastic strain amplitudes, most grains in polycrystalline material show a preference for single slip (Mughrabi and Wang, 1981). Dislocation arrangements similar to those found in Cu single crystals fatigued in the plastic strain range (Δε_pl), 10^{-5} < Δε_pl < 2 x 10^{-2} form in polycrystals at much lower Δε_pl, in the range 10^{-5} < Δε_pl < 2 x 10^{-3} (Mughrabi and Wang, 1981). At strain amplitudes Δε_pl > 10^{-3} the single slip characteristics of the majority of grains in polycrystalline material are lost and comparison should be made with single crystals in which multiple slip is occurring (Mughrabi and Wang, 1981).

The microstructures which develop during room temperature fatigue at various strain amplitudes in polycrystalline Cu and Cu crystals of easy glide orientation have been investigated in depth (Laufer and Roberts, 1966; Lukáš, Klesnil and Krejčí, 1968; Basinski, Basinski and Howie, 1969; Woods, 1973; Winter, Pedersen and Rasmussen, 1981; Mughrabi, 1981; Mughrabi and Wang, 1981; Ackermann, Kubin, Lepinoux and Mughrabi, 1984). PSBs are found to correspond to specific dislocation configurations consisting of parallel, narrow equally spaced dipolar (101) walls (ladder structure) after low amplitude fatigue (Antonopoulos and Winter, 1976; Winter et al, 1981; Ackermann et al, 1984). Fig. 2.6 shows a schematic diagram of the PSB dislocation structure. The spacing between the walls in this PSB
structure is found to be ~ 1.4 \( \mu \text{m} \) (in polycrystals and single crystals). Between the PSBs there is another less well ordered structure (matrix) containing a dense array of primary dislocation dipoles pierced by irregular winding channels of relatively undislocated material, which have a characteristic width of ~ 0.8 \( \mu \text{m} \) (Winter et al, 1981). The dislocation structure of PSBs is observed in the interior of both single crystal and polycrystalline Cu after fatigue (Winter 1973; Winter et al, 1981). The majority of dislocations in PSB and matrix structure in Cu single crystals oriented for single slip were found to be primary dislocations of \( \mathbf{b} = \mathbf{b}[\bar{1}01] \), although dislocations of all six possible Burgers vectors of \( \mathbf{b}<110> \) type have been found (Woods, 1973; Lukáš, Klesnil and Krejčí, 1968). In the dislocation walls, the density of edge dislocation dipoles which have the primary Burgers vector is estimated to be in excess of \( 10^{12} \text{ cm}^{-2} \). The density of secondary dislocations mixed with these primary dislocations is about \( 2 \times 10^{10} \text{ cm}^{-2} \) (to within a factor of two). The secondary dislocations are found to be mainly those co-planar with primary dislocations. In the regions between the walls, there is a primary screw dislocation density of about \( 1 \times 10^9 \text{ cm}^{-2} \). Estimates of the densities of non-primary dislocations between the walls are: \( 5 \times 10^8 \) for \( \mathbf{b} = \mathbf{b}[101], \mathbf{b}[110], \) and \( \mathbf{b}[\bar{1}10] \) and \( \mathbf{b}[0\bar{1}1], \) whilst for \( \mathbf{b} = \mathbf{b}[011] \) the density is estimated to be \( 3 \times 10^8 \) (estimates accurate to within a factor of two).

At stress amplitudes above the plateau region on the cyclic stress-strain curve (Mughrabi 1978), in addition to those structures
found at low amplitudes, misoriented cells and two-dimensional (100) wall structure (termed as labyrinth by Charsley, 1981) with a wall spacing of 0.75 μm are found in polycrystalline copper (Winter et al, 1981). Such misoriented cells are also observed in single Cu crystals (Woods, 1973; Jin and Winter, 1984 a). Labyrinth structure has recently been observed in single slip oriented Cu crystals after fatigue at a plastic shear strain amplitude of 2 x 10^{-3} (Ackermann et al, 1984) and in fatigued [001] Cu single crystals (Jin and Winter, 1984 b) with respective wall spacings of 0.75 μm and 0.52 μm.

After fatigue at low strain amplitude for a large number of cycles, the dislocation structure in Cu consists of a misoriented cell structure (Krejčí and Lukáš, 1971; Finney and Laird, 1975; Kuhlmann-Wilsdorf and Laird, 1980; Ackermann et al, 1984).

2.4.3: Dislocation Structures in Very Low SFE Materials

In materials with very low stacking fault energy e.g. Cu-31 wt% Zn (SFE = 6 x 10^{-3} Jm^{-2}; Swann, 1963) dislocations are arranged in planar arrays at both high and low stress amplitudes (Lukáš and Klesnil, 1970, 1971). No specific dislocation configuration under surface slip markings (PSBs) is observed and the only difference between foils prepared from the surface and those prepared from greater depth is that the density of dislocations in the latter case is found to be higher.
2.4.4: Dislocation Structures in Low SFE Materials

In Cu-15wt% Zn, which has a low stacking fault energy (SFE = 1.2 x 10^{-2} Jm^{-2}; Swann, 1963), coarse surface slip bands (PSBs) are always connected with ladder type dislocation structures surrounded by dislocation tangles, whilst in the interior a vein structure is found, after low stress amplitude fatigue. After fatigue at higher stress amplitudes, a cell structure is found in both interior and surface regions (Lukáš and Klesnil, 1970, 1971).

A further example of a material with low stacking fault energy is Cu-16at.% Al. The dislocation structure produced by fatigue in this material has been examined by Laird et al (1986). After low amplitude fatigue the dislocation structure is found to consist of a two dimensional slab of dislocations separated by regions which are lightly populated by dislocations. At higher amplitudes, \( \gamma_{pl} = 10^{-3} - 3 \times 10^{-3} \), typically PSBs consist of a dense slab of primary dislocations of several micrometers thickness, which are stepped with respect to contiguous planes; together with secondary dislocations, which have slipped on planes other than the primary slip plane, which intersect groups of primary dislocations. The volume fraction of these bands increases and secondary slip becomes more complex with increasing amplitude. Feltner and Laird (1967) have found that at very high amplitudes the bands exist on more than one set of slip planes.
2.4.5: Dislocation Structures in High SFE Materials

In materials which have a high stacking fault energy such as Al (SFE = 0.2 Jm\textsuperscript{-2}) and Ni (SFE = 0.25 Jm\textsuperscript{-2}), preliminary observations by Krejčí and Lukáš (1971) revealed bands of dislocations in the interior of specimens after low amplitude fatigue of polycrystalline material, similar to those observed in polycrystalline Cu. In the surface layer of specimens, cell structures were found to be limited to areas near surface slip markings, whilst bands of dislocations were observed in areas in which no slip bands were apparent. In deeper regions of Ni specimens a ladder type structure (similar to PSBs in Cu) was found, however such structures were not observed in Al.

Due to the considerable differences in the observed dislocation structures produced in these materials, the additional results of fatigue studies on these two materials will be reported separately.

2.4.5 (i): Dislocation Structures in Ni

matrix structure is formed in crystals of single slip orientation (specimen axis [149]) after fatigue at low amplitude (total strain amplitude = \( + 0.5 \times 10^{-3} \)). A second type of matrix structure is observed (Mecke and Blochwitz, 1982) with increasing strain amplitude (\( \pm 2.6 \times 10^{-3} \)). This structure consists of edge dislocation walls regularly arranged at an angle of \( \sim 45' \) and \( 135' \) respectively to the primary slip direction, and builds up a "parquet-like" structure. Fatigue at higher strain amplitudes is found to lead to the formation of PSBs which differ significantly from the surrounding matrix structure by their typical ladder-type structure. From investigations of [121] planes (Mecke, Blochwitz and Kremling, 1982), ladder rungs were found to lie along the [111] direction with a spacing of \( 1.3 \pm 0.1 \mu m \), and the ladders were found to be aligned along the primary slip direction [101]. The dense regions in the matrix type structure are found to occupy \( \sim 50\% \) of the volume, whereas dense regions in the ladder walls occupy only 10\% (Mecke and Blochwitz, 1982). It was confirmed that a cylinder-like cell structure was observed in Ni after fatiguing specimens for a greater number of cycles, as is found in copper.

In crystals with a stress axis [001], at low amplitudes the spatial distribution of the vein-like structure consists of volume fractions with different dislocation densities. The following types of dislocation structure were identified by Mecke and Blochwitz (1982): loosely packed bundles of dislocations along a \langle 110 \rangle \) direction; regions filled with dislocations in a random distribution; regions which appear relatively free of dislocations. With fatigue at
increased strain amplitude, the spatial distribution was found to consist of condensed and uncondensed labyrinth type walls. The condensed labyrinth structure consists of \( <100 \) walls which are much narrower than the low dislocation density channels between them, whereas the uncondensed labyrinth structure consists of \( <100 \) walls which are comparable with (or greater than) the width of the channels. Screw dislocations from two slip systems were clearly apparent in the dislocation poor regions.

2.4.5 (ii): Dislocation Structures in Al

The work of Mitchell and Teer (1970) which examined the low strain amplitude fatigue (\( N_f = 10^7 \) cycles) of Al single crystals of single slip orientation with stress axes \([\bar{1}23]\), expanded on their earlier work (Mitchell and Teer, 1969 b). At early stages of fatigue (\( 10^2 \) to \( 2 \times 10^3 \) cycles), many regions were almost devoid of dislocation structure, whilst in other regions the characteristic structure was composed of irregular bands of tangled dislocations together with a few dipoles. This structure was found beneath both PSB and matrix features. Similar observations of dislocation tangles and loops after low amplitude fatigue of polycrystalline Al have been made by Grosskreutz (1963), Grosskreutz and Waldow (1963), Segall and Partridge (1959) and Wilson and Forsyth (1959).

At intermediate stages of fatigue of single crystals (\( 4 \times 10^3 \) to \( 2 \times 10^5 \) cycles), and after low amplitude fatigue of polycrystalline Al
(Grosskreutz and Waldow 1963), matrix structure similar in appearance to that found in Cu and Ni has been observed. In single crystals this structure has been found to consist of clusters of dislocations containing a high proportion of dipoles and bands of tangled dislocations containing only a few dipoles, which are aligned almost vertically in the foil following the general direction of the critical slip plane. Channels cutting through the matrix structure were found by Mitchell and Teer (1970) to correspond to PSB markings, and dislocations were present here either as small loops distributed evenly throughout the channel or in 0.25 μm wide bands of tangled dislocations which crossed the channels. These bands were frequently extensions of bands of dislocations present in the matrix. Burgers vector analysis by Mitchell and Teer (1970) indicated that the clusters contained a high proportion of dislocations with \( \mathbf{b} = \frac{1}{2}[110] \), with all other types except \( \frac{1}{2}[110] \) (which was very rare) present in about equal numbers. Almost all of the dislocation loops in regions corresponding to PSB markings were of the three dislocation types common to the primary plane. In the boundaries crossing the channels dislocations were mainly of the three types common to the primary plane with appreciable numbers of dislocations with \( \mathbf{b} = \frac{1}{2}[011] \) and \( \frac{1}{2}[101] \) also evident.

At a late stage of fatigue of single crystals, a cell dislocation structure, which was general throughout the crystal, was characteristic of both matrix and PSB (Mitchell and Teer, 1970). In the cell boundaries dislocations with all six Burgers vectors were
found, although the type $\gamma[110]$ was much less common. Cell structures are also observed in polycrystalline Al (Segall and Partridge, 1959; Grosskreutz, 1963). It has been found that after low amplitude fatigue ($\varepsilon_t = \pm 2 \times 10^{-3}$) both equiaxed and elongated cells with cell walls approximately parallel to (010), (1\overline{1}0), (1\overline{1}1), (1\overline{1}1) and (111) are apparent in favourably oriented grains (Grosskreutz, 1963). Varying the strain amplitude in the range $\pm 1 \times 10^{-3}$ to $\pm 5 \times 10^{-4}$ was found to result in slower cell formation with a greater number of dislocation loops at the lower amplitude.

The investigations of Mitchell and Teer (1970) have revealed that similar dislocation structures are developed in surface and interior foils. As outlined above, in polycrystalline Al similar structures to those observed in single crystals are found, although it should be noted that the substructure developed after a given number of cycles was found to vary from grain to grain depending on the orientation for the polycrystalline material.

In addition to the types of dislocation structures described above, work by Wilson and Forsyth (1958-59) on room temperature fatigue of super purity Al in reverse plane bending, showed that some regions exhibited a "chevron" pattern dislocation structure after $2 \times 10^7$ cycles. This chevron type structure is similar in appearance to the labyrinth structure observed in copper, however Wilson and Forsyth do not specify the orientation of the dislocation walls or the extent of the structure observed in aluminium. Three main stages in the evolution of the dislocation structure have therefore been
suggested by Wilson and Forsyth (1959): (a) the production of a general dislocation distribution throughout a grain, as a result of the generation and interaction of dislocations; (b) the development of dislocation clusters which have a high density of dislocation lines and loops within grains; (c) a recovery process in which a well defined substructure is produced due to annihilation of dislocations.

2.4.6: Effect of Temperature on the Dislocation Structures Produced

Basinski, Korbel and Basinski (1980) examined the push-pull fatigue of copper crystals oriented for single glide at 77.4 K and 4.2 K. Attempts to reduce temperature ageing effects were not reported. At each temperature the crystal substructure was found to consist of two types of region, PSBs and matrix. The appearance of the observed microstructure was characteristic of structures produced after room temperature fatigue, but the scale of the structures differed with the test temperature. The observed ladder spacings in the PSBs were, for example, found to decrease sharply with test temperature to 0.7 ± 0.05 μm and 0.45 ± 0.03 μm at 77.4 K and 4.2 K respectively compared with the value of 1.4 ± 0.1 μm at 300 K.

Observations by Mughrabi (1986) of copper fatigued at 77 K which has then been aged at room temperature prior to TEM examination, indicate the occurrence of recovery of the dislocation structure.
Dislocation debris in annealed 99.99% polycrystalline Al fatigued in reverse plane bending at 78 K was examined by Feltner (1963). For low strain amplitudes, \( \pm 5 \times 10^{-5} \) for \( 2 \times 10^5 \) cycles, 90% of the dislocation structure was found to be composed of elongated dislocation loops, with a density of \( \sim 10^{15} \) loops \( \text{cm}^{-3} \). The loops were found to be almost homogeneously distributed, with their long dimension lying along the projection of a single \( <112> \) direction. At higher strain amplitudes, \( \pm 2 \times 10^{-3} \) for \( 4 \times 10^4 \) cycles, dislocation debris consisted mainly of elongated dislocation loops, with densities of the order of \( 10^{16} \) loops \( \text{cm}^{-3} \), which were found to aggregate into a cellular arrangement with an average cell size of 4 \( \mu \text{m} \). Such aggregation into cell walls occurs only when loops parallel to two or more \( <112> \) directions are present. It should be noted that attempts to reduce ageing effects following low temperature fatigue do not appear to have been made.

2.4.7: Effect of Environment on Dislocation Structures

Fatigue of Cu single crystals, which were oriented for single slip, in high vacuum up to \( 10^5 \) cycles, (the number of cycles to fatigue failure in air), was not found to produce any significant differences in the dislocation structures of the matrix or PSBs as compared with fatigue in air (Wang et al, 1984). On continued fatigue in high vacuum, (up to \( 2-3 \times 10^6 \) cycles), the dislocation
structures produced were found to consist almost entirely of misoriented cells, which were arranged in slabs parallel to the primary glide plane. The formation of this structure is attributed to the extended fatigue life in vacuum.
CHAPTER 3

INDENTATION EFFECTS ON FATIGUE

3.1: Introduction

The formation of persistent slip bands near regions of surface damage (Vickers microhardness indentations) has been examined by Charsley et al (1981), White (1984), and Charsley and White (1985, 1987). Such studies are important because in engineering situations the fatigue failure of components may be significantly affected by surface imperfections. The object of the work by Charsley and White has been to assess the effects of damage under simple laboratory conditions. In order to study the effects of indentation on fatigue it was necessary to examine a material which had already been extensively studied, for this reason the room temperature fatigue of pure copper was chosen.

3.2: Factors Affecting PSB Initiation and Development

3.2.1: General Observations

Indented copper single crystals fatigued by White (1984) were initially hardened at a low strain amplitude before cycling at a final amplitude of either $\varepsilon_t = 8 \times 10^{-4}$ (crystal orientations with
the tension-compression axis, t₀, 12° from [011] or [\bar{1}11] and 20° from [\bar{1}11]) or εₜ = 9 \times 10^{-4} (crystal orientations with t₀ 17° or 20° from [001] and 14° from [\bar{1}11], see Fig. 3.1). It was found that after < 1 \times 10^4 cycles of fatigue a distribution of PSBs developed near the indentations, however no bands were observed in the regions between them even after large numbers of cycles (> 7 \times 10^5). It was found that the distribution of PSBs was dependent on the crystal orientation, but the orientation of the indentation (S or D type, corresponding to the indentation side or diagonal respectively being parallel to t₀, see Figs. 4.3 a, b) did not greatly affect the distribution of PSBs. For unannealed indentations it was found that no PSBs initiated at the pit edge nor within the indentation pit. Two types of PSB could be distinguished; short bands which became more pronounced but which did not lengthen with continued cycling, and long bands which continued to extend over a large number of cycles. Electropolishing of PSBs revealed that the propagating bands extended much more deeply into the material than the short bands. It was also found that the rate of growth of these propagating bands decreased with increasing cycle number.

3.2.2: Effect of Crystal Orientation

For all crystal orientations with t₀ 12° from [011] and 12° or 20° from [\bar{1}11], White, (1984), observed that the first PSBs nucleated between 2 \times 10^3 and 8 \times 10^3 cycles. For orientations with t₀ 20° from [001], PSBs were not observed until 2 \times 10^4 cycles. The formation of short PSBs was found to depend on the crystal
orientation, and these were observed mainly on crystals with \( t_0 \) 12' from [011] and 17' from [001], although propagating PSBs occurred near indentations in all crystal orientations tested by White. The rate of propagation of PSBs was found to vary with crystal orientation.

3.2.3: Effect of Indentation Size

In one case, White (1984) examined the effect of indentation size on PSB formation by comparing a standard size indentation with one three times larger. A similar pattern of propagating PSBs and groups of very short bands were found to develop in both cases. The most significant size effect difference noted was that the larger indentation resulted in the earlier formation of PSBs, which occurred after \( 6 \times 10^3 \) cycles compared with \( 9 \times 10^3 \) cycles for the smaller indentation. Also the propagating PSBs were found to reach a greater length near the larger indentation.

3.2.4: Effect of Polishing-back and Annealing Indentations

In general the PSB distributions near indentations which had a reduced depth (3 \( \mu m \)), due to electropolishing after indentation, in crystals with \( t_0 \) 12' from [011], were similar to those which were polished back only lightly (10 \( \mu m \) indentation depth). However a different distribution of short bands was observed (White, 1984; Charsley and White, 1987).
An annealing temperature of 700°C for a number of indentations in crystals with ∠20° from [001] was not found to result in any significant differences in the PSB distributions compared with unannealed indentations, in particular the enhancement remained (Charsley and White, 1987). However, White observed that after annealing some indentations in crystals with ∠12° from [111] at 900°C prior to fatigue, PSBs were observed both at the edges and within S-type indentation pits contrary to the results for unannealed samples. A comparison between annealed and unannealed indentations in specimens with ∠12° from [111] revealed that although some differences in the PSB distributions were apparent, in many respects the PSB distributions developed near the indentations were closely similar. In addition the PSBs were observed earlier near the unannealed indentations compared with the annealed indentations.

White (1984) found that for the case of etch pits, PSBs nucleate at the pit edges but the effect is much less extensive than that which occurs near indentations.

3.3: Indentation Models

In the expanding cavity model of indentation the indenter is considered to be surrounded by a semi-cylindrical or hemispherical core of radius, a. Within this core there is a hydrostatic pressure,
P. Outside the core, stress and displacements are assumed to have radial symmetry. The elastic-plastic boundary lies at a radius $c$, where $c > a$. At the interface between the core and the elastic-plastic zone the following two conditions must be satisfied: within the core, $P$ must be equal to the radial component of the stress in the external zone at radius $a$; the radial displacement of particles lying on the boundary during an increment of penetration must accommodate the volume of material displaced by the indenter.

The basis of the Perrott Model (Perrott, 1977) is an elastic-plastic calculation in which the main mechanism for accommodating the volume of the indenter is assumed to be the radial displacement of the elastic-plastic boundary.

An explanation of the crystallography for ball indentations on the (001) surface of copper is given by the Dyer Model (Dyer, 1965). This model is based on the superposition and interaction of two sets of slip planes. One set is arranged as diverging truncated pyramids and causes a lowering of material below the indenter. The second set surround the first set and form converging truncated pyramids which causes a raising of surface material preferentially at $<110>$ azimuths. The dislocation mechanism causing surface lowering involves using dislocations of two different slip vectors for each plane, and their interaction leaves dislocation segments which intersect on the (001) face. The other major dislocation mechanism in the planes of diverging pyramids is an annihilation reaction that diverts slip downwards, which might be aided by a repulsive
interaction which prevents the outer surface segments of dislocations involved in surface lowering from escaping. The dislocation mechanism for the hill raising also leaves dislocations which intersect on the (001) face, but gives a repulsion of the dislocation segments which limits the spread of the hills. Interactions between converging and diverging pyramids lead mainly to the formation of Lomer-Cottrell locks, but possibly also to short segments of another barrier.

For the orientations of copper crystals used by White (1984), the planes which comprised the pyramids were assumed to be the \{111\} planes which make the largest angle of intersection at the surface. It was arbitrarily considered that the diverging pyramids were effective up to a radius of 2a (where a is half the diagonal length of the indentation). This modification to the Dyer model enabled the crystallography to be taken into account although the radial extent of the deformation could not be deduced from the model.

3.4: Models to Describe PSB Distributions

The enhanced formation of PSBs near indentations in copper is expected to arise from one or more of the following effects: a geometrical stress-raising effect due to the indentation; long-range internal stresses due to deformation around the indenter, and dislocation microstructure effects.
In the case of the etch pits studied in crystals with θ = 20° from [111] (White, 1984) the only contribution to stress enhancement is geometrical. An assumption of elastic isotropy was used by White to calculate the localized surface stresses expected near a hole, since the maximum in the applied fatigue stress is at the surface. These stresses were resolved on to all of the (111)/<110> slip systems for the specimen. Good agreement was found between the positions of the maximum resolved shear stress and the sites of PSB formation. For the case of indentations, in order to achieve a good approximation to geometrical stress enhancement at the surface, Greenspan's calculations (Greenspan, 1944) were used. These calculations were for a square hole with rounded corners in an infinite plate with isotropic elasticity. This is a reasonable approximation for the surface stress enhancement near a pyramidal indentation. The resolved shear stresses were calculated by White (1984) for all slip systems, and this model showed that near annealed indentations (i.e. where no residual stresses are present after the annealing process) not all PSBs nucleated in regions with predicted high stress concentration values. Furthermore, it was found that there was extremely poor correlation between the high stress concentration values and the positions of PSBs near unannealed indentations. Finally on removal of the indentation pit, the general PSB distribution features are found to remain, thus White (1984) suggested that the effect of indentations through geometrical stress raising effects is not attributable to a square hole. Better agreement between theory and experiment is achieved by a hardened core model (Goodier 1933), using an approximation that the
indentation is equivalent to a rigid cylinder of diameter equal to that of the indentation diagonal length.

The contribution from residual stresses was then assessed by White (1984) and Charsley and White (1987). Internal stress calculations were made using a number of models including: Swain and Hagan, (1976); Hill, (1950) and Perrott, (1977). In general terms, compressive stresses are predicted at the surface very close to the indentation, and these are assumed to inhibit PSB formation. It was found that only the Perrott model predicts significant tensile stresses. However the angular distribution of stress was not found to correlate well with observed PSB initiation sites.

White (1984) also considered the combination of the Perrott stress model with the stress concentration expected from a square hole and a circular rigid cylinder. For the case of a circular rigid cylinder, it was found that although the absolute stress values were unreliable, the largest groups of PSBs did correspond to regions in which both the stress range and tensile stress amplitude were close to their maximum values. The square hole model for stress concentration showed less satisfactory correlation. A reason suggested for the lack of correlation between predictions of PSB initiation sites and those observed, is that the extensive plastic relaxation which occurs when the indenter is removed is not allowed for in the models used (Charsley and White, 1985).

The fact that it was not possible to predict the very reproducible
angular distribution of PSB sites, despite a reasonable prediction of the radial PSB distribution being achieved by combining suitable models, led to the recognition of the importance of the dislocation microstructure produced during indenting.

3.5: Dislocation Microstructure

The major role of dislocation microstructures in affecting the observed distribution of PSBs was noted from the residual stress considerations above. It has been suggested by Kuhlmann-Wilsdorf and Nine (1967) that the formation of PSBs is assisted by non-primary slip. In addition, the formation of PSB wall structure from dislocation matrix structure through secondary slip has been proposed by Kuhlmann-Wilsdorf and Laird (1980). Charsley and White (1987) have suggested that a possible mechanism relevant to the studies of unannealed indentations is concerned with the formation of Lomer-Cottrell stair-rod dislocations, as the expected slip systems in some crystals examined are capable of forming such barriers. The limiting of slip distances during fatigue by sessile dislocations gives rise to a localized accumulation of dislocations. As a result of this, enhanced formation of matrix structure may occur which subsequently leads to enhanced formation of PSBs. Such a limitation in slip distance explains a number of features connected with the short groups of PSBs. Propagating PSBs may be thought to be in regions where dislocations are able to move into regions free of dislocation barriers. Alternatively there may be enhancement of the
transformation of the matrix structure into the condensed PSB structure due to localized stresses on a very small scale. At very low strain amplitudes, well away from the indentation, the conversion of matrix structure into wall structure is suggested to occur due to the stress concentration at the tip of the PSBs.
CHAPTER 4

EXPERIMENTAL TECHNIQUES

4.1: Growth of Al Single Crystals

Al single crystals were grown by a Bridgeman technique. High purity (99.99%) Al which had been cold rolled was cut to the shape shown in Fig. 4.1 to fit split graphite moulds. Specimens were cleaned using a solution of HCl/H₂O/HNO₃/HF in the ratio 9:5:3:2 (Barrett and Massalski, 1980) before being placed into the moulds, which were then bolted together and put inside an alumina tube. The alumina tube was positioned inside a vertical furnace (Johnson Mathey type V44A) such that the specimen was initially held above its melting point temperature (660°C) and was lowered through the furnace at a rate of 1.5 cm/hr. The lower end of the furnace was opened in order to achieve a suitable temperature gradient. A rotary pump was used to maintain a low pressure (0.2 Torr) within the alumina tube throughout the growth process.

4.2: Single Crystal Orientations

The single crystals were etched using the HCl/H₂O/HNO₃/HF etchant before being oriented by the Laue back reflection technique. The directions parallel to the edges of the specimen are indicated by \( t \).
and $t_2$, whilst the tension-compression axis is denoted by $t_0$ (see Fig. 4.2 a). Fig. 4.2 b shows the position of $t_0$ in the standard stereographic triangle for the orientations of the crystals used in Chapter 6.

4.3: Initial Preparation of Polycrystalline and Single Crystal Specimens

The surfaces of single crystal specimens were prepared by mechanically polishing using emery papers followed by diamond lapping compounds. A 35% nitric acid/methanol solution held at $-10^\circ$C was used to electropolish both the polycrystalline specimens which were cut from 90 $\mu$m thick 99.99% purity Al, and the single crystals. Specimens were then usually annealed for 1 hour at 425°C, although in some cases an annealing temperature of 550°C was used. During annealing, the pressure was maintained at $< 10^{-4}$ Torr.

4.4: Introduction of Surface Damage into Single Crystals

The surfaces of single crystals were indented at a number of points close to the specimen axis using a Vickers diamond indenter. This indenter is a square based pyramid (136° included angle between opposite faces) and the depth to diagonal ratio is approximately 1:7. An indenter load of 30gm was used to produce indentations which had a diagonal length $\sim 60\mu$m, and which were $\sim 8\mu$m deep.
Indentations were made such that either their edges were approximately parallel to $t_0$ (S-type), or their diagonal was approximately parallel to $t_0$ (D-type), as shown in Figs. 4.3 a, b. For details of indentations made in single crystal specimens see Table 4.1.

Etch pits were introduced by electrolytic polishing. The pits had sizes in the range 3 $\mu$m to 20 $\mu$m.

The positions of the indentations on the crystal surface are specified in terms of $l$, $\phi$ co-ordinates as defined in Fig. 4.4.

Positions around indentations/etch pits were defined in terms of the distance, $r$, from their centre, and an angle, $\theta$, measured relative to $t_0$ (see Figs. 4.3 a, b).

4.5: Additional Treatments of Indentations

In several cases, four indentations were annealed before making four additional indentations, to enable the PSB distribution developed near annealed and unannealed indentations to be compared on the same specimen. The annealing temperatures used were either 425°C or 550°C (see Table 4.1), for a period of 1 or 2 hours respectively.

A number of crystals, C3, C6 and C7, were electropolished by various amounts after indentation (see table 4.1).
The annealing and electropolishing treatments described above were combined for some indentations in specimens C3 and C6.

4.6: Preparation of Indented Discs for Microstructural Investigations

A Vickers diamond indenter was used to produce indentations of diagonal length (d) 56\(\mu\)m (30gm load) and 36\(\mu\)m (10gm load) in the single crystal and polycrystalline specimens respectively. These specimens were annealed at either 425 or 550 °C prior to indenting.

Some of the indentations in the polycrystalline material were annealed at 550°C for two hours, in order that a comparison between the dislocation structure near an unannealed and an annealed indentation could be made. In one case, an unannealed and an annealed indentation were introduced within the same grain, to enable a comparison to be made within a crystal of a given orientation.

4.7: Fatigue Techniques

4.7.1: Fatigue of Polycrystalline Al at Room Temperature and at Low Temperature

Polycrystalline Al foil strips were fatigued using a technique previously developed in which foils are glued with "Durofix" to an
Al alloy beam, which is cycled in reverse bending in air (at room temperature), Charsley and Robins (1974). Total strain amplitudes in the range $+4 \times 10^{-4}$ to $+1.9 \times 10^{-3}$ were used, corresponding to saturation plastic strain amplitudes of $+1.9 \times 10^{-4}$ to $+1.69 \times 10^{-3}$ (see Table 4.2). These total strain amplitude values were previously determined by Robins (1972) using a dial gauge extensometer. Foils were subsequently removed from the beam for examination by optical microscopy, scanning electron microscopy and for TEM preparation, by soaking in an acetone bath.

The technique outlined above was modified to fatigue Al specimens at low temperature, 77 K, by maintaining a jet of liquid nitrogen onto the surface of the specimen throughout fatigue cycling. Total strain amplitudes in the range used for room temperature fatigue were used. Furthermore every attempt was subsequently made to maintain the specimen temperature at a low value where possible. The specimens, which were 3 mm discs cut by spark erosion, were removed from the beam by soaking in acetone cooled with liquid nitrogen to a temperature of $\sim -50^\circ$C, and specimens were stored in liquid nitrogen both before and after the preparation of TEM discs.

4.7.2: Fatigue of Indented Single Crystals

Specimens were fatigued in reverse plane bending between fixed total strain limits, using a system previously developed by White (1984). Specimens were initially hardened using a low amplitude wheel, $W_0$ ($\epsilon_t = 1 \times 10^{-4}$, where $\epsilon_t$ is the strain at the surface of the
crystal), or $W_1$ ($\varepsilon_t = 2 \times 10^{-4}$), and were then fatigued using either $W_1$ or $W_2$ ($\varepsilon_t = 4 \times 10^{-4}$). The fatigue cycling of specimens was interrupted at frequent intervals, and the specimens were examined by optical microscopy and by scanning electron microscopy. The number of cycles was in the range $5 \times 10^2$ to $8 \times 10^4$. Cycling could not be continued to fracture since the narrow end of the specimen became distorted after a large number of cycles.

The total strain amplitude values for single crystals were calculated by using a travelling microscope to measure the deflection ($h$) at the point B in Fig. 4.5. Since the load was applied close to the point A in this figure, it may be assumed that the specimen was bent into a uniform radius of curvature. Thus the strain may be calculated using the expression,

$$\frac{th}{l_c^2}$$

where $t$ is the specimen thickness and $l_c$ is the distance between the clamps. The values for the total strain amplitude are tabulated for wheels $W_0 - W_2$ in Table 4.3.

4.7.3: Analysis of Strain into Plastic and Elastic Components at Saturation

The value of the saturation plastic strain amplitude, $\varepsilon_p$, can be
calculated from the expression,

$$\varepsilon_p = \left( \varepsilon_t - \sigma_o \right) \frac{1}{E}$$  \hspace{1cm} (see Fig. 4.6)

where $\varepsilon_t$ is the total strain amplitude, $\sigma_o$ is the saturation stress amplitude and $E$ is Young's Modulus.

From data presented by Pettersen (1984), $\sigma_o \approx 15$ MPa, using this information an approximate analysis of the total strain amplitude into plastic and elastic components may be carried out. A value for $E$ for Al of $7 \times 10^4$ MPa has been used in the calculations.

Table 4.2 and 4.3 list the saturation plastic strain amplitudes used for polycrystalline and single crystal material respectively.

The value of the cumulative plastic strain after $N$ cycles at saturation, $\varepsilon_{pl, cum}$, may be determined, and is given by the formula;

$$\varepsilon_{pl, cum} = 2N\Delta\varepsilon_p$$

where $\Delta\varepsilon_p$, the plastic strain range, is given by $2\varepsilon_p$ (see Fig. 4.6).

The values for $N$ used in the calculations of the cumulative plastic strain presented in Table 4.2, are the total numbers of cycles given to the specimen. Hence differences in the plastic strain amplitudes during cycles preceeding saturation have not been taken into account.
in the above $\epsilon_{pl, cum}$ values.

4.8: Preparation of TEM Specimens

4.8.1: Preparation of TEM Specimens from Fatigued Polycrystalline Al

Discs of 3mm diameter were cut from the polycrystalline material by spark erosion, using a Materials Science Spark Erosion Unit Mk 2. Both the polycrystalline material fatigued at 293 K, and that fatigued at 77 K were then prepared for TEM by jet thinning using a 35% nitric acid/methanol solution held at $-10^\circ$C. In a number of cases the surface of the specimen was related to the underlying dislocation structure by jet thinning from one side only, and by comparing low magnification TEM micrographs with optical micrographs (and in some cases with SEM micrographs). Further the tension-compression axis was related to TEM micrographs for some specimens by cutting discs which had a straight section parallel to this axis, and by comparing low magnification TEM and optical micrographs.

4.8.2: Preparation of Indented discs for TEM

Before cutting discs around the indentations in the polycrystalline and single crystal material by spark erosion, a section was spark eroded from the indented region in the single crystal and its thickness was reduced to $\sim 0.2 \mu\text{m}$, by mechanically polishing the
non-indented side only. Discs were then prepared for TEM by jet-thinning, generally from the non-indented side only. This usually gave rise to a perforation which covered a major part of the indentation but enabled the dislocation structure in the resulting thin areas to be related to the indentation edges by using appropriate low magnifications.

4.9: Microscopy

All optical micrographs were taken using a Reichert "zetopan" microscope.

A Cambridge Stereoscan 250 scanning electron microscope operated at 20 kV was used for all SEM studies.

All TEM specimens were examined using a JEOL 200CX microscope operated at 200 kV. Bright field, dark field and weak beam techniques have been used.
CHAPTER 5

FATIGUE OF Al AT ROOM TEMPERATURE AND AT 77K

5.1: Introduction

There have been few systematic studies of the dislocation configurations in fatigued Al and none in the last 17 years. In order to assess the effects of surface damage on fatigue it has therefore been necessary to characterise the dislocation microstructure in Al fatigued at different amplitudes at room temperature. There have been detailed studies on the dislocation configurations in fatigued Cu at room temperature, however the differences between Al and Cu are very considerable. For this reason observations have also been made at liquid nitrogen temperature. Initially Al specimens fatigued at 77 K were aged for various periods at room temperature, subsequently every attempt was made to maintain specimens at as low a temperature as possible at all stages (as outlined in section 4.7.1).

5.2: Microscopy of Al Fatigued at Room Temperature

The development of PSBs on a polycrystalline Al specimen was
examined with increasing numbers of cycles (N) in the range \(2 \times 10^4\) to \(2.2 \times 10^5\) at \(\varepsilon_t = \pm 4 \times 10^{-4}\) at room temperature (see Fig. 5.1a–d). It is apparent that with increasing numbers of cycles the PSBs become more pronounced and are found to spread across the grains.

Different types of surface deformation markings consisting of "rumpled bands", similar to those found by King and Teer (1969), and regions exhibiting a cross-hatched structure similar to that observed by Forsyth (1956) and King and Teer (1969), are marked A and B respectively on Fig. 5.2. These structures were apparent after \(1.8 \times 10^5\) cycles at \(\varepsilon_t = \pm 4 \times 10^{-4}\) at room temperature.

The surface appearance after \(N = 5 \times 10^3\) at \(\varepsilon_t = \pm 6 \times 10^{-4}\) is shown by the optical micrograph Fig. 5.3 revealing PSBs and "rumpled bands". An example of the surface appearance of a specimen after \(1 \times 10^4\) cycles at \(\varepsilon_t = \pm 1.4 \times 10^{-3}\) followed by \(N = 3 \times 10^3\) at \(\varepsilon_t = \pm 1.9 \times 10^{-3}\) is shown in Fig. 5.4. Approximately 80% of the grains are found to contain PSBs after fatigue testing as described above. By comparison, in specimens fatigued for \(5 \times 10^3\) cycles at \(\varepsilon_t = \pm 6 \times 10^{-4}\), and in specimens fatigued for \(2 \times 10^4\) cycles at \(\varepsilon_t = \pm 4 \times 10^{-4}\) approximately 75% of the grains are found to contain PSBs.

A low magnification SEM micrograph (Fig. 5.5a) illustrates regions with cross-hatched markings separated by regions free of surface deformation markings which were observed after \(5 \times 10^3\) cycles at \(\varepsilon_t = \pm 1.4 \times 10^{-3}\). The dark circular features are pits produced by electropolishing, and modifications in the surface slip markings in
the vicinity of these pits are apparent. A higher magnification micrograph of a region in this area, Fig. 5.5 b, shows an arrangement of circular features which are below the surface.

The dislocation structures developed in various specimens after \( N = 2 \times 10^4 \) at \( \varepsilon_t = \pm 4 \times 10^{-4} \) are shown in Figs. 5.6 - 5.13. In total approximately forty grains were examined by TEM, and the micrographs presented are typical of the structures observed after fatigue at this amplitude. In Fig. 5.6 dislocations can be seen to have formed into irregular walls approximately 0.8 \( \mu \text{m} \) apart and parallel to [\( \bar{1}10 \)]. Dislocations extend from the walls approximately parallel to [110]. The stereo-pair indicates that the walls appear to be perpendicular to the plane of the micrograph so the walls are approximately parallel to the (110) planes, and that the dislocation loops are distributed throughout the foil thickness. Dislocations of all six Burgers vector types are present in the area. Dislocations in the groupings which are approximately parallel to [110] have mainly \( b = \frac{1}{2}[110] \) (which is the prevalent Burgers vector in the area) and thus these are approximately screw dislocations. Dislocations in the walls parallel to [010] (i.e. (100) planes) have \( b = \frac{1}{2}[110] \) and \( b = \frac{1}{2}[\bar{1}10] \).

In Fig. 5.7 the dislocations have formed walls approximately parallel to [110] and [010] (i.e. (\( \bar{1}10 \)) and (100) planes), with dislocations crossing between the walls approximately parallel to [\( \bar{1}10 \)] and [100] apparent. Dislocation loops are shown by the stereo-pair to be distributed at various depths. Variations in
contrast for $g_{200}$ indicate misorientation across the walls which are approximately 0.7 $\mu$m apart. Dislocations of all six Burgers vector types are present in the area, however the majority of dislocations in the walls have $b = \frac{1}{2}[110]$ and $\frac{1}{2}[10\bar{1}]$. Dislocations which extend across the walls have $b = \frac{1}{2}[110]$, $\frac{1}{2}[10\bar{1}]$ and $\frac{1}{2}[110]$. In the channels, many of the small dislocation loops have $b = \frac{1}{2}[110]$. By comparing optical micrographs and low magnification TEM micrographs the dislocation structure may be related to surface features and is found to underlie a surface containing PSBs (which are apparent over the entire grain). A large fraction of grains in this specimen were found to exhibit PSBs.

Fig. 5.8 is a further example where the dislocations have formed into very ragged walls approximately parallel to [110] (i.e (110) planes), with dislocations crossing approximately parallel to [100] and [010]. Fig. 5.9 is a stereo-pair of the region in Fig. 5.8 at higher magnification. The walls of dislocations have a width of $\sim 0.7$ $\mu$m and the channel spacing is $\sim 0.8$ $\mu$m. Thus, the walls have a similar spacing to those in previous examples, however the dislocation density in the walls appears much higher. Dense dislocation patches separated by regions of low dislocation density are apparent, and there is also a large number dislocation loops in the low dislocation density region. Dislocations with at least four Burgers vectors are present in the area. Most of the dislocations extending from the walls (marked k, Fig. 5.8) have Burgers vectors $b = \frac{1}{2}[01\bar{1}]$ or $\frac{1}{2}[011]$ although the dislocation marked $b$ has $b = \frac{1}{2}[110]$. Many of the dislocations present in the walls have $b = \frac{1}{2}[110]$. 
Figs. 5.10 and 5.11 are further examples of areas with large patches of high dislocation density, separated by channels approximately parallel to \([100]\) and \([010]\) which are of lower dislocation density. The channels are approximately 1 - 2 \(\mu m\) wide and contain a number of dislocation loops. Some channelling of dislocations parallel to \([110]\) is apparent in these figures but this is not developed and the channel widths are noticeably less than those of \(<100>\) channels. Dislocations which cross between the patches are also apparent. The direction of the tension-compression axis during fatigue testing is indicated on Fig. 5.11. The slip systems which have the highest Schmid factors, and therefore the maximum resolved shear stresses, are \((111)\ [101]\) and \((111)\ [101]\) (see Appendix A). Burgers vector analysis of dislocations in Fig. 5.11 shows that Burgers vectors present are: \(b = \{110\} or \{110\}\), \(b = \{101\} or \{101\}\), and \(b = \{011\} or \{011\}\) (which correspond to slip systems with highest Schmid factors of 0.25, 0.49 and 0.25 respectively). Examples of dislocations with Burgers vectors \(b = \{011\} or \{011\}\) are marked \(k\) on Fig. 5.11. The majority of dislocations in the dense patches have \(b = \{110\} or \{110\}\).

Fig. 5.13 is a higher magnification micrograph of an area in Fig. 5.12 showing the large number of dislocation loops observed in the walls. Dislocations have formed into walls approximately parallel to \([110]\) (corresponding to \((110)\) planes), which are separated by \(~1\ \mu m\) wide channels of relatively low dislocation density. All six Burgers vector types are present in the area, the most common dislocation
type in the patches is $b = \overline{4}[1\bar{1}0]$. Dislocations extending into the
lower dislocation density channels have $b = \overline{4}[0\bar{1}1]$ or $\overline{4}[0\bar{1}1]$.

Figs. 5.14 - 5.16 show typical examples of dislocation structure
developed after $5 \times 10^3$ cycles at $\epsilon_t = \pm 6 \times 10^{-4}$. Approximately
forty grains were examined, subsequently areas which were
representative of the types of structures observed were examined in
detail. In Fig. 5.14 dislocations have formed loose tangles with a
large number of dislocation loops apparent. In Fig. 5.15
dislocations have formed quite dense tangles. There is some grouping
of dislocations approximately parallel to [010], [110], [100] and
[\bar{1}\bar{1}0] directions in Figs. 5.14 - 5.16. In Fig. 5.16 the structure
consists of dislocation loops and walls. The channels between these
walls are ~1 $\mu$m wide and are relatively free of dislocations.

Misorientation across the walls is indicated by the change in
contrast observed for $\overline{2}0\overline{2}0$ operating reflection. The small
dislocation loops in areas marked $b$ are out of contrast for $\overline{2}2\overline{2}0$ and
hence have Burgers vectors $b = \overline{4}[1\bar{1}0]$.

A total of four specimens were prepared from material fatigued for $N$
$= 2 \times 10^4$ cycles at $\epsilon_t = \pm 6 \times 10^{-4}$. An example of the type of
dislocation structure observed is shown in Fig. 5.17. For this
specimen the direction of the tension-compression axis during
fatigue testing is indicated. The slip systems which have the
highest Schmid factors and therefore the maximum resolved shear
stresses are (111) [0\bar{1}1] and (\bar{1}\bar{1}0) [0\bar{1}1] (see Appendix A). A channel
which is elongated approximately parallel to [1\bar{1}0] can be seen
between the dislocation tangles. Dislocations marked $b$ have Burgers vectors $\mathbf{b}[\{110\}]$ (corresponding to slip systems with a Schmid factor of 0.36) and those at $k$ have $\mathbf{b} = \mathbf{b}[\{011\}]$ or $\mathbf{b}[\{0\bar{1}1\}]$ (which correspond to slip systems with a highest Schmid factor of 0.48). There are also dislocations with $\mathbf{b} = \mathbf{b}[\{101\}]$ or $\mathbf{b}[\{10\bar{1}\}]$ in the area (corresponding to slip systems with a highest Schmid factor of 0.12).

Figs. 5.18 and 5.19 show typical examples of the dislocation structure developed after $5 \times 10^3$ cycles at $\varepsilon_t = \pm 1.4 \times 10^{-3}$. Fifteen specimens were prepared and ten of these were examined in detail by TEM after fatigue at this strain amplitude. Usually approximately ten grains were inspected in each specimen before selecting representative areas to be studied. The dislocation arrangements formed after fatigue at this strain amplitude show notable differences in comparison with those found after lower strain amplitude fatigue. Representative examples of the types of structures found (Figs. 5.18 and 5.19) show that dislocations have formed into narrow bands of high dislocation density which enclose areas which are relatively free of dislocations, i.e. a fatigue cell structure has been formed. These cells are elongated and are typically 0.6 $\mu$m wide with a wall width of $\sim 0.1 \mu$m. Dislocations crossing between the walls approximately parallel to $[110]$ and some dislocation loops are apparent. Burgers vector analysis of the area in Fig. 5.19 showed that dislocations marked $b$ have Burgers vectors $\mathbf{b}[\{110\}]$. Some loops in the area marked $m$ (Fig. 5.19) are out of contrast for $\mathbf{b}[\{020\}]$ and thus have Burgers vectors $\mathbf{b} = \mathbf{b}[\{101\}]$ or $\mathbf{b}[\{10\bar{1}\}]$. Since the dislocation walls are evidently very narrow, it
may be assumed that they lie in the plane which contains the [001] direction (and therefore these correspond to (100) planes).

Fig. 5.20 shows a typical example of the development of narrow dislocation walls after $5 \times 10^3$ cycles at $\varepsilon = \pm 1.4 \times 10^{-3}$. In one cell a small amount of misorientation is indicated by the change in contrast observed across the dislocation walls.

Fig. 5.21 a shows an additional example of the dislocation structure developed after $5 \times 10^3$ cycles at $\varepsilon = \pm 1.4 \times 10^{-3}$. For this specimen the dislocation structure has been related to the surface appearance using the technique outlined in section 4.8.1, and was found to lie beneath a region with cross-hatched markings (Figs. 5.21 b, c). These cross-hatched markings have a periodicity of $\sim 2 \mu m$. Furthermore this structure can be seen within the polishing pits in Fig. 5.21 c. Burgers vector analysis was carried out for this region and examples of dislocations with $b = \overline{410}$, $\overline{410}$ and $\overline{411}$ are marked a, b and e respectively on Fig. 5.21 a. At c there are dislocations which are visible under $g_{111}$ which are not apparent for $g_{111}$ and $g_{020}$ indicating $b = \overline{410}$. At d dislocations apparent for $g_{111}$ are out of contrast for $g_{020}$ and $g_{111}$ indicating $b = \overline{410}$, and at f dislocations which are visible under $g_{111}$ and $g_{111}$ are out of contrast for $g_{200}$ indicating the presence of $b = \overline{411}$. Thus all six possible Burgers vector types are present in the area.

Figs. 5.22 a, b show variations in the types of dislocation arrangements which were found to underlie regions of similar,
although less regular, cross-hatched appearance to that shown in Fig. 5.21 b (see Fig. 5.22 c). It should be noted that in total four specimens contained perforations in regions containing cross-hatched surface markings. The micrographs presented are typical of the structures found in such areas.

The dislocation structure developed after $1 \times 10^4$ cycles at $\varepsilon_t = \pm 1.4 \times 10^{-3}$ followed by $3 \times 10^3$ cycles at $\varepsilon_t = \pm 1.9 \times 10^{-3}$ was examined in five specimens and an example is shown in Fig. 5.23. This structure consists of narrow curved boundaries forming a more equiaxed cell structure than previously observed at lower strain amplitudes. Using a $g \cdot b = 0$ criterion, dislocation loops at c have Burgers vectors $b = \mathbf{M}[101]$. The dislocation patch at B appears to consist of dislocations which have two different Burgers vectors.

5.3: Microscopy of Al Fatigued at Low Temperature with Specimen Ageing at Room Temperature

Initially, four specimens which had been fatigued at $\varepsilon_t = \pm 4 \times 10^{-4}$ at low temperature (77 K) followed by various periods of ageing at room temperature were examined. A typical example of walls of dislocations observed after seven days ageing at room temperature after fatigue at 77 K for $2 \times 10^4$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$ is shown in Fig. 5.24. Walls of dislocations approximately parallel to [110] and [201] directions (i.e. corresponding to (111) and (131) planes) can be seen in this area. Fig. 5.25 shows the dislocation structure
in a specimen which was aged for two months after fatigue at low temperature. Regions exhibiting large areas of dense dislocation loop patches separated by regions which are relatively dislocation free, very similar in appearance to matrix structure observed after room temperature fatigue in Cu, can be seen. These relatively dislocation free channels are found to contain isolated dislocation loops. The channels are approximately parallel to [110] and [01\bar{1}] directions. Fig. 5.26 shows the region at higher magnification and the weak-beam dark field image (Fig. 5.27) shows the high density of elongated dislocation loops apparent in the dislocation dense patches. Burgers vector analysis showed that dislocations within the dense patches marked A on Fig. 5.25 have \( \mathbf{b} = \mathbf{M}[101], \mathbf{M}[10\bar{1}], \mathbf{M}[011] \) and \( \mathbf{M}[01\bar{1}] \), although \( \mathbf{b} = \mathbf{M}[01\bar{1}] \) and \( \mathbf{M}[10\bar{1}] \) appear to be predominant. Dislocations marked d extending from these patches have \( \mathbf{b} = \mathbf{M}[10\bar{1}] \). At c in Fig. 5.26 most dislocations are out of contrast and thus have \( \mathbf{b} = \mathbf{M}[101] \). After similar ageing times at room temperature (approximately 2\( \times \) months) matrix type structure was observed (Fig. 5.28) in addition to wall type dislocation structures (Fig. 5.29) resembling those in Fig. 5.24. In the matrix type structures, channels approximately parallel to [13\bar{1}] and [31\bar{1}] are apparent. The walls observed in Fig. 5.29 are roughly parallel to [100] and [33\bar{1}] (corresponding to (010) and (33\bar{1}) planes).

Following an increase in the number of fatigue cycles to \( 2.2 \times 10^4 \) at \( \ddot{\epsilon}_f = + 4 \times 10^{-4} \) four specimens were examined, and elongated cell structures were observed with dislocations crossing between the walls apparent after seven days ageing at room temperature (Fig.
5.30). The cell walls are approximately parallel to [33\overline{1}] and [1\overline{3}1] (i.e. (\overline{3}31) and (31\overline{1}) planes). Arrangements of dislocations approximately parallel to [02\overline{1}] and [201] (corresponding to (311) and (13\overline{1}) planes) are evident after four weeks ageing at room temperature following fatigue under these conditions (Fig. 5.31). Dislocations in this area extending from the dislocation walls which are visible for g=311, are out of contrast for g=111 showing that these have \( b = \frac{1}{2}[110] \) or \( \frac{1}{2}[10\overline{1}] \) or \( \frac{1}{2}[011] \). Furthermore, misorientation between these walls is indicated by the changes in contrast between them.

5.4: Microscopy of Al Fatigued at Low Temperature with Specimen Ageing Minimized

After fatigue for \( N = 2.2 \times 10^4 \) at \( \varepsilon_t = \pm 4 \times 10^{-4} \) with the specimen maintained at low temperature prior to TEM examination, the elongated cell dislocation structure in Fig. 5.32 was found. The cell walls are approximately parallel to [110] and [1\overline{1}1] (i.e. (12\overline{2}) and (110) planes). This structure is of the same type as found in specimens which were aged at room temperature after low temperature fatigue under the same conditions.

After increasing the strain amplitude to \( \varepsilon_t = \pm 6 \times 10^{-4} \) for \( N = 5 \times 10^3 \) four specimens were prepared and these were again maintained at a low temperature until TEM examination. Fatigue under these conditions was usually found to give rise to the matrix type
structures shown in Fig. 5.33, which are similar to those observed after 2 x 10^4 cycles at \( \varepsilon_t = \pm 4 \times 10^{-4} \) after two months ageing time at room temperature. A large number of small dislocation loops and pairs of approximately parallel dislocations are evident in Fig. 5.34 at higher magnification. Elongated dislocation loops are apparent in the weak-beam dark field image shown in Fig. 5.35. Areas exhibiting a lower density of dislocation tangles were also found in some areas. In Fig. 5.36 an example of a specimen which exhibited dislocation tangles after fatigue under these conditions is shown.

After examining three specimens fatigued at 77 K for \( N = 1.015 \times 10^4 \) at \( \varepsilon_t = \pm 8 \times 10^{-4} \), in addition to matrix type structures (Figs. 5.37 to 5.40), areas with regions containing both wall and matrix structures resembling those found in Cu after room temperature fatigue (Figs. 5.41, 5.42), and incompletely formed labyrinth structures (Figs. 5.43 to 5.45) were found. Matrix type structures were observed in about 30% of the grains examined, whereas labyrinth type structures were observed in about 60% of grains, and in one grain an area exhibiting wall and matrix type structures was found. The shape of the dislocation patches in the matrix structure was found to vary in different grains as shown in Figs. 5.37 and 5.40. Considering Figs. 5.38 and 5.39 the dislocation patches and channels tend to be elongated approximately parallel to [111] and occasionally strings of dislocation loops are apparent. Small dislocation free patches ~ 0.15 \( \mu m \) in size can be seen within the dense patches. Weak-beam dark field imaging of an area in Fig. 5.37 (Fig. 5.39) shows more clearly the high density of small dislocation
loops present. In one specimen, the wall and matrix structures shown in Fig. 5.41 were found. The walls in this region are approximately parallel to [010], [120] and [210] (corresponding to (100), (210) and (120) planes). The channel separation between both the (210) and (120) walls is ~ 2 µm whereas for the (100) walls the separation is ~ 3 µm. Examples of labyrinth type structure were frequently observed, as noted above. In the example of labyrinth structure shown in Fig. 5.43, slip lines approximately parallel to [110] and [1\bar{1}0] apparent in the dislocation free regions between the labyrinth walls indicate that some dislocation movement has occurred. There is a tendency for square dislocation free zones to be bounded by the (100) walls, and although the walls approximate to ⟨100⟩ directions there is some deviation of the walls away from these directions. The width of the low dislocation density channels between the walls is ~ 2 µm. Burgers vector analysis of an area near this region (see Fig. 5.44) indicated the presence of all six Burgers vector types. Examples of dislocations which have \( b = 4[110], 4[1\bar{1}0], 4[011] \) and \( 4[01\bar{1}] \) are marked a, b, e and f respectively on Fig. 5.44. At c and d dislocations which have \( b = 4[101] \) and \( 4[10\bar{1}] \) respectively are apparent for \( S_{220} \). Many of the dislocation loops at a are out of contrast for \( S_{220} \) indicating that these have \( b = 4[110] \). Another example to show the extent of labyrinth structure is shown in Fig. 5.45. The apparently featureless surface regions corresponding to the micrographs in Figs. 5.38, 5.40, 5.41 and 5.45 are marked A, B, C and D respectively on Fig. 5.46, which is an optical micrograph of the specimen.
After examining two specimens fatigued for \( N = 7.63 \times 10^3 \) at \( \varepsilon_t = \pm 1.0 \times 10^{-3} \) only an ill-defined labyrinth type structure was observed (Figs. 5.47 to 5.49). Burgers vector analysis revealed that dislocations extending from the labyrinth walls and in the channels, marked \( a_1 - a_4 \) in Fig. 5.47, have \( \mathbf{b} = \mathbf{M}[110] \). Loop \( a_2 \) is an example of a dislocation loop which has been pulled out of the dislocation wall.

Four specimens were prepared on increasing the strain amplitude to \( \varepsilon_t = \pm 1.4 \times 10^{-3} \) and two specimens (i.e. approximately twenty grains) were examined in detail. Areas which contained very few dislocations were occasionally observed (Figs. 5.50, 5.51), in addition to matrix (Figs. 5.52 and 5.53), condensed matrix-type (Figs. 5.54 - 5.57), ladder-type (Figs. 5.58, 5.59, 5.61 and 5.62) and labyrinth (Figs. 5.63, 5.64, 5.66, 5.67, 5.69, 5.70 and 5.72 a to 5.74) structures. In the area in Fig. 5.51 which contains few dislocations, Burgers vectors analysis indicates that dislocations present include those which have \( \mathbf{b} = \mathbf{M}[011] \) or \( \mathbf{M}[01\overline{1}] \), \( \mathbf{M}[101] \) or \( \mathbf{M}[10\overline{1}] \) and \( \mathbf{M}[110] \). Examples of the latter type of Burgers vector are marked \( a \) on Fig. 5.51. Dislocations marked \( c \) have \( \mathbf{b} = \mathbf{M}[101] \). Matrix structures were observed less often after fatigue at this strain amplitude than after lower amplitude fatigue. Moreover, the proportion of channel was often found to be greater at this higher strain amplitude forming a more condensed type of structure. In the matrix structure, the low dislocation density channels are again found to have a significantly higher density of isolated dislocation loops (see for example Fig. 5.52) than for Cu fatigued at room
temperature (Winter et al, 1981). Burgers vector analysis of the region in Fig. 5.52 showed that in the channels between the dense dislocation patches many dislocations are out of contrast for $g_{131}$ and $g_{111}$ indicating $\mathbf{b} = \mathbf{M}[101]$. The dislocation marked p in Fig. 5.53 has $\mathbf{b} = \mathbf{M}[110]$ or $\mathbf{M}[10\bar{1}]$. Within the dislocation patches, the presence of dislocations with $\mathbf{b} = \mathbf{M}[1\bar{1}0]$, $\mathbf{M}[01\bar{1}]$ and $\mathbf{M}[101]$ is indicated by dislocations out of contrast for $g_{220}$, $g_{311}$, and $g_{131}$. In Fig. 5.56 the disappearance of many small dislocation loops present in the channels between the dense dislocation patches for $g_{311}$ indicates that these have $\mathbf{b} = \mathbf{M}[011]$. The dislocation loop marked e in Fig. 5.57 is also of this type. Examples of dislocations with $\mathbf{b} = \mathbf{M}[110]$ and $\mathbf{M}[1\bar{1}0]$ are marked a and b respectively in Fig. 5.57. Burgers vector analysis of the dense dislocation patches in this area revealed the presence of dislocations with $\mathbf{b} = \mathbf{M}[01\bar{1}]$, $\mathbf{M}[110]$ and $\mathbf{M}[1\bar{1}0]$. Fig. 5.58 shows a region (denoted by dashed lines) which is similar in appearance, although more irregular, to the "ladder" structure which has been reported after room temperature fatigue of Cu. This type of structure was seldom observed (only on two occasions). The dislocation walls, denoted by arrows, are in this case found to be approximately parallel to [02\bar{1}] (corresponding to (311) planes) and there are suggestions of cell structure in some parts of the area. Burgers vector analysis of the area in Fig. 5.59 revealed that many of the small dislocation loops present within the channels and some dislocations in the walls are out of contrast for $g_{220}$ and $g_{522}$ indicating that these have $\mathbf{b} = \mathbf{M}[\bar{1}0\bar{1}]$ and $\mathbf{M}[011]$. Examples of dislocations with $\mathbf{b} = \mathbf{M}[\bar{1}0\bar{1}]$ are marked b and most of the dislocations in this area are of this type.
The dashed lines in Fig. 5.61 denote a second region which approximates to the ladder structure. Analysis of the area in Fig. 5.61 showed that the dislocations which cross the channels of the structure have a single value of the Burgers vector and are approximately of screw orientation; the walls, which are not completely planar, are approximately normal to the active Burgers vector. Near the centre of this micrograph is a region which can be interpreted as uncondensed matrix structure. The separation of the walls is approximately 2.8 μm i.e. about four times the spacing of the ladder structure in Cu fatigued at 77 K (Basinski, Korbel and Basinski, 1980) or about twice the wall spacing in Cu fatigued at room temperature (Winter, 1973). The apparently featureless surface structure corresponding to the ladder-type structure region in Fig. 5.58 is shown in Fig. 5.60.

A typical example of labyrinth structure which is not wholly formed after fatigue at this strain amplitude is shown in Figs. 5.63, 5.64, 5.66 and 5.67. This structure consists of well defined relatively dislocation free channels about 0.7 μm in width, with dislocation walls approximately parallel to {100} planes but which deviate significantly. The structures in Fig. 5.66 are very similar to those observed in earlier micrographs (e.g. Fig. 5.43) however the channels are much narrower at this higher strain amplitude. The corresponding featureless surface regions are shown in Fig. 5.65 and 5.68. On a few occasions the {100} walls were found to be less regular (see Fig. 5.69 and Fig. 5.70) and these appear to resemble the "parquet" type labyrinth structure observed by Mecke and
Blochwitz (1982) in some Ni specimens fatigued at room temperature. The parquet structure consists of edge dislocation walls which are irregularly arranged at angles of 45° and 135° to the primary slip direction building up a parquet-like structure (Mecke and Blochwitz, 1982). In Ni the walls in the parquet-like structure are parallel to (521) and (125) planes, whereas in Al the walls in Fig. 5.69 are parallel to (100) and (011) planes, whilst in 5.70 they are parallel to (031) and (100) planes. The width of the channels in this structure in Al is \( \approx 0.7 \ \mu m \) and the apparently featureless appearance of the surface corresponding to the structure is shown in the optical and SEM micrographs, Figs. 5.71 a – c. Burgers vector analysis of a region exhibiting labyrinth structure which is not completely condensed is shown in Figs. 5.72 a – f. In this area four Burgers vector types, \( \mathbf{b} = \mathbf{h}[110], \mathbf{h}[110], \mathbf{h}[101] \) and \( \mathbf{h}[10\bar{1}] \), were identified and examples are marked a, b, c, and d respectively on Figs. 5.72 a, c and d. Dislocations were found predominantly to have \( \mathbf{b} = \mathbf{h}[110] \) and \( \mathbf{h}[110] \). Most of the dislocations present in the walls approximately parallel to (010) are out of contrast for \( \mathbf{g}_{111} \) and \( \mathbf{g}_{220} \) indicating \( \mathbf{b} = \mathbf{h}[110] \). Many of the dislocation loops in the channels visible under \( \mathbf{g}_{200} \) and \( \mathbf{g}_{020} \) are out of contrast for \( \mathbf{g}_{220} \) indicating that these also have \( \mathbf{b} = \mathbf{h}[110] \). A typical example of more condensed labyrinth structure is shown in Fig. 5.73 and 5.74. The channel spacing in this area (\( \approx 2 \ \mu m \)) is much larger than in the non-condensed labyrinth regions. A large number of dislocations crossing between the \{100\} walls are apparent. Burgers vector analysis of this area revealed that many dislocations within the walls parallel to (010) had \( \mathbf{b} = \mathbf{h}[110] \) and \( \mathbf{h}[011] \) or \( \mathbf{h}[01\bar{1}] \).
Examples of dislocations crossing between the walls which have \( \mathbf{b} = \mathbf{M}[011] \) or \( \mathbf{M}[011] \) are marked \( \mathbf{k} \) on Fig. 5.74. Dislocations with \( \mathbf{b} = \mathbf{M}[101] \) or \( \mathbf{M}[101] \) are also present in the area indicated by the disappearance of some dislocations for \( g_{220} \). In the channels many of the small dislocation loops are out of contrast for \( g_{220} \) indicating that these have \( \mathbf{b} = \mathbf{M}[1\bar{1}0] \). A very small change in contrast across the walls is also indicated for this operating reflection showing that there is misorientation across the walls. Unlike the most usual observations of structures based on \{100\} walls in Cu fatigued at room temperature, frequent observations of large areas dominated by one set of \{100\} walls were made in Al fatigued at this strain amplitude at 77 K. An example of the extent of this feature is shown in Fig. 5.75 a. Burgers vector analysis of this structure (see Figs. 5.75 b to 5.75 h) revealed that dislocations of all six Burgers vector types are present in the area, although mainly \( \mathbf{b} = \mathbf{M}[1\bar{1}0] \) and \( \mathbf{M}[1\bar{1}0] \). Examples of dislocations which have \( \mathbf{b} = \mathbf{M}[1\bar{1}0], \mathbf{M}[1\bar{1}0], \mathbf{M}[101], \mathbf{M}[10\bar{1}], \mathbf{M}[011] \) and \( \mathbf{M}[011] \) are marked \( \mathbf{a}, \mathbf{b}, \mathbf{c}, \mathbf{d}, \mathbf{e} \) and \( \mathbf{f} \) respectively on Figs. 5.75 b, d, e and g. Many of the small dislocation loops within the channels are out of contrast for \( g_{220} \) and \( g_{220} \) indicating that these have \( \mathbf{b} = \mathbf{M}[1\bar{1}0] \) and \( \mathbf{M}[1\bar{1}0] \), which suggests that they are associated with dislocations which form the walls. A weak-beam dark field image in Fig. 5.75 i shows the presence of a large number of small dislocation loops in the low dislocation density channel. The observations of Jin and Winter on the room temperature fatigue of Cu crystals with a \{001\} orientation reveal a similar structure (see Fig. 1 (b), Jin and Winter, 1984 b), however in Al at 77 K the dominance of a single set of \{100\}
walls is much more extensive than that reported for Cu. In Al, the spacing of the walls in some labyrinth structure which is not fully condensed (e.g. Fig. 5.72 a) and in the single set of {100} walls is approximately 1.6 μm (± 0.1 μm), i.e. ~ 2 and ~ 3 times the typical respective separations for similar structures observed in Cu after room temperature fatigue (Winter, Pedersen and Rasmussen, 1981; Jin and Winter, 1984 b). The formation of cell-type dislocation structures within {100} labyrinth-type walls observed in one grain is illustrated in Fig. 5.76 and at higher magnification in Fig. 5.77. A high density of dislocation loops was apparent in the channels and within the cells small dense clusters of dislocation loops approximately 0.5 μm in size are apparent. Changes in contrast between the cells marked A and B in Fig. 5.77 for ₁₀₁ (not illustrated) indicate misorientation across the cell walls. The presence of dislocations with ₇ = ₁₀₁, ₀₁₁ and ₁₁₀ is indicated by dislocations which are out of contrast for ₁₀₁, ₁₀₁ and ₂₂₀. Examples of dislocations with ₇ = ₁₁₀ and ₀₁₁ are marked b and e respectively on Fig. 5.77. Many of the small dislocation loops are out of contrast for ₁₀₁ and ₁₀₁ indicating that these have ₇ = ₁₀₁ and ₀₁₁.

An unexpected type of dislocation configuration is shown in Figs. 5.78 to 5.80. This specimen was also fatigued at εₜ = ± 1.4 x 10⁻³ at 77 K for 5 x 10³ cycles, and in this case the specimen contained the surface. This structure can be described as a square "lattice" of dislocation dense regions with side parallel to <110> directions and with a lattice parameter of ~ 1.9 μm. It should be noted that on
tilting from $B = [001]$ to $B = [011]$ (not illustrated) the dislocation cluster image projections were found to satisfy a face-centred cubic structure, since a hexagonal "lattice" of dislocation dense regions with side parallel to $<100>$ and $<111>$ directions was observed. Such a well-defined lattice-type structure was observed on one occasion only. The structure is shown at higher magnification and under different operating reflections in Figs. 5.79a and b, whilst a low magnification TEM micrograph (Fig. 5.80) shows how extensive this structure can be. Fig. 5.81 is an optical micrograph of the surface of the specimen, where $B$ denotes the lattice-type dislocation structure region. The area marked $B$ is shown at higher magnification in Figs. 5.82 and 5.83, where the latter corresponds to the structure in Fig. 5.80. A SEM micrograph, Fig. 5.84, of part of the surface and the stereo-pair, Fig. 5.85, show a complementary arrangement of circular features which are below the surrounding surface. Examination of micrographs taken under different operating reflections ($g_{220}$, $g_{111}$, $g_{131}$, and $g_{131}$) in addition to those illustrated in Figs. 5.78 - 5.79b ($g_{200}$ and $g_{020}$) also reveal circular dislocation dense regions which have dark contrast. Thus, the dark contrast in Fig. 5.78 corresponds to thinner regions of the foil and is predominantly strain contrast, since the extinction distance for some of the reflections examined is large. These dark contrast areas correspond to regions with a high density of dislocations which have Burgers vectors parallel to $[101]$, $[10\bar{1}]$, $[110]$ and $[1\bar{1}0]$ in approximately equal numbers. Between the circular regions of dark contrast are areas relatively free of dislocations. The tension-compression axis for this specimen
was found to be parallel to [010].

These lattice type structures would appear to form from regions with a high density of dislocation tangles and several grains were found in which such structures were observed. A low magnification example of a region in which the formation of this lattice structure is apparent is Fig. 5.86. The area in this region where labyrinth type structure is evident is shown at higher magnification in Fig. 5.87. Dislocation clusters within the channels between the {100} walls are apparent. Full Burgers vector analysis was not possible for this area, however the disappearance of a number of dislocations at k (Fig. 5.87) for $g_{200}$ indicates the presence of dislocations which have $b = \alpha[011]$ or $\alpha[01\bar{1}]$. Dislocations marked $b$ have $b = \alpha[110]$, and those marked $a$ have $b = \alpha[110]$ (see Fig. 5.87). This area is adjacent to an area exhibiting dislocation tangles (Fig. 5.88). It is within such tangled dislocations that the clusters appear to form as shown in Figs. 5.89 and 5.90. In Fig. 5.89 irregular walls which are approximately parallel to [110] (corresponding to $(110)$ planes) are also evident. Misorientation between the region exhibiting labyrinth type structure and the region with dislocation tangles is indicated by changes in contrast apparent for $g_{220}$. A stereo-pair of clusters forming from dislocation tangles is shown in Fig. 5.91.

A further example of the formation of lattice type clusters in a different specimen fatigued at $\varepsilon_t = \pm 1.4 \times 10^{-3}$ at 77 K for $5 \times 10^3$ cycles is shown in Fig. 5.92 and at higher magnification in Fig. 5.93. Again, the region adjoining this area is found to exhibit
<100> walls as shown in Fig. 5.94. The surface structure which corresponds to the area in Fig. 5.92 in which lattice structure is forming is shown in Fig. 5.95, and at higher magnification in Figs. 5.96 to 5.98. This area is found to be adjacent to a grain in which matrix structure is found.

In a different specimen, on one occasion dark contrast features were found however in this case a fairly uniform arrangement of tangled dislocations was evident (Figs. 5.99 and 5.100), suggesting that this contrast may be due to variations in thickness. Full Burgers vector analysis of the area in Fig. 5.100 was not possible, however dislocations marked e in this area were found to have \( b = \mathbf{b}[011] \). Dislocations with \( b = \mathbf{b}[110] \) or \( \mathbf{b}[10\overline{1}] \) or \( \mathbf{b}[101] \) are also found to be present in this region. The surface structure corresponding to this dislocation structure is shown in the optical micrograph Fig. 5.101. It can be seen that the surface appearance is similar to that in Fig. 5.83.

Additional SEM of material fatigued at 77 K for \( N = 5 \times 10^3 \) at \( \epsilon_t = \pm 1.4 \times 10^{-3} \) revealed the occurrence of both less well defined cross-hatched structure and rumpled bands (Figs. 5.102 and 5.103). From comparisons of regions examined by TEM with associated optical or SEM micrographs, such features were not found to be related to any specific dislocation configuration.

5.5: Brief Summary of Experimental Results
In general, approximately five times the number of specimens were examined after fatigue at room temperature in comparison with those fatigued at low temperature, for any given number of cycles and strain amplitude. Usually about ten grains were examined in each specimen, and features considered to be representative of the types of structures observed were then examined in detail.

The very high densities of small dislocation loops seen in otherwise dislocation free regions after fatigue at 77 K are not a feature of room temperature fatigue, although the density of loops is appreciable at the latter temperature. In addition no clear examples of a matrix structure, PSB ladder structure or labyrinth structure are found after room temperature fatigue at any strain amplitude, however examples which approximate such structures are found after fatigue at low temperature. At the lowest strain amplitudes irregular dense patches of dislocations were seen after room temperature fatigue however irregular dislocation walls were commonly observed at all amplitudes. These irregular walls were frequently found to be approximately parallel to <110> and <100> directions in foils with <100> normals and are thus approximately parallel to {110} and {100} planes. Low temperature fatigue resulted in dislocation walls approximately parallel to {331}, {311}, {310}, {210}, {111} and {122} planes being observed, in addition to walls parallel to {100} and {110} planes. The wall separations in the range 0.7 μm to 1.0 μm following fatigue at room temperature contrast with the observed separations at 77 K which are in the range 0.7 μm to ~3 μm. After room temperature fatigue at the
highest amplitudes, a more clearly defined cell structure is evident, with narrow walls (which are oriented preferentially along \textlangle 100 \rangle \textrangle and \textlangle 110 \rangle \textrangle approximately (corresponding to \{100\} and \{110\} planes) enclosing large dislocation free regions.

Finally, a new type of condensed structure which consists of a well defined lattice of approximately circular dislocation clusters, has been observed after low temperature fatigue at the highest amplitude investigated.

5.6: Discussion

5.6.1: Determination of Planes

Regarding the determination of the indices of planes of dislocation walls, the following method was used. It is assumed that the dislocation wall is perpendicular to the plane of the micrograph, thus the plane indices are determined by taking the cross product between the beam direction and the direction to which the walls appear to be parallel. For dislocation walls which are very narrow this method is appropriate, however stereo-pair micrographs were not taken for every case thus it is possible that the planes of the dislocation walls may not always be exactly perpendicular to the plane of the micrograph (and therefore not exactly parallel to the beam direction). Consequently, in determining indices of planes it
has been necessary to consider all possible planes which are sufficiently close to the plane determined by the cross product method (i.e. such that any difference in wall orientation might not be distinguished) which could correspond to the observed dislocation wall direction. Thus, the most likely plane corresponding to the observed wall direction is subsequently selected from the possible planes determined.

5.6.2: Room Temperature Fatigue of Al

A summary of the dislocation structures which have been previously reported after room temperature fatigue of Al was included in section 2.4.5 (ii). The following section includes a comparison of results obtained during the present work on room temperature fatigue of Al with these earlier observations.

From the micrographs presented here it is apparent that the substructure developed at any given cycle level, at low amplitudes, varies from grain to grain, as found by Grosskreutz and Waldow (1963), and is thus dependent on the orientation of the grain with respect to the tensile axis (Mitchell and Teer, 1969 a).

Segall has reported that when the fatigue stress was sufficiently low no subgrain formation was observed but that dislocations exist in patches, which is in agreement with the trend of the data presented by Grosskreutz and Waldow (1963) for polycrystalline Al.
However, it has been observed by McGrath (1963) that a subgrain structure exists throughout Al fatigued at a strain amplitude causing failure in $2 \times 10^6$ cycles. In addition, observations of groupings of dislocations along crystallographic traces of low index planes in Al fatigued at room temperature have been made by Grosskreutz (1963) and Mitchell and Teer (1969 a). It should be noted that results presented here for low strain amplitude fatigue of Al reveal that some degree of organization of dislocations parallel to crystallographic traces of low index planes is always apparent, with many examples of (100) and (110) oriented loose wall structures found.

The appearance of dislocation structures in areas of Fig. 5.7 are similar to the parallel boundaries of dislocations observed by Mitchell and Teer (1969 a) within a crystal of (013) surface orientation and [100] stress axis, fatigued at a stress amplitude which caused specimen failure after 6-8 $\times 10^5$ cycles. The type of dislocation structure seen in Fig. 5.8 and 5.10 appears similar to the intense bands of dislocations observed by Mitchell and Teer (1969 a) in crystals of (001) surface orientation and [100] stress axis, and to the substructure observed in regions of Al crystals which are free from surface deformation markings which were fatigued at a stress causing failure at $10^7$ cycles (Mitchell and Teer, 1969 b). However, in Fig. 5.8 and 5.10 dislocations can be seen to extend from the intense bands of dislocations and in some cases cross the dislocation free channels, such features are not apparent in the micrographs presented by Mitchell and Teer (1969 a, b).
Segall and Partridge (1959) have observed structures similar to those in Fig. 5.12 after fatigue at low stress amplitudes. In addition, Segall and Partridge found a high dislocation density and an absence of well-defined wall configurations at these amplitudes, and structures similar to those in Fig. 5.15 were found with dislocations threading the channels.

Grosskreutz (1963) has examined the substructure developed in polycrystalline Al after various numbers of cycles at a strain amplitude of \( \pm 5 \times 10^{-4} \). The loose tangles of dislocations shown in Fig. 5.14 are similar to the structure observed by Grosskreutz after \( 2 \times 10^5 \) cycles at \( \pm 5 \times 10^{-4} \) strain, although in Fig. 5.14 a greater number of dislocation loops are apparent and the subgrain boundaries of dislocations appear to be less well defined.

At higher strain amplitudes cell and wall structures have been observed in the present work. These results are consistent with those of Krejčí and Lukáš (1971) on Al fatigued at high stress, and with those of Grosskreutz (1963). The structures in Fig. 5.18 to 5.23 are, for example, similar to the elongated and equiaxed subgrains observed by Grosskreutz (1963) after \( 5 \times 10^3 \) and \( 5 \times 10^2 \) cycles at \( \pm 2 \times 10^{-3} \) strain. Krejčí and Lukáš do not report the planes or directions parallel to which the walls in such structures lie, however Grosskreutz observed that dislocation walls and walls in subgrain structures were parallel to the crystallographic traces of low index planes, which is in agreement with the results.
presented here.

At both high and low strain amplitudes quite a large number of dislocation loops are apparent (more than observed than after room temperature fatigue of Cu), which is in agreement with the results obtained for Al by Wilson and Forsyth (1958–59, 1959), Segall and Partridge (1959), Grosskreutz (1963), Grosskreutz and Waldow (1963), Mitchell and Teer (1970) and Krejčí and Lukáš (1971).

Mitchell and Teer (1969 a, 1970) suggest that PSBs correspond to a cellular structure or to tangled dislocation boundaries, and that intense dislocation clusters correspond to surface regions which do not contain PSBs. Similar observations have been made by Krejčí and Lukáš (1971) who found that after low amplitude fatigue the cell structure beneath PSBs did not form narrow bands in Al as found in the case of medium stacking fault energy metals (e.g. Cu). In the present work, the structure shown in Fig. 5.7 corresponds to a surface in which PSBs were evident. Thus the present work reveals a wall structure rather than the cell or boundary structure previously reported by Mitchell and Teer and Krejčí and Lukáš.

It should be noted that no clear examples of a matrix structure, PSB ladder structure or labyrinth structure, as found in Cu fatigued at room temperature, were observed in Al after room temperature fatigue, despite the large number of specimens and areas examined. The surface PSBs in Al are also found to be very different in appearance, being much more irregular and wavy than for Cu. In
addition, surface features consisting of cross-hatched markings have been found after room temperature fatigue of Al whereas such features have not been observed in Cu.

5.6.3: Low Temperature Fatigue of Al with Ageing at Room Temperature

In Al examined after various ageing periods at room temperature following fatigue at 77 K, either matrix type structures or walls of dislocations are observed. Walls of dislocations approximately parallel to \langle110\rangle, \langle100\rangle and \langle331\rangle (corresponding to \{111\}, \{010\} and \{331\} planes) are observed. In addition, some dislocation walls are found to be approximately parallel to \langle012\rangle and \langle113\rangle, directions and these are found to correspond to \{113\} planes. The formation of such \{113\} walls during the fatigue of f.c.c. metals has been predicted by a recent model for dipolar walls containing dislocations of two Burgers vectors (Dickson, Boutin and L'Espérance; 1986). In connection, it should be noted that \{012\} wall orientations, which have also been predicted by the model by Dickson et al, have been identified in Cu fatigued at room temperature at a plastic strain amplitude of $5 \times 10^{-3}$ by Ackermann et al (1984). The results on aged material thus reveal structures which are more similar to structures observed after low temperature fatigue without specimen ageing, than to structures formed after fatigue at room temperature.
5.6.4: Low Temperature Fatigue of Al with Ageing at Room Temperature Minimized

The results presented on low temperature fatigue, with specimens maintained at as low a temperature as possible at all stages, would appear to provide good evidence for the dislocation structures produced during low temperature fatigue. This view is supported by the point that some important dislocation configurations which are closely similar to those observed in Cu fatigued at room temperature are found. There is expected to be some dislocation loss and rearrangement, but there is no a priori reason to believe that this will be greater than in other studies of fatigue using TEM, except for the possibility of enhanced dislocation movement due to climb caused by the condensation of point defects.

The observations of Ceresara (1969) on resistivity changes in Al fatigued in torsion at 77 K show that a large decrease in defect density, corresponding to a reduction of about 50% in the fatigue induced resistivity, occurs on annealing up to 300 K. It is likely that a major contribution to this resistivity change arises from point defect loss. An examination of the channels between the regions of high dislocation density in the present work (e.g. in Figs. 5.52 and 5.62) reveals a very high density of small loops, a higher density than observed in Cu or Al fatigued at room temperature. One possibility is that these loops may be from vacancies formed during fatigue at 77 K which become mobile at a
higher temperature. The condensation of point defects may partly act
to stabilize the large scale dislocation structures by pinning.
Another possible explanation for the large numbers of loops observed
in the channels is related to the ease of dislocation intersection
in Al, thus the greater number of loops may be attributable to more
widespread dislocation cutting during fatigue. Alternatively,
dislocation loops already present, which are formed by fatigue
deformation, may grow by vacancy condensation and become visible.

The dislocation structures which have been found after low
temperature fatigue are not observed when Al is fatigued at room
temperature. It must therefore be concluded that softening
mechanisms involving climb, cross-slip and vacancy diffusion must be
suppressed when the temperature is lowered, resulting in stability
of the classical wall structure. The point that different wall
structures are observed after room temperature fatigue suggests that
matrix and ladder-type structures are not lowest energy structures
but are metastable, i.e. some wall structures are more stable but
cannot be achieved at low temperatures.

It is evident from the results presented here that the dislocation
structures apparent after low temperature fatigue of pure Al are
very similar to those in other f.c.c. metals of medium and high
stacking fault energy. However, it should be noted that the results
described here for Al reveal that a larger number of Burgers vectors
are active after low temperature fatigue than would be expected in
Cu after fatigue at room temperature. Although most grains in Al had
a \langle 100 \rangle \) foil normal (which would indicate orientations for multiple rather than single slip), the stress axis for many grains would not in fact have been close to \([100]\). For such orientations in the case of Cu, many grains would show predominantly single slip. This suggests that secondary dislocations are able to move more easily in Al due to the higher stacking fault energy of the material. This enables dislocations to cross-slip and avoid obstacles more easily, and the stress required to force secondary dislocations through a forest of primary dislocations is lower.

In many Al grains after low temperature fatigue at strain amplitudes less than \(\varepsilon_t = \pm 1.4 \times 10^{-3}\), and in a few grains after fatigue at this amplitude, matrix structures similar to those found in Cu and Ni after room temperature fatigue are found. However the density of dislocation loops is higher in Al. Following low temperature fatigue at \(\varepsilon_t = \pm 1.4 \times 10^{-3}\) usually the matrix type dislocation structures observed appeared narrower with a greater channel proportion than generally observed after room temperature fatigue of Cu and Ni.

It should also be noted that in Cu and Ni the ladder wall spacing is 1.4 \(\mu m\) and 1.3 \(\mu m\) respectively after room temperature fatigue (Winter et al, 1981; Gai, 1974; Mecke et al, 1982) compared with \(~ 2.8 \mu m\) in Al after low temperature fatigue. Similarly, considering the wall spacing in fully formed labyrinth structures after low temperature fatigue of Al the spacing is \(~ 2 \mu m\), compared typically with 0.75 \(\mu m\) in Cu (Winter et al, 1981) and 0.78 \(\mu m\) in Ni (Mecke et al, 1982) after room temperature fatigue. Hence the respective
spacings of similar ladder type and labyrinth type wall structures in Al at 77 K are approximately 2 and 2.6 times the values typically observed for Cu and Ni at 293 K. In the work on Al, a testing temperature of 77 K is 0.08 $T_m$ whereas in the cases of Cu and Ni fatigued at room temperature the testing temperatures are 0.22 $T_m$ and 0.17 $T_m$ respectively, where $T_m$ is the appropriate melting point temperature. Measurements made by Basinski, Korbel and Basinski (1980) on the dislocation microstructure observed in Cu after fatigue at different temperatures have shown that the spacing in the wall structures decreases with decreasing temperature. For Cu it was found that the wall spacings in the ladder structures were 0.7 $\mu$m and 0.45 $\mu$m after fatigue at 77 K and 4.2 K respectively.

The results here suggest that in Al the wall spacing in the ladder structure is approximately four times as large as in Cu at equivalent temperatures (77 K). If the scale of the structure depends on the length of screw dislocations which can be moved through the channels, at the applied (saturation) fatigue stress, then the scale can perhaps be related to stacking fault energy. For higher stacking fault energy materials the passing distance for screw dislocations is larger (Mughrabi, 1979). It has been suggested by Watt and Ham (1966) and Hancock and Grosskreutz (1969) that the wall spacing should be related to the flow stress. Strong support for these suggestions is indicated by the work of Kwadjo and Brown (1978) who have compared results obtained on magnesium with Cu and Ni. Kwadjo and Brown (1978) assume that the flow stress is the saturation stress, and for saturation stresses of 2 MPa and 7.4 MPa
(corresponding to testing temperatures of 0.33 $T_m$ and 0.08 $T_m$ respectively) the wall spacing in magnesium is found to decrease from 8 $\mu$m to 2 $\mu$m. In Cu and Ni tested at room temperature, the saturation stresses are 30 MPa and 55 MPa respectively and the wall spacings are found to be 1.4 $\mu$m and 1.3 $\mu$m respectively (as noted above). Thus Kwadjo and Brown conclude that the wall spacing and saturation stress are strongly temperature dependent and this can only reflect the temperature dependence of the cross-slip of screw dislocations. Another factor contributing to the variation of wall spacing and saturation stress with testing temperature is the intersection of dislocations on different systems and with "debris" such as small loops. It should be noted that ladder type structures were infrequently observed in Al and were found to be more irregular than those found in Cu, Ni or Mg after room temperature fatigue. A tendency for ladder type structures in Al to readily form cell structures, indicating that ladder type structures are not very stable, is also apparent after the low temperature fatigue of Al. These points may be related to the ease of movement of secondary dislocations in Al whereas the formation of classical wall structure requires predominantly one Burgers vector. In some cases, walls in structures which approximate to ladder structures in Al have been found to be approximately parallel to {113} planes, i.e. walls of the type predicted by Dickson et al (1986). Such walls have also been observed in Al subjected to ageing after fatigue, as noted previously.

After low temperature fatigue of Al, in similar types of $\langle 100 \rangle$
labyrinth walls which are not fully formed the channel width decreases by a factor of approximately three with increasing strain amplitude in the range \( \varepsilon_t = \pm 8 \times 10^{-4} \) to \( \pm 1.4 \times 10^{-3} \). In regions exhibiting such features, the structure often has areas which are of hollow box-like appearance, with pairs of parallel dislocation walls enclosing square dislocation free areas. Thus there are indications of a hard shell enclosing a soft inner core, which is consistent with the suggestion by Kuhlmann-Wilsdorf and Laird (1977) that an accumulation of point defects causes softening of relatively hard wall interiors. Comparing channel widths in the labyrinth structure, which is not fully formed, with that in the fully formed structure after fatigue at \( \varepsilon_t = \pm 1.4 \times 10^{-3} \), it is found that the width in the latter is about three times as large as in the former.

In areas exhibiting labyrinth walls in Al, significant deviations from \{100\} planes were often observed. In addition, more than two Burgers vectors are often found in the \{100\} walls. In areas exhibiting (010) type walls only, the walls are found to be non-uniformly narrow, and there is evidence of channels forming within holes in the walls. Non-uniformity of (001) walls in [001] Cu single crystals fatigued at room temperature is also apparent, the deviations from (001) tending to result in short dislocation wall sections perpendicular to these walls (Jin and Winter, 1984 b).

In view of the large number of Burgers vectors active in Al after low temperature fatigue, the labyrinth and cell structures may form more readily in Al and the simple well defined classical PSB
structure might not form. The testing technique itself cannot be a reason for this, since well-defined PSB wall structures have been found in [125] orientation Cu specimens fatigued at room temperature by Razzak (1987), after using the same testing technique as in the present work. It should be noted that Jin and Winter (1984 b) only observed many Burgers vectors in labyrinth structure in [001] Cu single crystals after room temperature fatigue, where the labyrinth walls were each found to consist of two different Burgers vectors. On the contrary, for Al, in many cases the specimen stress axis would not have been close to [001]. Furthermore, for Cu labyrinth structures were found to be an effective barrier against the formation of normal PSB structures, especially in the areas containing predominantly one set of dislocation walls, since only a small number of PSB ladders were observed, and these occurred only in regions containing both longitudinal and transverse walls.

The lattice type structure has not been reported in Cu or Ni at the present time. It is possible that it would be more unlikely to form in these metals if the observed difference in the spacing of the dislocation structures is taken into account, and if it is assumed that the dislocation clusters themselves would remain relatively constant in size. If the clusters begin to overlap the structure would be expected to be unstable.

The occurrence of the lattice type structure would appear to depend on the presence of significant numbers of dislocations with four distinct Burgers vectors. It seems likely that a tensile axis with
an orientation very close to [010] is required. The observed surface structure can be understood as the result of an extrusion process in which the regions surrounding the clusters are relatively soft. Thus a square lattice of approximately circular depressions is observed on the surface.

The approximately circular patches of dislocations in the lattice type structure are found to be a three dimensional array, since on tilting from B = [001] to B = [011] the cluster image projections are found to satisfy a face centred cubic structure. The regularity of projection of a two dimensional array would not be orientation dependent. These observations suggest that the cluster arrangement corresponds to spherical groupings of dislocations, and since the array is three dimensional not all clusters are in the surface. It should be noted that similar observations of a void array in molybdenum (resulting from particle bombardment) which forms a body cented cubic lattice have been reported by Evans (1971). The regular arrangement of symmetrical groupings on a lattice strongly suggests that a low energy dislocation configuration is formed in which a repulsive interaction between clusters plays a prominent role. This repulsion could arise in part from the local dilatation due to the localized high densities of dislocations such as suggested by Charsley and Kuhlmann-Wilsdorf (1981).

Finally, considering optical micrographs of surface structures after low temperature fatigue of Al, it is clear that at all strain amplitudes the irregular wavy surface PSBs which are apparent after
fatigue at room temperature have not been observed in the present study. However, Mughrabi (1985) has reported "bulgy" extrusions in Al single crystals fatigued at 77 K for $6 \times 10^3$ cycles at a shear strain amplitude, $\gamma_{pl}$, of $2 \times 10^{-3}$. It is important to note that the surface corresponding to the areas exhibiting labyrinth structure after fatigue at 77K appears featureless. This may be understood in view of the fact that the labyrinth structure is a much more "homogeneous" dislocation arrangement than a set of PSB ladders or cells in a matrix. Also, since slip occurs on more than one system in labyrinth prominent extrusions would not be expected. Areas exhibiting cross-hatched surface markings after fatigue at 77K do resemble structures found after room temperature fatigue of Al.
CHAPTER 6

FATIGUE OF INDENTED SINGLE CRYSTALS

6.1: Introduction

Single crystals of Al with several different orientations have been annealed, indented (with a separation between indentations of ~ 33 d) and then fatigued at a low amplitude. The PSB distributions which have developed near the indentations have been examined and are presented. For most crystal orientations both S and D type indentations (i.e. either indentation edges or diagonal respectively parallel to the specimen tensile axis) have been examined. The influence of the crystal orientation, the indentation size, and the effects of annealing and polishing treatments are described. The fatigue slip distributions near indentations for one particular orientation, C3, are examined in detail.

Notation used to identify indentations is in terms of the crystal orientation, the indentation number and type (S or D type), and whether the indentation is unannealed (U) or annealed (A). Thus, for example, indentation number 6 in crystal C7 which is of S type and is unannealed, is identified as C7.6SU. Regions near the indentations are defined by an r, e coordinate system as illustrated in Figs. 4.3 a, b. The direction of the specimens' tensile axes (corresponding to t₀ shown in the standard stereographic triangle in
Fig. 4.2 b) are denoted by t on micrographs presented in this chapter.

6.2: Experimental Results

6.2.1: Crystals of Orientation C7

Figs. 6.1 a - 6.2 c are optical micrographs which show the effects of fatigue near S type indentations in C7 after $N = 1.6 \times 10^3$ cycles using wheel $W_1$, and after $N = 6.5 \times 10^3$ using $W_2$ (the values of the strain amplitudes corresponding to various wheels are given in Table 4.3). No PSBs have nucleated after fatigue using $W_1$, and PSBs were first observed near all indentations after $6.5 \times 10^3$ cycles using $W_2$. Furthermore, PSBs have not nucleated within the indentation pits nor directly at their edges after $N = 6.5 \times 10^3$ using $W_2$. In general near all indentations the closest group of PSBs occurs in the angular region $\theta \approx 160^\circ - 240^\circ$ at $r \approx d$. A second group of PSBs occurs in the angular region $\sim 10^\circ - 40^\circ$ at $r \sim 2.5 \, d$ for indentations C7.3SU and C7.6SU. The angular regions $260^\circ - 10^\circ$ and $40^\circ - 150^\circ$ near C7.3SU are essentially free of PSBs except for an isolated PSB at $\theta = 100^\circ$, $r \approx 1.5 \, d$ (see Fig. 6.1 b). Similarly near C7.6SU, the angular regions $40^\circ - 150^\circ$ and $240^\circ - 10^\circ$ are free of PSBs except for the two groups of PSBs at $\theta \approx 250^\circ$ and $\theta \approx 290^\circ$ at $r \sim 1.2 \, d$ (see Fig. 6.2 b).

SEM micrographs showing the details of PSBs near C7.3SU and C7.6SU are shown in Figs. 6.3 a - 6.4 b.
The slip bands apparent near indentations in C7 correspond to (111) slip planes (i.e. the cross slip plane).

6.2.2: Crystals of Orientation C10

Figures 6.5 a to 6.8 show the effects of fatigue after $1.05 \times 10^4$ cycles and $1.25 \times 10^4$ cycles, using W2, near unannealed and annealed (annealing temperature 425°C) indentations in C10.

Fatigue slip bands were first observed after $1.05 \times 10^4$ cycles using W2 near the unannealed indentation C10.2SU at $\theta = 350° - 90°$ and $150° - 210°$. Some of the slip bands initially apparent after $N = 1.05 \times 10^4$ have developed to form more pronounced PSBs with continued cycling to $N = 1.25 \times 10^4$. Furthermore, at this latter cycle level PSBs have also formed in areas which appear free from slip markings at the lower cycle level, for example the PSBs developed at $\theta \approx 220°$, $r \approx 4$ d near C10.2SU. It is clear that after $N = 1.25 \times 10^4$, no PSBs are formed in the angular regions 120°-135° and 280°-320° near the unannealed indentation C10.2SU. Outside these regions PSBs form at distances ranging from 2.4 d - 11.1 d.

Near the annealed indentation C10.3SA PSBs are not apparent after $N = 1.05 \times 10^4$, and are first observed after $1.25 \times 10^4$ cycles using W2. In general, no PSBs form closer than 1.1 d except at $\theta = 130°$, where a PSB occurs at $r \approx 0.8$ d. PSBs appear to be present in all
other radial regions around the indentation.

The slip bands apparent near indentations in C10 correspond to (1\bar{1}1) slip planes (marked D on Fig. 6.5 a) and (\bar{1}11) slip planes (marked C on Fig. 6.5 a) i.e. cross and conjugate planes respectively.

Again PSBs have not nucleated within the pit or directly at the pit edges in any case, as clearly revealed by the SEM micrographs Figs. 6.7 and 6.8.

6.2.3 (i): Crystals of Orientation C11

Figs. 6.9 a - 6.10 b are optical micrographs of the PSB distributions developed near annealed and unannealed indentations in C11. For annealed indentations in this crystal a higher annealing temperature (550°C) was used than for those in C10. Both S and D type indentation orientations were investigated. PSBs were first observed after N = 2 x 10^3 cycles using wheel W_1. It was not possible to continue fatigue of this crystal after N = 2 x 10^3 due to deformation of the narrow end of the crystal on continued cycling.

Near both the S and D type unannealed indentations in C11 PSBs are closest in the angular region 150° - 210° at r ∼ 0.7 d. These PSBs extend out to r > 6.5 d. The angular regions ∼ 230° - 110° appear to be free of PSBs near C11.7DU, whereas near C11.5SU PSBs are apparent
in all angular regions and some relatively short and deep PSBs are seen, for example, at: \( \theta = 0^\circ - 20^\circ \) at \( r = 2.4 \, d \) and \( 3.1 \, d \); \( \theta = 160^\circ - 200^\circ \), \( r = 1.2 \, d \); \( \theta = 330^\circ - 340^\circ \), \( r = 4.4 \, d \) (see low magnification micrographs Figs. 6.10 a and b). In the angular region \( 330^\circ - 20^\circ \) the closest PSBs near C11.5SU are at \( \theta = 0^\circ, r \approx 2 \, d \).

Near the annealed S type indentation, C11.4SA, PSBs are also found in all angular regions around the indentation, and the most prominent PSBs in this case are longer and straighter than those seen near the unannealed S type indentation. The closest PSBs are at \( \theta = 260^\circ \) where a PSB appears to approach the indentation edge and is apparent at \( r = d/4 \). Near the annealed D type indentation, C11.6DA, PSBs are apparent in the angular region \( \theta = 120^\circ - 290^\circ \) and are closest at \( \theta = 180^\circ \) at \( r \approx d \).

PSBs apparent near unannealed and annealed standard size indentations in C11 correspond to slip on (111) (cross slip) planes and are marked D on Fig. 6.9 a.

PSBs do not appear to have nucleated within the indentation pits for either S or D type unannealed, or for annealed D type, indentations. However it should be noted that PSBs appear to be present within the annealed S type indentation, C11.4SA.

6.2.3 (ii): Influence of Indentation Size in Crystal of Orientation C11
Examples of unannealed indentations with $d = 74 \, \mu m$ and $107 \, \mu m$ i.e. approximately 1.3 and 1.8 times the usual indentation diagonal length were used to investigate any effects which might arise due to indentation size.

The effects of indentation size are shown by the optical micrographs Figs. 6.11 and 6.12 and SEM micrographs 6.13 a, b. Slip produced by indenting is evident at $\theta = 80^\circ - 120^\circ$ from the indentation edge out to $r \approx 2/3 \, d$, and at $\theta = 240^\circ - 280^\circ$ and $350^\circ - 20^\circ$ out to $r = d/2$. The slip markings due to indenting correspond to slip on (111) planes (i.e. primary planes, marked B on Fig. 6.11) and to slip on (\bar{1}11), (\bar{1}11) and (1\bar{1}1) planes (i.e critical, conjugate and cross planes, denoted A, C, and D respectively on Fig. 6.12). Fine slip bands formed during fatigue can be seen in all angular regions near C11.10SU, and the occurrence of slip on two slip planes is evident. Slip bands corresponding to slip on (\bar{1}11) planes are denoted C and bands corresponding to (1\bar{1}1) planes are denoted D. The closest fatigue slip bands near C11.10SU are at $\theta = 180^\circ$ at $r = 1.5 \, d$.

Slip due to indenting is apparent within the unannealed S type indentation, C11.1SU (Fig. 6.13 a), but again after fatigue PSBs have not nucleated within the indentation pits for either C11.10SU or C11.1SU as shown by the SEM micrographs (Figs. 6.13 a and b).

6.2.4: Crystals of Orientation C6
Figs. 6.14 a and b are optical micrographs of slip produced by indenting the crystal of orientation C6. Details of these slip bands are shown on the SEM micrographs Figs. 6.15 a and b. The slip bands correspond to slip on the (111) plane, denoted D on Fig. 6.14 a. It is evident that the sides of the indentations are bowed inwards, forming the pincushion shape characteristic of indentations made in annealed materials.

Figs. 6.16 a - 6.19 c are optical micrographs which show the effects of fatigue near indentations in C6 after $N = 2 \times 10^3$, $4 \times 10^3$ and $8 \times 10^3$ cycles using wheel $W_1$. All indentations examined were of S type orientation. The PSB distributions near two unannealed indentations, one of which was polished to remove approximately 6 $\mu$m from the surface after indenting, and near two annealed (annealing temperature of 550°C) polished-back indentations are presented.

Considering the unannealed indentation C6.5SU, slip bands apparent after $N = 2 \times 10^3$, $W_1$, develop with increasing cycling, the first PSBs being observed after $N = 4 \times 10^3$ cycles using $W_1$ in the angular regions $\sim 315'$ - $110'$ and $170'$ - $285'$. After $N = 8 \times 10^3$ there are PSBs in most angular regions around the indentation, with the closest PSBs in the angular region $290'$ - $350'$ at $r = 0.7$ d. PSBs extend out from regions close to the indentation to distances of $> 6$ d. Regions which seem to be relatively free from surface slip markings near C6.5SU are $\theta \approx 305'$ - $315'$ and $155'$ - $165'$. Near C6.11SU, which was not polished-back after indenting, only very fine
slip is apparent in the region around the indentation at all stages of fatigue cycling.

Near annealed indentations, C6.4SA and C6.6SA, in C6, again it is clear that PSBs which have nucleated in most regions after \( N = 4 \times 10^3 \), \( W_1 \), become more pronounced with increased cycling. After \( N = 8 \times 10^3 \), \( W_1 \), PSBs are seen in all angular regions around the indentations with the closest PSBs at 0.7 d in the angular region 190° - 30°. These PSBs extend radially outwards to > 6 d.

The PSBs developed near indentations in C6 following fatigue at low amplitude correspond to slip on the (111) (cross slip) plane, denoted D on Fig. 6.16 a. Slip due to indenting on all types of \{111\} plane, with an approximately uniform slip distribution, is found near C6.11SU.

SEM micrographs of PSBs developed after \( N = 2 \times 10^3 \) and \( 6 \times 10^3 \), \( W_1 \), near indentations in C6 clearly show that no PSBs are formed within either unannealed or annealed indentation pits for this crystal orientation (see Figs. 6.20 a - 6.23 b).

6.2.5 (i): Crystals of Orientation C3

The slip distribution produced by making S and D type indentations in a crystal of orientation C3 is shown in Figs. 6.24 a to d. Slip corresponding to (111) and (111) planes (cross and primary slip
planes respectively) can be seen, and is denoted D and B respectively on these figures. Again the indentations have the characteristic pincushion shape.

An example of PSBs developed near the narrow end of the crystal is shown in Fig. 6.25 after $2 \times 10^4$ cycles, using the lowest amplitude wheel, $W_0$. These PSBs correspond to slip on the (111) plane. PSBs on (111) and (111) slip planes (denoted D and B respectively) near edge $t_1$ are found after $7 \times 10^3$ cycles, $W_1$, (see Fig. 6.26). After $8 \times 10^4$ cycles using $W_1$, a typical example of PSBs developed at $l = 12.7$ mm, $\phi = 0.5'$ (which correspond to slip on the (111) plane), is shown in Fig. 6.27. PSBs on two slip planes, (111) and (111) (denoted D and B), are apparent after fatigue under these conditions at $l = 31$ mm, $\phi = 1.2'$ (see Fig. 6.28).

A low magnification SEM micrograph of PSBs which correspond to slip on the (111) plane, developed near edge $t_1$ at the wide end of C3 is shown in Fig. 6.29 a, and the "wavy" appearance of these PSBs is evident from Fig. 6.29 b, which is at higher magnification.

The development of PSBs with increasing cycles up to $8 \times 10^4$ using wheel $W_1$ near unannealed and annealed indentations in C3 is shown at low magnification in Figs. 6.30 a - 6.37 c.

Considering the S type unannealed indentations, the first PSBs nucleated after $2.2 \times 10^4$ cycles using $W_1$ in the angular region 250' - 20' near C3.2SU. After $8 \times 10^4$ cycles at this strain amplitude,
there are PSBs in all angular regions around the indentations C3.2SU and C3.6SU, and these extend outwards to > 6 d. The closest PSBs have formed in the angular region \( \theta = 310' - 340' \) at \( \sim 0.9 \) d.

Intense slip bands correspond to slip on (111) slip planes (marked B on Figs. 6.30 a and 6.31 a). Figs. 6.30 a – c show that with increasing numbers of fatigue cycles, short fragmented bands initially apparent join up and then deepen. Some slip is also apparent on (111) slip planes (marked D on Figs. 6.30 a and 6.31 a).

Near annealed S type indentations similar distributions of PSBs have developed, however one group of PSBs in the angular region 80' – 190' has developed earlier in comparison with those near unannealed S type indentations, being first apparent after \( 2.2 \times 10^4 \) cycles using \( W_1 \). After \( N = 8 \times 10^4 \) cycles, the closest PSBs are at \( \theta = 10' - 40' \) near C3.1SA where one PSB can be seen at 0.5 d. Slip on two systems, again corresponding to (111) and (1\bar{1}1) slip planes, is apparent near C3.1SA.

Examples of unannealed D type indentations are C3.4DU and C3.8DU. The first PSBs are observed after \( 2.2 \times 10^4 \) cycles using \( W_1 \) in the angular region 235' – 25' near C3.4DU. After \( N = 8 \times 10^4 \) at this strain amplitude, PSBs are again apparent in all angular regions near C3.4DU, although PSBs are more intense in the angular regions \( \theta = 240' - 30' \) and 50' – 230'. The closest PSBs are at \( r = 0.7 \) d in the angular region 290' – 350'. The PSBs extend outwards to > 6 d. However, near C3.8DU there are regions exhibiting fine slip only in the angular regions 170' – 230' and 20' – 50', moreover the PSB
length does not extend further than 3 d.

Considering the annealed D type indentations, the first slip bands are observed in the angular region 220° - 10° near C3.3DA after 2.2 x 10^4 cycles using W1. After 8 x 10^4 cycles, the closest PSBs have formed at r = 0.5 d, θ = 20° - 30° for C3.3DA, and at r = 0.7 d, θ = 270° - 350° for C3.7DA. PSBs are seen in all angular regions around these indentations after fatigue at the latter cycle level, and extend outwards to > 6 d.

PSBs corresponding to slip on the (111) plane are also apparent near unannealed and annealed D type indentations. It should be noted that although PSBs corresponding to slip on the (111) plane can be seen near the unannealed D type indentations, these are not apparent near the annealed D type indentations.

Considering the PSBs developed near annealed and unannealed indentations of either S or D type orientations, there are similarities in the distributions developed, however PSBs are generally more pronounced near both types of annealed indentations in comparison with unannealed indentations of each type.

Comparing the PSB distributions developed near S and D type annealed indentations, some differences are apparent: it is clear that PSB development is more pronounced near S type indentations than near D type indentations at any given cycle level; in addition, PSBs form closer to such S type indentations over greater angular regions.
Moreover, considering non-primary slip, differences are apparent between S and D type indentations for both annealed and unannealed cases. Slip on (111) planes is more pronounced near unannealed D type indentations after \( N = 8 \times 10^4 \), than near S type unannealed indentations. For annealed indentations, slip on this plane is clearly only apparent near S type indentations and is not seen near D type indentations.

Slip which corresponds to slip on the (111) slip plane seems to be of the non-propagating type as described by Charsley and White (1987), and does not appear to increase in length with continued fatigue cycling. PSBs which correspond to slip on the (111) slip plane appear to be of the propagating type and PSB length increases with increasing numbers of fatigue cycles.

Higher magnification optical micrograph examples of PSB development with increasing numbers of fatigue cycles near selected indentations, C3.6SU, C3.5SA, C3.4DU and C3.3DA, are shown in Figs. 6.38 a - 6.41 d.

Details of the PSBs developed near indentations in C3 indicating their "wavy" appearance after \( N = 8 \times 10^4 \), \( W_1 \), are shown in the SEM micrographs Figs. 6.42 a - 6.42 h, and in one case, for C3.2SU, at higher magnification in Fig. 6.43.

It is clear from the higher magnification optical micrographs and
the SEM micrographs presented here that PSBs are not found within any of the indentation pits in any of the cases examined.

6.2.5 (ii): Fatigue Slip Near Etch Pits in Crystals of Orientation C3

PSBs were also found to develop near etch pits. An example of this is shown in Fig. 6.44 a, for an etch pit of ~ 20 μm diameter, at l = 12.7 mm, Φ = 12.8°. PSBs corresponding to slip on (111) and (1̅1̅1) planes are marked B and D respectively on this figure. Slip corresponding to slip on (1̅1̅1) planes is clearly less well defined than that corresponding to slip on (111) planes. The SEM micrograph 6.44 b reveals that at θ = 100° the extension of a PSB within the etch pit is apparent.

6.3: Brief Summary of Experimental Results

Characteristic fatigue slip band distributions which are dependent on crystal orientation were developed near both unannealed and annealed indentations in Al crystals fatigued in reverse-plane bending. The first PSBs nucleated between $2 \times 10^3$ to $4.2 \times 10^4$ cycles of fatigue (using either wheel W1 or W2) for all crystal orientations tested. In general, the effect of annealing indentations was either to cause PSBs to form closer to indentations and over greater radial regions, or to be more pronounced. However,
PSBs were very rarely (on one occasion only) observed to occur inside annealed indentation pits. PSBs of the long propagating type were usually observed near indentations in crystals of all orientations, however short non-propagating PSBs were observed only near indentations in crystals of orientation C3. The PSB distributions were often found to extend to large distances from the indentations. The influence of indentation size was examined for indentations with diagonal lengths of 1.3 and 1.8 times the standard indentation diagonal length. The effect of larger indentations was to result in indentation slip on all \{111\} slip planes, although during fatigue only fine slip markings were developed in the area around the indentations. Some differences in PSB development near S and D type annealed indentations have been observed, and also differences in the non-primary slip developed near both indentation types for unannealed and annealed cases have been found for one crystal orientation. Finally, near etch pits similar pronounced PSB distributions as those developed near indentations were found, and in addition PSBs were clearly seen to extend within etch pits.

6.4: Discussion

Following low amplitude fatigue, characteristic intense slip distributions are formed near indentations in Al, and the PSB distribution clearly varies depending on the crystal orientation. In Cu, an orientation dependence of the characteristic PSB distributions developed near indentations is also found (White,
1984; Charsley and White, 1987). It should be noted that generally for Al crystal orientations, unlike Cu, the effect of indenting was often to cause fatigue slip bands to occur at, and to extend over, large distances from the indentation centres, the indentation thus causing PSBs not to be formed in the immediate region around the indentation pit itself. Hence regarding the effect of indenting single crystals there appear to be two counteracting effects; localized hardening around the indentation, and enhancement of PSB initiation during low amplitude fatigue. In Al the localized hardening effect is much greater than in Cu and the enhancement of PSB initiation is weaker than observed in Cu.

From the results presented here, it is evident that PSBs occur much closer to unannealed indentations in crystals of orientation C7, with PSBs apparent at \( r \approx d \), than in crystals of orientation C10. Near unannealed indentations in C11, C6 and C3 PSBs are observed to initiate in the range \( 0.7d \) to greater than \( 6d \), (the indentations themselves are separated by a distance of about 33 \( d \)). In comparison, in Cu PSBs were found to initiate readily in the range \( d/2 < r < 3d/2 \) (White, 1984) i.e. PSBs were found to occur closer to, and in a more limited region near, indentations in Cu than is usually observed in Al for the range of crystal orientations investigated. It should be noted that for Al the final strain amplitude used was either \( \epsilon_t = 2 \times 10^{-4} \) or \( 4 \times 10^{-4} \), whereas for Cu the final amplitude used was either \( \epsilon_t = 8 \times 10^{-4} \) or \( 9 \times 10^{-4} \). In particular, comparing Al and Cu crystals of similar orientation (crystal C11 and the Cu crystal with \( \theta_0 \sim 20^\circ \) from [001]), PSBs were
observed at distances in the range 0.7 d to > 6.5 d for Al, and in the range 0.5 d to 0.9 d for Cu. In this case the indented Al crystal (C11) was fatigued at a lower plastic strain amplitude than the Cu crystal of similar orientation.

Near unannealed S type indentations in C7 and near unannealed S type indentations in C10, there are reproducible angular zones which are free or nearly free of PSBs, although in C10 PSBs occur over a greater angular region than in C7. In C11, near both D type indentations there are also angular regions which are free of PSBs. Similarly, PSB free angular zones were observed near indentations in Cu (Charsley, Puttick and White, 1981; White, 1984; Charsley and White, 1987). It should however be noted that PSBs are generally apparent in all angular regions around indentations for S type unannealed and annealed indentations in C11 (except near C11.1SU and C11.1OSU where fine slip lines are apparent in all angular regions), annealed S type indentations in C10, and also near indentations in C6 (apart from C6.1SU where only fine slip is observed) and C3.

Characteristic PSB distributions are also developed in Al near indentation pits which have been polished back by various amounts. This is also found to occur in Cu (White (1984), Charsley and White (1987)).

In Cu two distinct types of PSBs are found near indentations following fatigue. These consist of bands which continue to lengthen throughout the test, and very short non-propagating bands which form
in groups. The occurrence of these very short bands is very dependent on crystal orientation (Charsley and White, 1987). In Al bands were, for most crystal orientations, found to be exclusively of the propagating type. It was only in C3 that groups of short bands were observed, and these bands were very fine and less well defined than those typically found in Cu. In Cu, crystals which exhibited short non-propagating bands were mainly those orientations with \( t_0 \) 12\(^\circ\) from [011] and 17\(^\circ\) from [001], whereas the Al crystal C3 was of similar orientation to a Cu crystal with \( t_0 \) ~ 22\(^\circ\) from [011].

With regard to the effect of annealing treatments, similar PSB distributions are developed near unannealed and annealed indentations in Al after a given number of fatigue cycles, although some differences between the distributions for these two cases are found. Comparing annealed and unannealed indentations in C10 and C3, and S type indentations in C11, PSBs are apparent closer to annealed indentations, and are either observed over greater angular regions (C10) or are more pronounced (C3, C11), than for unannealed indentations. Near annealed indentations in C11 PSBs appear to be longer and straighter than those near unannealed indentations. Near these annealed indentations in C3, C10 and C11 the closest PSBs usually form at a distance \( r > d/2 \) in all cases except near the annealed S type indentation in C11 where PSBs have nucleated at \( \leq d/2 \) (see Fig. 6.9 c) and also near some D and S type annealed indentations in C3 where PSBs are apparent at \( d/2 \) (see Fig. 6.32 c and 6.36 c). Near S type annealed and unannealed indentations in C6
the closest PSBs form at the same distances in each case. Near D type indentations in C11 the closest PSBs form near unannealed indentations (see Fig. 6.9 b). Similar observations, where localization of slip band formation remains after annealing, have been made near annealed indentations in Cu. It should be noted that near annealed Cu indentations PSBs were found to nucleate at the indentation edges, although this was not observed near unannealed Cu indentations.

PSBs were not observed within either annealed or unannealed indentations in Al for any crystal orientation apart from C11. In the crystal of orientation C11, PSBs were observed only within the annealed S type indentation pit which had been annealed at 0.88 Tm (where Tm is the melting point temperature of the material). However, for annealed S type indentations in Cu crystals of orientations with θo 12° from [111], PBSs were frequently observed at an early stage within the indentation pit for indentations which were annealed at 0.87 Tm (White, 1984; Charsley and White, 1987).

In Cu, larger indentations were found to result in the earlier formation of PSBs, although the distribution of fatigue slip bands developed was closely similar to that near a standard size indentation. Such an effect has not been observed in Al, in fact near larger indentations (C11.1SU and C11.10SU) only very fine slip bands have been observed, although slip on more than one slip plane is found near such indentations.
Near etch pits in Al, PSB distributions similar to those found near indentations were developed, however PSBs are apparent at the edges and within the etch pit in C3, whereas no PSBs have been observed within the indentation pits for this crystal orientation. In comparison, although fatigue slip bands near etch pits in Cu were found to nucleate at the pit edges at \( \theta = 90^\circ \), the effect was found to be much less extensive than that which occurs near indentations. Thus much more pronounced PSBs are observed near etch pits in Al than observed in Cu.

Regarding similarities in the crystal orientations used in this study for Al and those used by White for Cu (White, 1984; Charsley and White, 1987), orientation C3 is close to a Cu crystal with \( t_o \sim 22^\circ \) from [011], and orientation C11 is close to a Cu crystal which had \( t_o \sim 20^\circ \) from [001]. Crystals orientations successfully tested for Cu were those which had an angle between the surface normal and the primary slip direction \( > 59^\circ \). For the Cu crystal with \( t_o \sim 22^\circ \) from [011] this angle was less than 59°, hence White found that the crystal was unsuitable for the fatigue testing system due to permanent distortion of the crystal after fatigue cycling at a total strain amplitude of \( \varepsilon_t = 8 \times 10^{-4} \). On the contrary, the Al crystal of orientation C3 was successfully fatigued to \( 8 \times 10^4 \) cycles using wheel \( W_1 (\varepsilon_t = 2 \times 10^{-4}) \), which resulted in prominent PSB formation as outlined in sections 6.2.5 (i) and 6.2.5 (ii). Comparing the crystal of orientation C11 for Al with the Cu crystal of similar orientation investigated by White (1984), clear differences in the PSB distributions developed are apparent. In the crystal of
orientation C11 propagating PSBs only were developed parallel to the
cross slip planes, whereas in Cu crystals short non-propagating
bands were formed parallel to the critical plane and long
propagating bands were developed parallel to the primary plane.
Comparing the same type of indentation (unannealed D-type) in Al
crystals of orientation C11 with those in Cu crystals of similar
orientation, PSBs are not apparent in the angular region $\theta \sim 230^\circ - 110^\circ$ for the former, whereas for the latter PSB free regions occur
at $\theta \sim 90^\circ - 150^\circ$ and $270^\circ - 300^\circ$. It should be noted that
differences in the PSB distributions developed near unannealed and
annealed indentations were apparent for Al crystals of orientation
C11 (as outlined above and in section 6.2.3 (i)), however for Cu
crystals of similar orientation no significant differences were
observed between the distributions near annealed and unannealed
indentations. This may be due to differences in the annealing
temperatures used for the two materials in this case; for Al,
annealed indentations were annealed at a temperature of $0.88 T_m$
whereas in Cu the annealing temperature used was $0.72 T_m$. Finally,
in Al crystals of orientation C11 the first PSBs appeared after $2 \times 10^3$ cycles using wheel $W_1$ and on continued fatigue cycling
deformation of the narrow end of the crystal occurred, whereas in Cu
crystals of similar orientation no fatigue slip bands were observed
until $2 \times 10^4$ cycles (at a total strain amplitude of $\varepsilon_t = 9 \times 10^{-4}$)
and cycling was continued to $6.1 \times 10^4$ cycles without crystal
deformation. It should be noted that the value plastic strain
amplitude used to fatigue the Al crystal C11 was less than that used
in the case of the Cu crystal of similar orientation.
In conclusion, it is apparent that indentations in Al clearly affect the nucleation of PSBs. Considering, for example, Figs. 6.42 a - h (crystal C3) the indentation does appear to be a determining factor for the formation of PSBs: despite the point that PSB arrangements are not identical near the various indentations, certain PSBs occur in almost the same position in relation to the pit for all cases (e.g. those PSBs occurring at positions corresponding to the angular regions 240° - 350° and 100° - 170°). Some similarities in crystal orientation effects for Al and Cu specimens are found, since in both materials propagating PSBs occurred near indentations in crystals of all orientations, and the occurrence of the short non-propagating bands was very dependent on crystal orientation. Nevertheless considerable differences exist between PSB formation near regions of surface damage in Al compared with Cu, as outlined above.
CHAPTER 7

TEM OF DISLOCATION STRUCTURE NEAR INDENTATIONS IN AL

7.1: Introduction

Initially, the dislocation structures produced near unannealed indentations made within a single grain in polycrystalline Al are examined. In one case, a detailed study of changes in the dislocation structure as a function of distance from the indentation centre is made. In addition, the dislocation structure observed near the edge of an unannealed indentation in a single crystal of Al is examined. The types of dislocation structures observed within indentations which have been annealed in polycrystalline material are then considered. Subsequently, a comparison between the structures near unannealed and annealed indentations within a single grain in polycrystalline Al is made. Finally, the combined effects of indentation followed by fatigue on the resulting dislocation structure are investigated.

It has been possible to study the dislocation structure at various depths within indentations by examining foils with perforations which extend to different depths within the indentation pits, or which have been electropolished thus removing some thickness of the original surface. For a number of areas around various indentations, determination of the indices of the planes of the
observed dislocation walls and boundaries have been made (where appropriate) using the method outlined in section 5.6.1.

Many of the indentations examined have been made in polycrystalline Al grains which have [001] surface normals. This enables any effects on dislocation microstructure due to indenter orientation to be examined, and permits the dislocations observed experimentally to be compared with slip systems predicted by the Dyer model for indentation (Dyer, 1965) which has been outlined in section 3.3.

With regard to identifying indented polycrystalline specimens, indentations are denoted by the foil number ($F_n$) and the indentation number ($x$), thus indentation number 1 in foil 7 is referred to as $F_71$.

7.2: Experimental Results

7.2.1 (i): Unannealed Indentations in Polycrystalline Al

Fig. 7.1 is an optical micrograph of the unannealed indentation made in specimen $F_71$ on which the positions of areas within the indentation pit examined by TEM are denoted A, B and C respectively. The original indented surface is present for this specimen, and the areas examined within the indentation are at a depth of 3.7 $\mu m$ below the specimen surface. A typical example of cell structures and some regions exhibiting Moiré fringes found in area A is shown in Fig.
7.2. Changes in contrast across the cell walls are also apparent. An additional example of such cell structures is shown in Fig. 7.3 (Area B). Burgers vector analysis of area B revealed that dislocations marked e which extend from the cell walls have $b = \frac{1}{2}[011]$. Burgers vector analysis was also carried out for region C (Fig. 7.4) and this showed that dislocations extending from the cell walls, denoted f, have $b = \frac{1}{2}[01\bar{1}]$. Dislocations marked n and p have Burgers vectors $b = \frac{1}{2}[1\bar{1}0]$ or $\frac{1}{2}[101]$ and $b = \frac{1}{2}[110]$ or $\frac{1}{2}[10\bar{1}]$ respectively. The cell structures found in all areas are usually elongated in appearance and typically have a width of 0.5 \(\mu m\). This is smaller than the cell sizes usually observed after fatigue of Al at room temperature or at 77 K, which indicates a higher flow stress than the peak fatigue stress.

Examples of the dislocation structure observed at various positions within the unannealed indentation pit in specimen F131 are presented in Figs. 7.6 - 7.12. The positions of these areas are denoted A - G on Fig. 7.5, which is an optical micrograph of the specimen. The areas examined within this indentation are at a depth of 2.1 \(\mu m\) below the surface of the specimen, and the indented surface has not been electropolished during TEM preparation. For this indentation the dislocation structure at 0.9 \(d\) (area H) and 1.4 \(d\) (area I) is also illustrated (see Figs. 7.13 - 7.15). As shown by the micrographs presented, within the indentation pit elongated or irregular cell structures which typically have a width of 0.5 \(\mu m\) are again present in all areas. Dislocations (denoted a) extending across the cells in area A (Fig. 7.6) are found to have $b = \frac{1}{2}[110]$. 
In area B the majority of dislocations are visible for both $g_{020}$ (Fig. 7.7) and $g_{200}$ (not illustrated), indicating that these dislocations have $b = \#[110]$ or $\#[1\bar{1}0]$. By considering images of area C taken under a number of different operating reflections (examples of all $g$ not illustrated) many of the dislocations in Fig. 7.8 at $d$ are out of contrast for $g_{131}$, revealing that these have $b = \#[10\bar{1}]$. The presence of dislocations which have $b = \#[10\bar{1}]$ is also indicated in this area. By examining micrographs with $g_{020}$ and $g_{220}$ (neither operating reflection shown) in addition to $g_{200}$ for area F, those dislocations marked a (Fig. 7.11) are found to have $b = \#[110]$ and the majority of dislocations in the cell walls are of this type; dislocations which have $b = \#[10\bar{1}]$ or $\#[1\bar{1}0]$ are marked j. There are also dislocations which have $b = \#[011]$ or $\#[01\bar{1}]$ present in area F. In area G (Fig. 7.12) dislocations which are parallel to $[11\bar{1}]$ are found to be out of contrast for $g_{220}$ and hence correspond to dislocations which have $b = \#[110]$. At H, which is at $\sim 0.9$ d from the indentation centre, dislocation tangles are evident (see Figs. 7.13 and 7.14). Some grouping of dislocations approximately parallel to $[0\bar{1}0]$, $[110]$ and $[1\bar{1}0]$ (corresponding to $(100)$, $(1\bar{1}0)$ and $(110)$ planes respectively) can be seen in this area. In Fig. 7.14 the disappearance of dislocations which are approximately parallel to $[110]$ in the arrowed vertical groupings for $g_{020}$ indicates that these have $b = \#[10\bar{1}]$ or $\#[1\bar{1}0]$. Many dislocations which are approximately parallel to $[110]$ are out of contrast for $g_{200}$ indicating the presence of dislocations with $b = \#[011]$ or $\#[01\bar{1}]$. At I (located at $\sim 1.4$ d from the indentation centre) there are again dislocation tangles, with some grouping of dislocations.
approximately parallel to [100] and [120] (corresponding to (010) and (210) planes respectively). The dislocation density in the regions between the grouped dislocations is noticeably less than at 0.9 d. Slip traces parallel to [110] and [110] are apparent in both areas H and I.

A more detailed analysis of the dislocation structure around an unannealed indentation has been carried out for specimen F133. The dislocation structure around this indentation has been studied at the following distances from the indentation centre, 0.5 d (i.e. at the edge of the indentation), 1.2 d, 2.3 d and 3.3 d (see Fig. 7.16a and 7.16b). The indentation was made within a grain which was ~180 μm in size, positioned such that the indentation corners were approximately 10 μm, 25 μm, 30 μm and 150 μm from the grain edges. The indented surface was not electropolished during TEM preparation and has thus been retained for this specimen.

At 3.3 d (Fig. 7.17) the dislocation arrangement consists of loose tangles of dislocations with areas of low dislocation density between the tangles. Examination of micrographs taken using different operative reflections, g, show that the majority of dislocations have Burgers vectors \( \mathbf{b} = \mathbf{M}[110] \) with the vertical grouping of dislocations (indicated by the arrow) mainly of this type, and those approximately parallel to [\(1\overline{1}0\)] mainly with \( \mathbf{b} = \mathbf{M}[011] \) or \( \mathbf{M}[01\overline{1}] \). Dislocations with Burgers vectors \( \mathbf{b} = \mathbf{M}[\overline{1}10] \) and \( \mathbf{M}[101] \) or \( \mathbf{M}[10\overline{1}] \) are also present in the area.
At 2.3 d (Fig. 7.18) a similar arrangement is apparent, although a greater number of dislocations appear to be present in the tangles. The majority (~70%) of the dislocations which are approximately parallel to [110] have Burgers vectors $b = \frac{1}{4}[110]$, and in the arrowed vertical grouping approximately 70% have Burgers vectors $b = \frac{1}{4}[110]$. There are also dislocations with Burgers vectors $b = \frac{1}{4}[011]$ or $\frac{1}{4}[011]$ and $b = \frac{1}{4}[101]$ or $\frac{1}{4}[101]$ present.

Dislocation tangles are again evident at 1.2 d (Fig. 7.19) but the dislocation density is much higher than in the previous two areas mainly because the dislocation free areas are very much smaller. The majority (~80%) of dislocations have Burgers vectors $b = \frac{1}{4}[110]$ or $\frac{1}{4}[110]$ and there is some grouping of dislocations along the directions indicated by the arrows. Dislocations which also have Burgers vectors $b = \frac{1}{4}[011]$ or $\frac{1}{4}[011]$ and $b = \frac{1}{4}[101]$ or $\frac{1}{4}[101]$ are present in the area.

At the corner of the indentation (0.5 d) an elongated cell structure lying approximately parallel to [110] is apparent (Fig. 7.20). Dislocations present in this area have the following Burgers vectors: $b = \frac{1}{4}[110]; \frac{1}{4}[101]; \frac{1}{4}[110]$ or $\frac{1}{4}[101]$ and $\frac{1}{4}[011]$ or $\frac{1}{4}[011]$.

The extent of the effect of indenting with regard to the dislocation structure produced is illustrated by the observation of loose dislocation tangles and dislocation networks (Fig. 7.21) observed at position E in a grain adjoining the indented grain. Full Burgers vector analysis of the dislocations in this area was not possible,
however dislocations marked j which are approximately parallel to [100] have \( b = \frac{1}{2}[110] \) or \( \frac{1}{2}[101] \), and the presence of dislocations which have \( b = \frac{1}{2}[011] \) or \( \frac{1}{2}[011] \) is indicated by comparing Fig. 7.21 with the area imaged under \( g_{020} \) (not illustrated).

Finally, dislocation structures observed at various positions around the unannealed indentation in F268 are presented in Figs. 7.23 - 7.26 d. Fig. 7.22 is an optical micrograph to show the positions of the areas A, B, C and D investigated. For this specimen a thickness of approximately 2 \( \mu \text{m} \) was electropolished from the indented surface during TEM preparation, and areas at depths of \(~7 \mu \text{m} \) (area D) and 2 \( \mu \text{m} \) (areas A - C) below the original indented surface were examined. In area A which is located at 0.5 \( d \) in Fig. 7.22, dislocation tangles are apparent with some grouping of dislocations parallel to [110]. Burgers vector analysis of the area in Fig. 7.23 shows that most of the dislocations parallel to [110] had \( b = \frac{1}{2}[110] \) since these were out of contrast for \( g_{220} \). Analysing images of area B (Figs. 7.24 a and b) taken under different operating reflections (not illustrated) reveals that the dislocations which extend from the cell walls have \( b = \frac{1}{2}[011] \) or \( \frac{1}{2}[011] \) (denoted \( k \) on Fig. 7.24 b). Most of the dislocations (approximately 70%) in the dislocation networks surrounding the dislocation free areas in Fig. 7.24 b are found to be out of contrast for \( g_{131} \) and hence have \( b = \frac{1}{2}[101] \). The elongated cells in this area are typically \( ~0.6 \mu \text{m} \) wide. Burgers vector analysis of area C (Figs. 7.25 a to f) shows that dislocations which are approximately parallel to [210] (corresponding to \( 120 \) planes) in Fig. 7.25 b and to [210]
(corresponding to (120) planes) in Fig. 7.25 c are out of contrast for $g_{311}$ and thus have $b = \mathcal{N}[011]$, which is the predominant Burgers vector in region C. In area D (Fig. 7.26 a) which is within the indentation pit dislocation cell structures are seen. The dislocation extending into the cell denoted a in Fig. 7.26 b has $b = \mathcal{N}[110]$, and approximately half the dislocations present in the cell walls are also of this type. Other dislocation types present within the cell walls have $b = \mathcal{N}[101]$ and $\mathcal{N}[1\bar{1}0]$ and examples of the latter type are marked b on Fig. 7.26 b. Small dislocation loops marked c on Fig. 7.26 b have $b = \mathcal{N}[101]$. Changes in contrast across the cell walls in Figs. 7.26 a and b indicate misorientation across them. In order to resolve details of the dislocation structure in the area in Fig. 7.26 c, which adjoins the area in Fig. 7.26 a, the weak beam dark field image Fig. 7.26 d was taken. This shows an example of a dislocation network (denoted N on this figure) present in the cell wall. This network consists of dislocations which have $b = \mathcal{N}[011]$ or $\mathcal{N}[01\bar{1}]$ and $b = \mathcal{N}[101]$ or $\mathcal{N}[10\bar{1}]$ in approximately equal numbers. The cells in this area are typically 0.5 μm in width.

7.2.1 (ii): Unannealed Indentations in Single Al Crystals

In single crystal material, of orientation $\sim 5^\circ$ from [013], the dislocation structure has been studied for an unannealed indentation at 0.5 d only (area A, Fig. 7.27). The indented surface was retained during TEM preparation for this specimen. It is found that cell structures, which are typically of $\sim 0.5 \mu m$ width, are apparent in
this area. By comparing images taken using different operating reflections, the cell walls are found to be made up of a number of different Burgers vectors. At e in Fig. 7.28 a dislocations present have Burgers vectors \( b = \frac{1}{2}[011] \). The weak beam dark field image shown in Fig. 7.28 b shows details of the dislocations in the cell walls and dislocation networks present in this area.

7.2.2: Annealed Indentations in Polycrystalline Al

Fig. 7.29 is an optical micrograph of the surface appearance of the annealed indentation in F274 and shows the positions of the areas (A - C) examined. These areas are related to a low magnification TEM micrograph of the region around the perforation in Fig. 7.30. For this specimen the original specimen surface has been retained, and areas at depths of 3.1 \( \mu m \) (area C) and 3.4 \( \mu m \) (areas A and B) below the indented surface have been investigated. In regions within the indentation pit, the dislocation density is relatively low and dislocation boundaries and isolated dislocations are observed in all areas. Walls of dislocations approximately parallel to \([2\overline{1}0]\)
(corresponding to (120) planes) are apparent in Fig. 7.31 a (area A) but cell structures (which are found in similar areas near unannealed indentations) are not seen. Dislocations marked k in Fig. 7.31 a have \( b = \frac{1}{2}[011] \) or \( \frac{1}{2}[011] \); those marked j in Fig. 7.31 b have \( b = \frac{1}{2}[101] \) or \( \frac{1}{2}[101] \). Another example of a dislocation boundary observed in a different area (B) is shown in Fig. 7.32. In this case the dislocations appear to be approximately parallel to \([\overline{1}1\overline{1}]\)
(corresponding to (110) planes), and by considering micrographs taken under different operative reflections (not illustrated) the boundary is found to consist of dislocations which have $b = \frac{1}{2}[110]$. In area C (Figs. 7.33 a - c) similar microstructure is found, and the dislocations denoted r are out of contrast for $g_{111}$ and thus have $b = \frac{g}{2}[110]$ or $\frac{g}{2}[101]$ or $\frac{g}{2}[011]$. Dislocations are found to form boundaries parallel to [111] and [021] (corresponding to (110) and (211) planes respectively) in area C.

A second specimen containing an annealed indentation, F272 showed that in all areas around the indentation pit either a relatively low dislocation density (compared with similar regions for unannealed indentations) or networks or boundaries of dislocations were observed. Fig. 7.34 is a schematic diagram to show the areas examined within the indentation pit. Areas at depths of 1.8 $\mu$m (areas A and B) and 2.4 $\mu$m (area C) below the indented surface, which was retained during TEM preparation, have been examined in this specimen. In area A, shown under different operating reflections in Figs. 7.35 a - h, there is some grouping of dislocations parallel to [100] and [010] (corresponding to (010) and (100) planes respectively). Examples of dislocations which have $b = \frac{1}{2}[110]$, $\frac{g}{2}[110]$, $\frac{g}{2}[101]$ and $\frac{g}{2}[011]$ are marked a, b, c and f respectively on Figs. 7.35 a and c. The boundary with dislocations marked b in Fig. 7.35 a shows strong contrast only in Figs. 7.35 a, f and h but not in Fig. 7.35 c. By considering the Bragg angles for these cases, the angle across this boundary is estimated to be 0.4'. In area B (Figs. 7.36 a - d) a low density of dislocations,
consisting of dislocation tangles, is observed. The presence of dislocations which have $\mathbf{b} = \mathbf{M}[011]$ or $\mathbf{M}[0\bar{1}1]$ and $\mathbf{b} = \mathbf{M}[101]$ is indicated in this area. Most dislocations are out of contrast for $g_{311}$ indicating that these have $\mathbf{b} = \mathbf{M}[011]$. Fig. 7.36 d shows some grouping of dislocations parallel to [311] and [2\bar{1}0] directions, which correspond to (011) and (\bar{1}22) planes respectively. Figs. 7.37 a - h show the dislocation structure in area C under different operating reflections. The dark field micrographs, Figs. 7.37 c and g, resolve details of the dislocation structure which are not readily seen in the bright field images. At N (see Fig. 7.37 a) a network of dislocations is apparent. This network consists of dislocations which have $\mathbf{b} = \mathbf{M}[1\bar{1}0]$, $\mathbf{b} = \mathbf{M}[101]$ and $\mathbf{b} = \mathbf{M}[0\bar{1}1]$. Dislocations marked f in Fig. 7.37 b have $\mathbf{b} = \mathbf{M}[0\bar{1}1]$.

7.2.3: Comparison of an Unannealed and an Annealed Indentation Within a Single Grain in Polycrystalline Al

The dislocation structure around an unannealed and an annealed indentation within a single grain of polycrystalline Al was examined for specimen F2711. The positions of TEM areas near the edges and corners of these indentations are shown schematically in Fig. 7.38. By comparing the dislocation structure observed near the indentation edges, A - D, for both cases (see Figs. 7.39 a - 7.40 d) it can be seen that near the unannealed indentation a higher density of dislocations is apparent than for the annealed indentation. In particular near edges C and D of the unannealed indentation cell
structures are apparent whereas such structures are not found near the annealed indentation. Near the annealed indentation there is evidence that dislocation movement has occurred, indicated by slip traces which are visible parallel to [110] and [110].

Considering the indentation corners, near the unannealed and annealed indentations (Figs. 7.41 a - 7.42 d) it is clear that near the unannealed indentation cell structures are observed in all cases, however near the indentation which has been annealed, dislocation tangles are found in all areas. The higher density of dislocations near corner A/B of the annealed indentation may be related to its proximity to the unannealed indentation, because as shown previously, in section 7.2.1 (i), indenting has been found to affect the microstructure to distances of ~ 3.3d.

7.2.4: Effect of Fatigue on the Microstructure Near an Unannealed Indentation

The microstructure observed near an unannealed indentation which was fatigued for \( N = 5 \times 10^3 \) cycles at \( \epsilon_t = \pm 6 \times 10^{-4} \) was examined for specimen F4. For this specimen the original indented surface was retained during TEM preparation, and the area examined was ~ 15 \( \mu m \) below the indented surface. Fig. 7.43 a shows the indentation and perforation within the pit at low magnification. The TEM area examined is denoted A on Fig. 7.43 b, which is a higher magnification optical micrograph of the area. Again, irregularly
shaped cell structures are found in area A (see Fig. 7.44 a) and these typically have a width of \( \sim 0.5 \mu m \). The dislocation structure in this area appears to be similar to that near unannealed indentations which have not subsequently been fatigued. Area A in Fig. 7.44 a is shown under two different operating reflections in Figs. 7.44 b and c, and examination of these micrographs reveals the presence of dislocations which have \( b = \frac{1}{2}[1\bar{1}0] \) and \( b = \frac{1}{2}[1\bar{1}0] \).

7.3: Discussion

Considering observations of dislocation structure near unannealed indentations in polycrystalline Al, at 0.5 d or less irregularly shaped cell structures are found. These cells have curved walls and their interiors are relatively free of dislocations. Changes in contrast observed across the cell walls indicate misorientation between the cells. The point that dislocation walls appear curved indicates the presence of internal stress. The areas examined at 0.5 d or less correspond to the indentation edge or to regions within the indentation pit, which are not favourable sites for PSBs to nucleate in Al during low strain amplitude fatigue. This is not unexpected in view of the expected stability of such a structure, which is formed at much higher values of stress than applied during fatigue at room temperature. It should be noted that the cell sizes produced by indentation are generally smaller than those typically observed after fatigue of Al at room temperature or at 77 K, which suggests a higher flow stress in the case of the former. Thus any
recovery effects will have already occurred during indentation and therefore no recovery effects can be expected subsequently during low amplitude fatigue. This is supported by observations of dislocation structure within the indentation pit after fatigue for \(5 \times 10^3\) cycles at \(\epsilon_t = \pm 6 \times 10^{-4}\), where the structure is similar to that found in similar areas near unannealed non-fatigued indentations. The curvature of the cell walls reveals, however, that this structure cannot be fully recovered, since even with the same cell size a lower energy configuration would result from approximately plane walls. Thus the point that low amplitude fatigue does not produce any recovery indicates that there is no dislocation movement during fatigue.

The cell type dislocation arrangements apparent at 0.5 \(d\) or less resemble the structures produced in Al which has been cold-rolled (Von Heimendahl, 1980; Barlow, Bay and Hansen, 1985). There are high dislocation density regions formed at greater distances from unannealed indentations, although cell structures are not formed. These areas (for example regions in area H in F131) also correspond to areas which are not favourable sites for PSB formation during fatigue. At distances greater than \(\sim d\), where PSBs form readily during fatigue in Al, generally the dislocation density is low (see, for example, Figs. 7.17 and 7.18 at 3.3d and 2.3d) and is found to decrease with increasing distance from the indentation centre. Thus a high dislocation density inhibits the formation of PSBs unless the fatigue amplitude (and stress) are large enough.
In some areas in the range 0.9 d to 3.3 d near unannealed indentations (see Figs. 7.13 - 7.15 and 7.17 - 7.18) slip traces which are approximately parallel to [110] and [110] are apparent, indicating that dislocation movement has occurred. Such dislocation movement, resulting in the formation of slip traces, has been observed in the electron microscope. The absence of slip traces at distances of 0.5 d or less is an indication that dislocations cannot move readily in these areas. This may be due to the high stresses at the cell boundaries or due to dislocation pinning in such areas.

With regard to the effect of annealing on the dislocation structures produced by indenting, evidence of dislocation movement is apparent closer to annealed indentations (see for example, slip traces at 0.5 d (Fig. 7.40 a) in F2711) and the density of dislocations is much lower than in similar regions near unannealed indentations. The cell type structures observed near unannealed indentations are not observed near annealed indentations, instead boundaries of dislocations, isolated dislocations or areas with dislocation tangles (similar to those observed at ~ 2 d near unannealed indentations) are evident. It should be noted that during fatigue PSBs generally nucleate closer to annealed indentations in comparison with unannealed indentations.

The tangle-type of dislocation structure observed at all distances near annealed indentations, and at distances of 1.4 d and at 2.3 d or greater for unannealed indentations in F131 and F133
respectively, appears to be similar to the structure found by Segall and Partridge (1959) in Al stressed up to 3 kg mm\(^{-2}\) in tension.

In order to assess whether any effects on dislocation microstructure due to indenter orientation with respect to directions in the crystal were apparent, unannealed indentations were made in a number of [001] grains in polycrystalline Al such that their edges were parallel to different pairs of directions. Consequently, the edges of unannealed indentations F\(_{131}\), F\(_{133}\) and F\(_{266}\) were made parallel to; [110] and [1\(\bar{1}\)0], [100] and [010], [120] and [2\(\bar{1}\)0] respectively. For comparison, annealed indentations were positioned such that their edges were parallel to [2\(\bar{1}\)0] and [120] (F\(_{274}\)), and [110] and [1\(\bar{1}\)0] (F\(_{272}\)). Considering the dislocation microstructure observed, there do not appear to be any effects due to indenter orientation with respect to directions in the crystal: cells, which are usually elongated to some extent, are apparent at all positions within unannealed indentations (and at all depths examined within the pits) and at their edges. It should be noted that generally cells showed a greater degree of elongation at the indentation corners. Similarly, the planes parallel to which there is a tendency for dislocations to be grouped in regions outside the indenter contact area near unannealed indentations, are \{100\}, \{110\} and \{2\(\bar{1}\)0\} for all indenter orientations. Considering regions examined for annealed indentations (all within the indenter contact region) boundaries of dislocations and dislocation networks are also observed in all regions irrespective of indenter orientation with respect to directions in the crystal. Dislocation boundaries are found to be parallel to
{100}, {110}, {210}, {112} and {122} planes within annealed indentations. At depths of approximately 3 μm below the indented surface dislocation boundaries are found in all regions; at depths of ~2 μm a number of dislocation networks are apparent in addition to boundaries of dislocations. With regard to the Burgers vectors observed at different positions around unannealed and annealed indentations in [001] grains; no specific relationship was found between the indenter orientation with respect to directions in the crystal and the Burgers vectors identified underneath the indenter, at the indentation edges nor outside the indenter contact area. In addition, it should be noted that examining unannealed indentations made in an [011] grain in polycrystalline material and in a single crystal of orientation close to [013], the cell dislocation structures observed within the indentation and at the indentation edge were of the same appearance as those found in [001] grains.

Most of the polycrystalline grains containing indentations which were examined by TEM had [001] surface normals, and thus indentations are made along [001]. In this connection, it should be noted that Dyer (1965) has proposed a dislocation model for f.c.c. metals to account for the experimental observations of ball indentations on (001) Cu crystals (see section 3.3). This model enables preferred slip planes and directions of slip to be determined. Thus TEM examination of a number of indentations in [001] grains permits the Dyer model predictions for various regions within and outside the indenter contact area to be compared with the observed Burgers vectors of dislocations in these regions. It should
be noted that in the Dyer model sets of planes which accomplish lowering of material under the indenter are modelled by diverging pyramids, whilst outside the indenter contact area another set of planes (surrounding the first set) are modelled by converging pyramids.

Considering region H near the unannealed indentation in F131, dislocations were experimentally found to have \( \mathbf{b} = \mathbf{h}[101] \) or \( \mathbf{h}[10\bar{1}] \), and \( \mathbf{b} = \mathbf{h}[011] \) or \( \mathbf{h}[0\bar{1}1] \). The Dyer model predicts that in this region the preferred slip direction is \([0\bar{1}1]\) on the \((\bar{1}1\bar{1})\) slip plane. Thus there is agreement between the model prediction and one of the observed possible Burgers vector types. In areas C and G within the indentation in F131, the Dyer model predicts slip on the \((\bar{1}1\bar{1})\) plane in \([01\bar{1}]\) and \([10\bar{1}]\) directions respectively. This prediction is not consistent with the observed Burgers vectors of dislocations in region G (\( \mathbf{b} = \mathbf{h}[110] \)) nor in region C (\( \mathbf{b} = \mathbf{h}[101] \) and \( \mathbf{h}[0\bar{1}1] \)). In area A within this indentation, slip is predicted on the \((11\bar{1})\) plane in the \([0\bar{1}1]\) direction by the Dyer model, however Burgers vector analysis revealed dislocations which have \( \mathbf{b} = \mathbf{h}[110] \) in area A. In region B, the Dyer model predicts that the preferred slip system is the \((11\bar{1})\) slip plane with the \([0\bar{1}1]\) slip direction, whereas dislocations were found experimentally to have \( \mathbf{b} = \mathbf{h}[110] \) and \( \mathbf{h}[1\bar{1}0] \) in this area within the indentation pit. Finally in area F within the indentation in F131 dislocations had \( \mathbf{b} = \mathbf{h}[1\bar{1}0], \mathbf{h}[101] \) or \( \mathbf{h}[10\bar{1}] \) and \( \mathbf{h}[011] \) or \( \mathbf{h}[0\bar{1}1] \). Thus one of the possible Burgers vectors types is in agreement with the Dyer model prediction that the preferred slip direction is \([0\bar{1}1]\) on the \((\bar{1}1\bar{1})\) slip plane.
For regions A and B which are outside the indentation pit \( F_{133} \), the Dyer model predicts that the \((\overline{1}1\overline{1})\) slip plane and \([10\overline{1}]\) slip direction are preferred. This predicted slip direction is consistent with one of the possible Burgers vectors of dislocations in these areas although this was not the prevalent Burgers vector. In addition, dislocations with \( \mathbf{b} = \mathcal{M}[110], \mathcal{M}[1\overline{1}0] \) and \( \mathcal{M}[01\overline{1}] \) or \( \mathcal{M}[011] \) were also apparent in these areas. (It should be noted that \( \mathbf{b} = \mathcal{M}[1\overline{1}0] \) and \( \mathcal{M}[011] \) are on the same planes as \( \mathbf{b} = \mathcal{M}[10\overline{1}] \), which means that shear on the same slip plane could be involved). In region C near this indentation, the \((1\overline{1}1)\) slip plane and \([01\overline{1}]\) slip direction is predicted to be the preferred slip system. Whilst some dislocations in region C are found to have possible Burgers vectors which are consistent with the Dyer model, in addition dislocations which have \( \mathbf{b} = \mathcal{M}[110] \) (which is on the same slip plane as \( \mathbf{b} = \mathcal{M}[011] \)) or \( \mathcal{M}[1\overline{1}0] \) and \( \mathcal{M}[10\overline{1}] \) or \( \mathcal{M}[10\overline{1}] \) (which is also on the same slip plane as \( \mathcal{M}[011] \)) are found. (Thus, three possible Burgers vectors on the same planes are again found, which implies that shear on the same slip plane could be involved). In area D in \( F_{133} \) the Dyer model predicts that the preferred slip direction is \([110]\) on either the \((\overline{1}1\overline{1})\) or \((1\overline{1}1)\) plane. This is consistent with one of the possible Burgers vectors types observed in the area, although dislocations which have \( \mathbf{b} = \mathcal{M}[1\overline{1}0], \mathcal{M}[10\overline{1}] \) and \( \mathcal{M}[01\overline{1}] \) or \( \mathcal{M}[011] \) are also present.

Considering areas A and C around the indentation \( F_{266} \), the Dyer model predicts that the preferred slip directions are \([01\overline{1}]\) and
[101] respectively on the (111) plane. In these areas the observed Burgers vectors are not consistent with the Dyer model prediction since in area A dislocations have $b = \{110\}$ mainly, and in area C the predominant dislocation type is $b = \{011\}$. In area B in F266 slip in the [011] direction on the (111) plane is the preferred system predicted by the Dyer model, and this is in agreement with one of the possible Burgers vectors of dislocations in this area, although dislocations which have $b = \{101\}$ are also present. In the last area examined in this specimen, area D (which was within the indentation pit), the possible Burgers vector types for dislocations ($b = \{110\}, \{101\}$ and $\{101\}$) are not in agreement with the predicted preferred slip direction [011] on the (111) slip plane.

Considering the annealed indentations F274 and F272 all regions examined are within the indentation pits. For specimen F274, the Dyer model predicts that in area A the (111) slip plane and the [011] slip direction are preferred, whilst experimentally determined Burgers vectors of dislocations in this area are $b = \{011\}$ or $\{011\}$, and $\{101\}$ or $\{101\}$. Thus one of the possible Burgers vectors of dislocations in area A is consistent with the Dyer model. In area B, the (111)[011] slip system is predicted by the Dyer model, however dislocations have $b = \{110\}$ in this area. Finally in region C of F264 the Dyer model predicts that the (111)[101] slip system is preferred, which is not consistent with the possible Burgers vectors ($b = \{110\}$ or $\{101\}$ or $\{011\}$) of dislocations in this area.
In specimen F272 the \((\overline{1}11)\) slip plane and \([101]\) and \([01\overline{1}]\) slip directions are predicted by the Dyer model to be the preferred systems in areas A and B respectively. These preferred directions are consistent with possible Burgers vectors of dislocations in these areas, however in both areas additional dislocations have been identified which have possible Burgers vectors which are not consistent with the predicted slip directions determined using the Dyer model. In region C within this indentation, dislocations with \(b = \frac{1}{2}[10\overline{1}], \frac{1}{2}[101]\) and \(\frac{1}{2}[01\overline{1}]\) are found, which are not consistent with the Dyer model prediction that the preferred slip system is \((111)[10\overline{1}]\).

Applying the Dyer model to the indentation in the single crystal specimen, the predicted preferred slip direction is \([\overline{1}10]\) on the \((111)\) plane, which contrasts with the experimental observation of dislocations which have \(b = \frac{1}{2}[011]\).

Considering regions near the indentation edges or under the indenter for annealed and unannealed indentations in polycrystalline and single crystal Al specimens, poor correlation is found between the preferred slip direction predicted by the Dyer model and the experimentally determined Burgers vectors of dislocations in such areas. The preferred slip direction according to the Dyer model agrees with the possible Burgers vector type found for dislocations in \(~12\%\) of areas beneath or at the indentation edges, and in a further \(23\%\) of such areas one or more additional Burgers vector types are identified, in addition to the type associated with the
preferred direction predicted by the Dyer model. It should be noted that in approximately 53% of all regions examined more than one Burgers vector type has been identified in each case.

With regard to regions away from the indenter contact area, in all areas the preferred slip direction predicted by the Dyer model agrees with one of the Burgers vector types identified in a given region. Nevertheless, in each area studied dislocations present are found to have (up to four) additional Burgers vectors.

Thus, the slip directions predicted by the Dyer model show greater correlation in regions away from the indenter contact area and indenter edges, although generally dislocations with a greater number of Burgers vector types are found in both types of region, compared with the preferred slip direction determined using the Dyer model. This is consistent with the observation that after low amplitude fatigue of Al a larger number of Burgers vectors are active than is usually found in Cu, which is related to the higher stacking fault energy of Al, resulting in greater ease of movement of secondary dislocations.

Considering the effect of indenting on PSB formation, for Cu White (1984) and Charsley and White (1987) suggest that pre-existing dislocations formed during indentation can enhance PSB formation, as outlined in section 3.5. In the present work on Al direct evidence to support this has not been found, although the point that PSBs are most readily formed in regions where there is a moderate density of
dislocations is consistent with Charsley and White's proposal. As noted previously (in section 3.5) it has been suggested by Kuhlmann-Wilsdorf and Nine (1967) that the formation of PSBs is assisted by non-primary slip, whilst Charsley and White (1987) have suggested that the formation of Lomer-Cotrell stair-rod dislocations is a possible mechanism relevant to PSB formation in indented crystals. From the present work on Al, the Burgers vectors of dislocations observed in regions of moderate dislocation density are consistent with these suggestions. Finally it should be noted that the effect of an unannealed indentation in Al in comparison with Cu, is to form a larger PSB-free region around the indentation site. In general terms this can be directly related to the lower work-hardening rate which enables the plastic deformation zone to be more extensive in Al in comparison with Cu. This is related to the larger number of Burgers vectors observed in fatigue and enables cells to be more easily formed. Hence the fatigue plastic strain is to a great extent reduced in this region so that at the edges of the hard region PSB formation is more likely.
CHAPTER 8

CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

8.1: Introduction

In this chapter outlines of the main experimental results presented in earlier chapters are given, and suggestions for future work to be carried out are made.

8.2: Summary of Main Experimental Results

8.2.1 (i): Room Temperature Fatigue of Polycrystalline Al

Dislocation configurations and surface slip markings produced in Al specimens after room temperature fatigue have been studied by TEM and by optical microscopy. Values of total strain amplitude in the range $\pm 4 \times 10^{-4}$ to $\pm 1.9 \times 10^{-3}$ were used to fatigue specimens with a number of cycles between $5 \times 10^3$ to $2 \times 10^4$. Considering the surface slip bands in Al, these were found to be more irregular and wavy than those observed in Cu. With regard to the dislocation structures: after low strain amplitude fatigue dense patches of dislocation loops were found, however at all amplitudes the formation of irregular dislocation walls was apparent. In foils with $<100>$ normals these irregular walls, which had spacings in the range
0.7 μm to 1.0 μm, were often found to be approximately parallel to (100) and (110) planes. Analysis revealed that even at the lowest amplitude at least three, and often six, different Burgers vectors may be present in quite small areas. Thus multiple slip occurs more easily during fatigue at room temperature in Al compared with Cu. At the highest strain amplitudes, a more clearly defined cell structure is formed with narrow cell walls enclosing larger dislocation free regions. The cell walls are found to be preferentially oriented along <100> and <110> directions approximately (corresponding to (100) and (110) planes). In conclusion, structures similar to those found after room temperature fatigue of Cu and Ni at low amplitude (e.g. matrix, PSB ladder structure and labyrinth structure) were not found after room temperature low amplitude fatigue of Al. Dislocation structures typically found in Al after room temperature low amplitude fatigue are more similar to the cell structures found in Cu fatigued at room temperature at higher amplitudes (or to large cumulative plastic strains), the formation of which is associated with increased secondary slip activity.

8.2.1. (ii): Low Temperature Fatigue of Polycrystalline Al

A number of Al specimens were examined after various ageing periods at room temperature following fatigue for $2 \times 10^4$ and $2.2 \times 10^4$ cycles at a total strain amplitude of $\pm 4 \times 10^{-4}$ at 77 K. Either matrix structures similar to those found after room temperature
fatigue of Cu and Ni, but with a greater number of dislocation loops, or well-defined walls of dislocations were found. The walls of dislocations were found to be approximately parallel to \{100\}, \{111\}, \{113\} and \{331\} planes. The structures observed after ageing are more similar to structures observed after low temperature fatigue of Al with specimen ageing minimized than to those produced by fatigue at room temperature.

Dislocation configurations produced in specimens after fatigue at 77 K in which a minimum of specimen annealing was then permitted, were studied for Al fatigued with total strain amplitudes in the range $\pm 4 \times 10^{-4}$ to $\pm 1.4 \times 10^{-3}$ after a number of cycles between $5 \times 10^3$ to $2.2 \times 10^4$. During the early stages of fatigue a matrix structure very similar to that in Cu and Ni is observed, i.e. dense patches of dislocation loops separated by channels which are relatively dislocation free. These low dislocation density channels contain isolated dislocation loops; the density of such loops is significantly higher in Al than for Cu and Ni after room temperature fatigue. At later stages of fatigue (after cumulative strains of $\sim 20$ with a total strain amplitude of $\pm 1.4 \times 10^{-3}$) usually the matrix structures in Al appeared narrower with a greater channel proportion than observed after room temperature fatigue of Cu and Ni. Several Burgers vectors were found to be present in areas exhibiting matrix or matrix-type structures.

For cumulative strains in excess of $\sim 20$ at total strain amplitudes of $\pm 8 \times 10^{-4}$ or greater, a few grains in polycrystalline specimens
exhibited more condensed structures which approximated to the classical PSB wall structure. These structures were less regular than those found in Cu and Ni and with much larger wall separations, e.g., the value of wall separation in ladder-type structures observed in Al is approximately four times larger than that in Cu after fatigue testing at temperatures of 77 K. In one case, walls in the structure which is similar in appearance, although more irregular, to ladder structures observed in Cu have been found to be approximately parallel to (311) planes in Al.

A large fraction of grains in Al were found to contain a labyrinth structure after cumulative strains of ~20 at total strain amplitudes of, or greater than, ±8 x 10^{-4}. This consists of two sets of (100) dislocation walls which are mutually perpendicular, although significant deviations from these planes were often observed. The presence of four or more Burgers vectors was frequently observed in regions which exhibit these walls. The channel width between the (100) walls in areas exhibiting labyrinth structure which was not wholly formed ("uncondensed") was ~2 μm at ε_t = ±8 x 10^{-4}, and decreased by a factor of approximately three for similar types of (100) wall observed after fatigue at ε_t = ±1.4 x 10^{-3}. The channel width (~2 μm) in the fully formed ("condensed") labyrinth structure in Al after fatigue at ε_t = ±1.4 x 10^{-3} was found to be approximately three times that observed in the uncondensed structure, which was apparent in some grains after fatigue under the same conditions. By comparison in Cu and Ni after room temperature fatigue the channel widths in similar labyrinth
structures are typically 0.75 µm and 0.78 µm respectively. Moreover, in Al it was often found that areas in which {100} walls were not fully formed contained regions in which pairs of parallel dislocation walls enclosed square dislocation free areas. The labyrinth structure in Al was not found to be associated with any specimen surface slip marking.

A number of grains contained a single set of parallel {100} walls over large areas after low temperature fatigue at a total strain amplitude of ± 1.4 x 10⁻³. Although this has sometimes been observed in Cu fatigued at room temperature it appears to be more extensive in Al fatigued at 77 K. In the channels between the walls in this structure there is again a high density of dislocation loops. All Burgers vectors types were found to be present in areas in which one set of {100} walls dominated, with \( b = \frac{1}{2}[110] \) and \( \frac{1}{2}[110] \) most prevalent. It should be noted that dislocations having these two Burgers vectors have been predicted for {100} wall formation by Charsley (1981).

In addition, after fatigue under the conditions above (i.e. \( \varepsilon_t = \pm 1.4 \times 10^{-3}, N = 5 \times 10^3 \)) a completely new type of condensed structure was observed in some specimens which had been thinned to retain the surface. This was a well defined "square lattice" of approximately circular dislocation clusters lying in lines parallel to \(<110>\) directions for a foil normal [001] and with a "lattice parameter" of ~ 1.9 µm. The occurrence of this structure was associated with the presence of approximately equal numbers of
dislocations with four distinct Burgers vectors and with a specimen tensile axis close to [010]. The channels between the clusters had a low dislocation density and the structure could be related to a unique surface structure using both optical and scanning electron microscopy. It was concluded that the circular clusters were approximately spherical and formed a three dimensional array which approximated to a face centred cubic lattice. The clusters were considered to represent hard regions, resulting in material being extruded out of the surface between the clusters during fatigue.

Some other wall structures were also observed, these were parallel to \{122\}, \{120\} and \{013\} planes or were of orientations similar to those already seen after room temperature fatigue or after fatigue at low temperature with subsequent ageing at room temperature.

Finally, no clear examples of wavy surface PSBs (as typically found after room temperature fatigue) were apparent after fatigue of Al at low temperature in the present work. However, Mughrabi (1985) has reported observations of "bulgy" extrusions in Al single crystals fatigued at 77 K at a shear strain amplitude \(\gamma_{pl} = 2 \times 10^{-3}\) for 6.5 x \(10^3\) cycles. The "rumpled band" and cross-hatched structures have been observed in the current work after fatigue of Al at 77 K.

8.2.2: Fatigue of Indented Single Crystals
After indenting Al single crystals of different orientations these were cycled in reverse plane bending for several hundred cycles using a small surface strain amplitude (1 x 10^{-4} or 2 x 10^{-4}) before being cycled at higher amplitudes (2 x 10^{-4} or 4 x 10^{-4}). Characteristic intense slip distributions were formed near indentations in Al following low amplitude fatigue, and the PSB distribution developed varied depending on the crystal orientation. Although it was found that the PSB arrangements were not identical for various indentations made within a crystal of a given orientation, certain PSBs were found to occur in almost the same position relative to the pit in all cases. Thus it was concluded that indentations clearly affect the nucleation of PSBs. Similarly, observations of preferential PSB formation near indentations have also been made on fatigued Cu crystals (White, 1984; Charsley and White, 1987). In Al, short non-propagating bands of PSBs were observed only in a crystal of orientation C3 (with an orientation such that t_0 \sim 22\' from [011]), and unlike for pure Cu in general PSBs were found to be exclusively of the propagating type for all Al crystal orientations. Secondly, PSBs were not observed to initiate at distances closer than 0.7 d from unannealed indentation centres in Al and usually extended to distances greater than 6 d. For Cu however, PSBs were observed in the range d/2 < r < 3d/2. Thus in Al a larger PSB free region is formed around the indentation site, and PSBs were apparent over a more extensive region than found in Cu. Comparing Al and Cu crystals of similar orientation (Al crystal C11 and the Cu crystal with t_0 \sim 20\' from [001]) PSBs were observed to initiate in the range 0.7 d to > 6.5 d for Al and in the range 0.5 d
- 0.9 d for Cu. It should be noted that in this case the Al crystal (C11) was fatigued at a lower plastic strain amplitude than the Cu crystal of similar orientation. With regard to the effect of indenting, two counteracting effects therefore seem apparent: a localized hardening around the indentation and an enhancement of PSB initiation during low amplitude fatigue. In Al the first effect is much greater than in Cu, and the enhancement of PSB initiation appears weaker than observed in Cu.

Characteristic distributions were also found near indentations in Al which had been annealed after indenting (this effect has also been found by White (1984) for Cu). The effect of annealing indentations in Al (at either 0.75 T_m or more usually at 0.88 T_m) was generally (in about 70% of cases) to result in PSBs forming closer to indentations and over greater angular regions. Nevertheless, PSBs were seldom observed at the indentation edge and only one example of a PSB occurring within an indentation pit was found. In comparison, in Cu PSBs were frequently observed within S-type indentation pits annealed at temperatures of 0.87 T_m and also at their edges (Charsley and White, 1987).

With regard to the effect of indentation size in Al, unannealed indentations 1.3 and 1.8 times the standard indentation size were examined for orientation C11. Although slip on more slip systems was apparent near the larger indentations, only very fine slip bands were observed after fatigue cycling. In Cu however larger indentations were found to result in earlier PSB formation.
Considering regions near etch pits in Al, similar distributions of PSBs to those observed near indentations were found. It should be noted that clear examples of PSBs extending within etch pits were also apparent. This was in marked contrast to results obtained on Cu crystals (White, 1984; Charsley and White, 1987) where etch pits resulted in much less extensive PSB formation than observed near indentations.

Finally, regarding similarity in Al crystal orientations investigated in the present work and those examined by White (1984) for Cu, the Al crystal of orientation C3 is close to a Cu crystal with $t_0 \sim 22^\circ$ from [011], and C11 is similar to a Cu crystal with $t_0 \sim 20^\circ$ from [001]. Crystal C3 was successfully fatigued to $8 \times 10^4$ cycles at a strain amplitude $\varepsilon_t = 2 \times 10^{-4}$, whereas White (1984) found that Cu crystals of similar orientation were unsuitable for fatigue testing due to distortion of the crystal after fatigue cycling at a total strain amplitude of $\varepsilon_t = 8 \times 10^{-4}$.Comparing the Al crystal of orientation C11 with a similarly oriented Cu crystal, differences in the PSB distributions developed were apparent: in Al propagating PSBs were formed parallel to the cross slip plane, however in Cu short non-propagating bands were formed parallel to the critical plane and long propagating bands were developed parallel to the primary plane. Moreover differences in the angular regions in which PSBs were formed in Al and Cu crystals were found. Differences were also noted in the PSB distributions developed near unannealed and annealed indentations in the Al crystal C11, whereas
no significant differences were observed for Cu. (This may be due to the annealing temperatures used in this case for the two materials; the annealing temperatures used for annealed indentations were 0.88 $T_m$ and 0.72 $T_m$ respectively for Al and Cu). As noted previously, C11 was fatigued at a lower plastic strain amplitude than the Cu crystal of similar orientation, although distortion of the Al crystal was observed on continued cycling after $2 \times 10^3$ cycles, whereas the Cu crystal was fatigued to $6.1 \times 10^4$ cycles with no deformation apparent.

8.2.3: TEM of Dislocation Structure Near Indentations

The results presented show that near the edges of, and within, unannealed indentations (which are not favourable sites for PSBs to nucleate during fatigue in reverse plane bending) the microstructure consists of irregularly shaped dislocation cells in both polycrystalline and single crystal specimens. The curvature of the narrow dislocation walls which enclose relatively dislocation free interiors indicates the presence of internal stress in these areas. Changes in contrast observed across the cell walls indicates misorientation across these walls. Subsequent fatigue of unannealed indentations does not cause changes in this cell structure, and such stability of the structure is expected since it is formed at much higher values of stress than applied during room temperature fatigue. With increasing distance from unannealed indentations the density of dislocations decreases, and in areas where PSBs readily
form during fatigue (e.g. at distances greater than ~ d from the indentation centre) loose dislocation tangles are found. In these areas slip traces are evident indicating dislocation movement, whereas an absence of such traces at 0.5 d or less is a further indication that dislocations cannot move readily in these areas.

The effect of annealing indentations at 0.88 Tm for two hours is to result in dislocation movement being apparent closer to the centre of the indentations (e.g. at 0.5 d from the indentation centre), and to reduce the density of dislocations compared with similar regions near unannealed indentations. Areas with isolated dislocations, dislocation tangles and dislocation boundaries are observed near annealed indentations but areas exhibiting cell structures are not found. It should be noted that during the fatigue of annealed indentations PSBs are generally found to nucleate closer to the annealed indentations in comparison with unannealed indentations.

A number of different indentation orientations with respect to crystallographic directions in polycrystalline Al grains with [001] surface normals were examined. This revealed that the dislocation microstructure and Burgers vectors observed at various positions relative to the indentation were not affected by the indenter orientation. As mentioned previously cell structures, usually elongated to some extent, were observed at all positions within and at the edges of unannealed indentations, whilst at sufficient distances from the indenter contact region dislocation tangles were observed. Cell structures tended to show the greatest degree of
elongation at the indentation corners, irrespective of the indenter orientation with respect to the [001] foil. Similarly dislocation boundaries and networks were found at all positions within annealed indentions irrespective of indenter orientation in the [001] foil.

The indentation of polycrystalline Al grains with [001] surface normals enabled a comparison to be made between the predicted preferred slip directions according to the Dyer model, which was developed for ball indentation of (001) Cu crystals (Dyer 1965), and the observed Burger vectors of dislocations found near indentations in Al.

In general poor correlation was found between the preferred slip direction predicted by the Dyer model and the experimentally determined Burgers vectors of dislocations in areas within, or at the edges of, indentations. Agreement between the model prediction and the observed Burgers vectors was only found in 12% of such areas, and in a further 23% of areas additional Burgers vectors were identified apart from those which correspond with the preferred directions predicted by the Dyer model. In all regions away from the indenter contact area, the preferred slip directions predicted by the Dyer model agreed with one of the possible Burgers vectors identified for dislocations in each region, although dislocations are also found which have (up to four) additional Burgers vectors.

Applying the Dyer model to predict the preferred slip direction for the area examined in the indented single crystal (orientation ~ 5°
from [013]) and comparing this with the observed Burgers vectors of dislocations revealed that the Dyer model was not consistent with the Burgers vectors of dislocations which were identified.

8.3: Suggestions for Future Work

It would be useful to extend the work begun on pure Al to the structurally important Al-6%Zn-2%Mg alloy, which like Al, also shows significant differences in slip in comparison with Cu (due to stacking fault energy and precipitation hardening). Moreover, because the alloy is susceptible to corrosion pitting, it would be useful to examine the effects of well characterised pits produced by spark erosion as well as pits produced by indentation which have associated plastic deformation. The effects should be examined using the optical, scanning electron and transmission electron microscopy techniques developed in the present work on Al.

In the course of the current work, a Woods metal grip system for push-pull fatigue testing of single crystals using a servo-hydraulic machine was developed, although there was insufficient time to enable this technique to be used. The use of this type of apparatus in connection with a strain gauge extensometer would give superior strain control compared with the system used for the experiments described in this work, and it would be useful therefore to extend future work to this type of testing apparatus.
The importance of SEM for revealing PSB details is clear from the micrographs presented here. It would be useful to use the techniques of combined optical and scanning electron microscopy again, to investigate the effects of surface damage on PSB formation for experiments on Al and Al alloys fatigued in push-pull.

In addition, the technique for producing TEM specimens with perforations in the neighbourhood of indentations has been established, and preliminary investigations of the dislocation structures near indentations have been undertaken. Hence experiments should be carried out to relate the surface structures developed near given fatigued indentations in single crystal specimens to the dislocation microstructures: i.e. by examining the development of PSBs near a given indentation and then preparing a TEM specimen with thin areas near this indentation. Such experiments should be carried out for both Cu and Al in order to see whether any of the observed differences in the PSB distributions developed near indentations can be related to the dislocation microstructures. Similarly in the Al alloy it would also be of interest to relate the dislocation structures to the observed PSB distribution.

Since fatigue testing of Ni at low temperature has not previously been reported, it would be of interest to undertake such experiments in order to study the effect on the dislocation microstructure produced, particularly because of the high stacking fault energy of the material.
Finally, since work concerned with the characterisation of the cyclic hardening curve for single Al crystals has not been reported, it would be useful for such experimental studies to be carried out.
Table 4.1: Specimen C3 Indentation Details

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<th>\Phi/°</th>
<th>d/\mu\text{m}</th>
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* U/A indicates whether the indentation is unannealed or annealed: U indicates an unannealed indentation; whilst for annealed indentations the annealing temperature is given in °C.

** Estimated surface thickness removed by electropolishing after indenting.
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<th>(t_r^{\text{E}}/\mu\text{m})</th>
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* U/A indicates whether the indentation is unannealed or annealed: U indicates an unannealed indentation; whilst for annealed indentations the annealing temperature is given in °C.

# Estimated surface thickness removed by electropolishing after indenting.
Table 4.1: Specimen C7 Indentation Details

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* U/A indicates whether the indentation is unannealed or annealed: U indicates an unannealed indentation; whilst for annealed indentations the annealing temperature is given in °C.

** Estimated surface thickness removed by electropolishing after indenting.
Table 4.1: Specimen C10 Indentation Details

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* U/A indicates whether the indentation is unannealed or annealed: U indicates an unannealed indentation; whilst for annealed indentations the annealing temperature is given in °C.

* Estimated surface thickness removed by electropolishing after indenting.
Table 4.1: Specimen C11 Indentation Details

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* U/A indicates whether the indentation is unannealed or annealed: U indicates an unannealed indentation; whilst for annealed indentations the annealing temperature is given in °C.

** Estimated surface thickness removed by electropolishing after indenting.
Table 4.2: Saturation plastic strain amplitudes corresponding to the total strain amplitudes used for polycrystalline Al. Values for $\varepsilon_{pl,cum}$ are also included.

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<td>$W_2$</td>
<td>0.26</td>
<td>$4 \times 10^{-4}$</td>
<td>$1.9 \times 10^{-4}$</td>
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</table>
Fig. 2.1: Cyclic stress-strain curve of a fatigued copper monocystal.
Fig. 2.2: Schematic diagram of a PSB showing dislocation walls composed of vacancy dipoles; the long range effects may be understood by considering the wall as a single dipole of height $h_b$.

The boundaries of the PSB can be thought of as arrays of edge dislocations separated by a wall spacing $d$, and with an extra half plane pointing outwards. The length DE thus spans fewer atomic planes in the PSB than in the matrix and the material in the PSB is strained elastically in tension in the direction of the primary Burgers vector ($AA'$). (After Antonopoulos et al, 1976).
Fig. 2.3 a, b: Schematic diagram of the basic mechanisms of (a) extrusion and (b) intrusion by combined glide and annihilation of dislocations. Full and open symbols correspond to the glide sequences $A-A'$ and $B-B'$ in tension and compression respectively. All dislocations drawn in dipole configurations should be considered to have annihilated, and only the individual dislocations at $A$, $B$, $A'$ and $B'$ have survived the annihilation. The latter dislocations which glide out result in the formation of slip steps as indicated above.

Fig. 2.4: Schematic representation of the formation of rumules. The rumpled band direction lies close to that of the primary plane intersection with the surface and the individual rumules lie along the cross-plane intersection with the surface.

(After King and Teer, 1969).
Fig. 2.5: Diagram to show the variation in fatigue dislocation structures observed with stacking fault energy (SFE) and number of cycles to failure ($N_f$).

(After Lukáš and Klesnil, 1971).
Fig. 2.6: Schematic representation of the dislocation arrangement in persistent slip bands.

(After Mughrabi, 1979).
Fig. 3.1: The directions, \( t_0 \), of the specimen tension-compression axes in the standard stereographic triangle for crystals fatigued by White (1984).
Fig. 4.1: Shape and dimensions of the single crystal specimens.
Fig. 4.2 a: Direction of the specimen axis, $t_0$, and edges, $t_1$ and $t_2$. 
Fig. 4.2 b: Orientations of the single crystal specimens.
Fig. 4.3 a and b: Orientations of indentations with respect to \( t_o \), and definitions of \( r \) and \( \theta \) used to identify areas around indentations. Fig. 4.3 a depicts an S-type indentation; Fig. 4.3 b depicts a D-type indentation.
Fig. 4.4: $\ell, \phi$ coordinates used to specify positions of indentations or etch pits.

Fig. 4.5: Diagram to show the position A, at which the load was applied to the single crystal specimens, and the position B, at which the deflection $h$, was measured.
Fig. 4.6: Schematic diagram of a hysteresis loop.
Fig. 5.1 a: Optical micrograph of PSBs developed after $N = 2 \times 10^4$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. Marker = 100 $\mu$m.

Fig. 5.1 b: Optical micrograph of PSBs developed after $N = 6 \times 10^4$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. Marker = 100 $\mu$m.

Fig. 5.1 c: Optical micrograph of PSBs developed after $N = 1 \times 10^5$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. Marker = 100 $\mu$m.

Fig. 5.1 d: Optical micrograph of PSBs developed after $N = 2.2 \times 10^5$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. Marker = 100 $\mu$m.

Fig. 5.2: Optical micrograph of intersecting bands after $N = 1.8 \times 10^5$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. Marker = 50 $\mu$m.

Fig. 5.3: Optical micrograph of PSBs observed after $N = 5 \times 10^3$ cycles at $\varepsilon_t = \pm 6 \times 10^{-4}$. Marker = 100 $\mu$m.
Fig. 5.4: Optical micrograph of the surface of polycrystalline Al foil after $1 \times 10^4$ cycles at $\varepsilon_t = +1.4 \times 10^{-3}$ and $3 \times 10^3$ cycles at $\varepsilon_t = +1.9 \times 10^{-3}$. Marker = 100 $\mu$m.

Fig. 5.5 a: SEM micrograph of intersecting bands developed at $\varepsilon_t = +1.4 \times 10^{-3}$ after $N = 5 \times 10^3$.

Fig. 5.5 b: SEM micrograph of an area in Fig. 5.5 a at higher magnification.
Fig. 5.6: Stereo-pair showing the dislocation structure developed after $2 \times 10^4$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. \( B = [001] \); \( \varepsilon_0 \geq 0 \); Marker = 1 \( \mu m \).

Fig. 5.7: Stereo-pair showing the dislocation structure developed after $2 \times 10^4$ cycles at $\varepsilon_t = \pm 4 \times 10^{-4}$. \( B = [001] \); \( \varepsilon_0 \geq 0 \); Marker = 1 \( \mu m \).
Fig. 5.8: Dislocation structure developed after $2 \times 10^4$ cycles at $\varepsilon_t = 1.4 \times 10^{-4}$. $B = [001]$; $\delta_{020}$; Marker = 1 $\mu$m.

Fig. 5.9: Higher magnification stereo-pair of the area in Fig. 5.8. $B = [001]$; $\delta_{020}$; Marker = 0.5 $\mu$m.
Fig. 5.10: TEM of substructure produced after $N = 2 \times 10^4$ at $\epsilon_t = \pm 4 \times 10^{-4}$.

$B = [001]$; $g_{200}$; Marker = 1 $\mu$m.

Fig. 5.12: TEM of substructure produced after $N = 2 \times 10^4$ at $\epsilon_t = \pm 4 \times 10^{-4}$.

$B = [001]$; $g_{200}$; Marker = 1 $\mu$m.

Fig. 5.13: Area in Fig. 5.12 at higher magnification.

$B = [001]$; $g_{020}$; Marker = 0.4 $\mu$m.

Fig. 5.14: TEM of substructure produced after $N = 5 \times 10^3$ at $\epsilon_t = \pm 6 \times 10^{-4}$.

$B = [001]$; $g_{020}$; Marker = 0.5 $\mu$m.

Fig. 5.15: TEM of substructure produced after $N = 5 \times 10^3$ at $\epsilon_t = \pm 6 \times 10^{-4}$.

$B = [\bar{1}12]$; $g_{111}$; Marker = 1 $\mu$m.
Fig. 5.16: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 6 \times 10^{-4}$.
B = [112]; $\Sigma_{111}$; Marker = 1 $\mu$m.

Fig. 5.17: Substructure after $N = 2 \times 10^4$ at $\varepsilon_t = \pm 6 \times 10^{-4}$.
T = tension-compression axis.
B = [001]; $\Sigma_{020}$; Marker = 1 $\mu$m.

Fig. 5.18: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 1.4 \times 10^{-3}$.
B = [001]; $\Sigma_{020}$; Marker = 0.5 $\mu$m.

Fig. 5.19: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 1.4 \times 10^{-3}$.
B = [001]; $\Sigma_{020}$; Marker = 0.5 $\mu$m.

Fig. 5.20: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 1.4 \times 10^{-3}$.
B = [001]; $\Sigma_{020}$; Marker = 0.5 $\mu$m.

Fig. 5.21 a: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 1.4 \times 10^{-3}$.
B = [001]; $\Sigma_{020}$; Marker = 1 $\mu$m.
A corresponds with the area in Fig. 5.21 a. Marker = 25μm.

Fig. 5.21 b: Optical micrograph of specimen surface appearance.

B 10011; 9020; Marker 0.25μm.

Fig. 5.21 c: SEM of area featured in Fig. 5.21 b, showing details of region with cross-hatched features.

Fig. 5.22 a: TEM of substructure produced after N = 5 x 10³ at εₜ = ±1.4 x 10⁻³. B = [001]; ε₀₂₀; Marker =0.25μm.

Fig. 5.22 b: TEM of substructure produced after N = 5 x 10³ at εₜ = ±1.4 x 10⁻³. B = [001]; ε₀₂₀; Marker =0.25μm.

Fig. 5.22 c: Specimen surface appearance, where A and B correspond to the areas in Fig. 5.22 a and b. Marker = 20μm.

Fig. 5.23: Substructure after N = 10⁴ at εₜ = ±1.4 x 10⁻³ and N = 3 x 10³ at εₜ = ±1.9 x 10⁻³. B = [112]; ε₁₁₁; Marker = 1 μm.
Fig. 5.24: TEM of substructure after \(N = 2 \times 10^4, \varepsilon_t = \pm 4 \times 10^{-4}\) at 77 K. Aged for 7 days at 293 K.
\(B = [\bar{1}12]; g_{220};\) Marker = 0.5 \(\mu m\).

Fig. 5.25: Substructure after 
\(N = 2 \times 10^4, \varepsilon_t = \pm 4 \times 10^{-4}\) at 77 K. Aged for two months at 293 K.
\(B = [\bar{1}11]; g_{220};\) Marker = 4 \(\mu m\).

Fig. 5.26: Higher magnification micrograph of an area in Fig. 5.25 above.
\(B = [\bar{1}12]; g_{\bar{1}31};\) Marker = 1 \(\mu m\).

Fig. 5.27: Weak-beam dark field image of part of Fig. 5.25.
\(B = [\bar{1}12]; g, 3g\ with g_{111};\)
Marker = 0.25 \(\mu m\).

Fig. 5.28: TEM of substructure after \(N = 2 \times 10^4, \varepsilon_t = \pm 4 \times 10^{-4}\) at 77 K. Aged for 10 weeks at 293 K.
\(B = [\bar{1}12]; g_{\bar{1}31};\) Marker = 2 \(\mu m\).

Fig. 5.29: TEM of substructure after \(N = 2 \times 10^4, \varepsilon_t = \pm 4 \times 10^{-4}\) at 77 K. Aged 11 weeks at 293 K.
\(B = [013]; g_{200};\) Marker = 0.5 \(\mu m\).
Fig. 5.30: TEM of substructure after $N = 2.2 \times 10^4$, $\varepsilon_t = \pm 4 \times 10^{-4}$ at 77K. Aged for 7 days at 293K. 
$B = [013]$; $g_{200}$; Marker = 0.5 $\mu$m.

Fig. 5.31: Substructure after $N = 2.2 \times 10^4$, $\varepsilon_t = \pm 4 \times 10^{-4}$ at 77K. Aged for one month at 293K. 
$B = [\bar{1}12]$; $g_{311}$; Marker = 0.5 $\mu$m.

Fig. 5.32: Dislocation structure after $N = 2.2 \times 10^4$ at $\varepsilon_t = \pm 4 \times 10^{-4}$ at 77K. 
$B = [\bar{1}12]$; $g_{\bar{1}1\bar{1}}$; Marker = 0.25 $\mu$m.

Fig. 5.33: Substructure after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 6 \times 10^{-4}$ at 77K. 
$B = [011]$; $g_{1\bar{1}1}$; Marker = 1 $\mu$m.

Fig. 5.34: Higher magnification micrograph of part of area in Fig. 5.33. 
$B = [\bar{1}12]$; $g_{\bar{1}1\bar{1}}$; Marker = 0.5 $\mu$m.

Fig. 5.35: Weak-beam dark field image of part of area in Fig. 5.33 showing dislocation loops. 
$B = [011]$; $g_{11\bar{1}}$; Marker = 0.5 $\mu$m.
Fig. 5.36: TEM of substructure produced after $N = 5 \times 10^3$ at $\varepsilon_t = \pm 6 \times 10^{-4}$ at 77 K.
$B = [001]$; $g_{200}$; Marker = 1 $\mu$m.

Fig. 5.37: TEM of substructure produced after $N = 1 \times 10^4$ at $\varepsilon_t = \pm 8 \times 10^{-4}$ at 77 K.
$B = [011]$; $g_{200}$; Marker = 2 $\mu$m.

Fig. 5.38: Area in Fig. 5.37 at higher magnification and with different operating $g$.
$B = [011]$; $g_{111}$; Marker = 0.5 $\mu$m.

Fig. 5.39: Weak-beam dark field image of part of area in Fig. 5.37. $B = [011]$; $g$, $3g$ with $g_{111}$. Marker = 0.4 $\mu$m.

Fig. 5.40: TEM of substructure produced after $N = 1 \times 10^4$ at $\varepsilon_t = \mp 8 \times 10^{-4}$ at 77 K.
$B = [\bar{1}12]$; $g_{220}$; Marker = 3 $\mu$m.

Fig. 5.41: TEM of substructure produced after $N = 1 \times 10^4$ at $\varepsilon_t = \pm 8 \times 10^{-4}$ at 77 K.
$B = [001]$; $g_{020}$; Marker = 5 $\mu$m.
Fig. 5.42: TEM of substructure in an area in Fig. 5.41 to show details of walls.
\( B = [001]; \ g_{020}; \text{Marker } = 1 \mu \text{m}. \)

Fig. 5.44: Area near region in Fig. 5.43 above showing labyrinth type structure.
\( B = [001]; \ g_{020}; \text{Marker } = 1 \mu \text{m}. \)

Fig. 5.46: Surface after \( N = 1 \times 10^4 \) at \( \varepsilon_t = \pm 8 \times 10^{-4} \) at 77K. Areas in above Fig.s denoted (see text). Marker = 50\( \mu \text{m}. \)

Fig. 5.43: Labyrinth type structure observed after \( N \approx 1 \times 10^4, \varepsilon_t = \pm 8 \times 10^{-4} \) at 77K.
\( B = [001]; \ g_{200}; \text{Marker } = 2.5 \mu \text{m}. \)

Fig. 5.45: Low magnification to show extent of labyrinth after \( N = 1 \times 10^4, \varepsilon_t = \pm 8 \times 10^{-4} \) at 77K.
\( B = [001]; \ g_{020}; \text{Marker } = 5 \mu \text{m}. \)

Fig. 5.47: Labyrinth type structure after \( N \approx 7.6 \times 10^3 \) at \( \varepsilon_t = \pm 1 \times 10^{-3} \) at 77K.
\( B = [001]; \ g_{030}; \text{Marker } = 0.5 \mu \text{m}. \)
Fig. 5.48: Lower magnification of substructure in area in Fig. 5.47 above.
B = [001]; g_{200}; Marker = 1 \mu m.

Fig. 5.49: Labyrinth type structure observed after N = 7.6x10^3, \varepsilon_t = \pm 1x10^{-3} at 77K.
B = [001]; g_{020}; Marker = 1 \mu m.

Fig. 5.50: Area showing a low dislocation density after N = 5x10^3, \varepsilon_t = \pm 1.4x10^{-3} at 77K.
B = [001]; g_{200}; Marker = 2 \mu m.

Fig. 5.51: Higher magnification of area in Fig. 5.50 to show details of structure.
B = [001]; g_{020}; Marker = 1 \mu m.

Fig. 5.52: Substructure after N = 5x10^3, \varepsilon_t = \pm 1.4x10^{-3} at 77K of matrix type appearance.
B = [011]; g_{111}; Marker = 2 \mu m.

Fig. 5.53: Area in Fig. 5.52 under a different operating reflection.
B = [011]; g_{111}; Marker = 2 \mu m.
Fig. 5.54: Different matrix substructure after $N = 5 \times 10^3$, $\epsilon_t = 1.4 \times 10^{-3}$ at 77K. $B = [001]$; $g_{300}$; Marker = 0.5 $\mu$m.

Fig. 5.55: Matrix type structure observed after $N = 5 \times 10^3$, $\epsilon_t = +1.4 \times 10^{-3}$ at 77 K. $B = [\bar{1}12]$; $g_{\bar{1}11}$; Marker = 2 $\mu$m.

Fig. 5.56: Different area with matrix type structure, after $N = 5 \times 10^3$, $\epsilon_t = +1.4 \times 10^{-3}$ at 77K. $B = [011]$; $g_{\bar{1}11}$; Marker = 2 $\mu$m.

Fig. 5.57: High magnification of area in Fig. 5.56 to show details of matrix structure. $B = [011]$; $g_{\bar{1}11}$; Marker = 1 $\mu$m.

Fig. 5.58: Ladder type structure after $N = 5 \times 10^3$, $\epsilon_t = 1.4 \times 10^{-3}$ at 77K. $B = [\bar{1}12]$; $g_{\bar{1}11}$; Marker = 5 $\mu$m.

Fig. 5.59: Area in Fig. 5.58 at higher magnification to show details of structure. $B = [\bar{1}12]$; $g_{\bar{1}11}$; Marker = 1 $\mu$m.
Fig. 5.60: Surface structures after $N=5 \times 10^3$ at $\varepsilon_t = +1.4 \times 10^{-3}$
Arrow denotes grain featured in Fig. 5.58. Marker = 100 \text{ \mu m}.

Fig. 5.61: Ladder type structure observed after $N = 5 \times 10^3$, $\varepsilon_t = +1.4 \times 10^{-3}$ at 77K.
$B = [011]$; $\{11\overline{1}\}$; Marker = 5 \text{ \mu m}.

Fig. 5.62: Area in Fig. 5.61 at higher magnification to show small dislocation loops.
$B = [011]$; $\{11\overline{1}\}$; Marker = 1 \text{ \mu m}.

Fig. 5.63: Example of uncondensed labyrinth after $N = 5 \times 10^3$, $\varepsilon_t = +1.4 \times 10^{-3}$ at 77K.
$B = [001]$; $\{02\overline{1}\}$; Marker = 5 \text{ \mu m}.

Fig. 5.64: Area in Fig. 5.63 at higher magnification to show dislocations in channels.
$B = [001]$; $\{02\overline{1}\}$; Marker = 1 \text{ \mu m}.

Fig. 5.65: Surface structures observed, where A denotes the area featured in Fig. 5.63.
Marker = 25 \text{ \mu m}. 
Fig. 5.66: Less well defined labyrinth structure after \( N = 5 \times 10^3, \epsilon_\text{t} = \pm 1.4 \times 10^{-3} \) at 77K. 
\( \mathbf{B} = [001]; \mathbf{g}_{220}; \text{Marker} = 2 \mu\text{m}. \)

Fig. 5.67: Area in Fig. 5.66 at higher magnification to show dislocations in channels. 
\( \mathbf{B} = [001]; \mathbf{g}_{220}; \text{Marker} = 1 \mu\text{m}. \)

Fig. 5.68: SEM of surface after \( N = 5 \times 10^3 \) at \( \epsilon_\text{t} = \pm 1.4 \times 10^{-3} \). Arrow denotes area which corresponds to Fig. 5.66.

Fig. 5.69: Less regular labyrinth structure after \( N = 5 \times 10^3, \epsilon_\text{t} = \pm 1.4 \times 10^{-3} \) at 77K. 
\( \mathbf{B} = [011]; \mathbf{g}_{211}; \text{Marker} = 1 \mu\text{m}. \)

Fig. 5.70: Area with parquet-like appearance after \( N = 5 \times 10^3, \epsilon_\text{t} = \pm 1.4 \times 10^{-3} \) at 77K. 
\( \mathbf{B} = [013]; \mathbf{g}_{220}; \text{Marker} = 5 \mu\text{m}. \)

Fig. 5.71 a: Surface structure which corresponds to area in Fig. 5.70 denoted A. 
\( \text{Marker} = 25 \mu\text{m}. \)
Fig. 5.71 b: SEM of area in Fig. 5.71 a. A corresponds to the substructure shown in Fig. 5.70.

Fig. 5.72 a: Labyrinth type structure observed after N = 5x10^3, εT = +1.4x10^{-3} at 77K. B = [001]; θ200; Marker = 1 μm.

Fig. 5.72 b: Area in Fig. 5.72 a under different operating reflection. B = [001]; θ020; Marker = 1 μm.

Fig. 5.72 c: Area in Fig. 5.72 a under different operating reflection. B = [001]; θ220; Marker = 1 μm.

Fig. 5.72 d: Area in Fig. 5.72 a under a different operating reflection. B = [001]; θ220; Marker = 1 μm.
Fig. 5.72 e: Area in Fig. 5.72 a under a different operating reflection. 
\[ B = [011]; \ 
\] Marker = 1 \( \mu \text{m.} \)

Fig. 5.72 f: Area in Fig. 5.72 a under a different operating reflection. 
\[ B = [011]; \ 
\] Marker = 1 \( \mu \text{m.} \)

Fig. 5.73: Condensed labyrinth type structure found after \( N = 5 \times 10^3 \), \( \varepsilon_t = \pm 1.4 \times 10^{-3} \) at 77K. 
\[ B = [001]; \ 
\] \( \varepsilon_{200} \); Marker = 2 \( \mu \text{m.} \)

Fig. 5.74: High magnification of area in Fig. 5.73 to show channel dislocation details. 
\[ B = [001]; \ 
\] \( \varepsilon_{020} \); Marker = 1 \( \mu \text{m.} \)

Fig. 5.75 a: Area exhibiting predominantly [100] walls after \( N=5 \times 10^3 \), \( \varepsilon_t = \pm 1.4 \times 10^{-3} \) at 77K. 
\[ B = [001]; \ 
\] \( \varepsilon_{020} \); Marker = 4 \( \mu \text{m.} \)

Fig. 5.75 b: Area in Fig. 5.75 a under different operating reflection. 
\[ B = [001]; \ 
\] \( \varepsilon_{200} \); Marker = 1 \( \mu \text{m.} \)
Fig. 5.75 c: Area in Fig. 5.75 a under a different operating reflection.

*B* = [001]; \(200\); Marker = 1 \(\mu m\).

Fig. 5.75 d: Area in Fig. 5.75 a under a different operating reflection.

*B* = [001]; \(220\); Marker = 1 \(\mu m\).

Fig. 5.75 e: Area in Fig. 5.75 a under a different operating reflection.

*B* = [001]; \(220\); Marker = 1 \(\mu m\).

Fig. 5.75 f: Area in Fig. 5.75 a under a different operating reflection.

*B* = [001]; \(111\); Marker = 1 \(\mu m\).

Fig. 5.75 g: Area in Fig. 5.75 a under a different operating reflection.

*B* = [011]; \(111\); Marker = 1 \(\mu m\).

Fig. 5.75 h: Area in Fig. 5.75 a under different operating reflection.

*B* = [011]; \(022\); Marker = 1 \(\mu m\).
Fig. 5.75 i: Weak-beam dark field image of an area in Fig. 5.75 a. \( B = [001] \); \( g = 3g \) with \( g_{200} \); Marker = 0.3 \( \mu m \).

Fig. 5.75 a. \( B = [001] \); \( g = 3g \) with \( g_{200} \); Marker = 0.3 \( \mu m \).

Fig. 5.77: Area in Fig. 5.76 at higher magnification to show details of structure. \( B = [011] \); \( g_{200} \); Marker = 1 \( \mu m \).

Fig. 5.76: Cell type regions forming in walls after \( N = 5 \times 10^3, \varepsilon_t = \mp 1.4 \times 10^{-3} \) at 77K. \( B = [011] \); \( g_{111} \); Marker = 2 \( \mu m \).

Fig. 5.78: Cluster structure at low magnification after \( N = 5 \times 10^3, \varepsilon_t = \mp 1.4 \times 10^{-3} \) at 77K. \( B = [001] \); \( g_{200} \); Marker = 2 \( \mu m \).

Fig. 5.79 a: Area in Fig. 5.78 at higher magnification to show cluster details. \( B = [001] \); \( g_{020} \); Marker = 0.5 \( \mu m \).

Fig. 5.79 a: Area in Fig. 5.78 at higher magnification to show cluster details. \( B = [001] \); \( g_{020} \); Marker = 0.5 \( \mu m \).

Fig. 5.79 b: Area in Fig. 5.79 a under a different operating reflection. \( B = [001] \); \( g_{200} \); Marker = 0.5 \( \mu m \).
Fig. 5.80: Area in Fig. 5.78 at very low magnification to show extent of cluster region.

B = [001]; Marker = 10 µm.

Fig. 5.81: Low magnification optical micrograph of specimen surface after 5x10³ cycles, εₜ = ±1.4x10⁻³ at 77K. A denotes labyrinth type area corresponding to Fig. 5.73 and 5.74; B Marks area where cluster type features (Fig. 5.80) observed. Marker = 50 µm.
5.81 illustrated at higher magnification.
Marker = 25 μm.

Fig. 5.82: Area B in Fig. 5.81 illustrated at higher magnification.
Marker = 25 μm.

Fig. 5.83: Area corresponding to cluster structure shown in Fig. 5.80.
Marker = 20 μm.

Fig. 5.84: SEM of surface after
N = 5x10^3 at \( \varepsilon_t = \pm 1.4 \times 10^{-3} \).
Area corresponds to that in Fig. 5.82.

Fig. 5.85: SEM stereo-pair of structure in a region of Fig. 5.84 which has been found to correspond to cluster type dislocation structures.
Fig. 5.86: Formation of cluster and labyrinth structures within one grain after $N = 5 \times 10^3$, $\varepsilon_t = +1.4 \times 10^{-3}$ at 77K.

$B = [001]$; $\theta_{020}$; Marker = 5µm.

Fig. 5.87: Labyrinth area in Fig. 5.86 at higher magnification.

$B = [001]$; $\theta_{020}$; Marker = 1µm.

Fig. 5.88: Region in Fig. 5.86 showing areas of dislocation tangles.

$B = [001]$; $\theta_{020}$; Marker = 1µm.
Fig. 5.89: Area in Fig. 5.86 in which cluster structures appear to be forming.

\( B = [001]; \, g_{200}; \, \text{Marker} = 1 \, \mu m. \)

Fig. 5.90: Another example of clusters forming after \( N = 5 \times 10^3, \, \epsilon_t = +1.4 \times 10^{-3} \) at 77K.

\( B = [001]; \, g_{200}; \, \text{Marker} = 1 \, \mu m. \)

Fig. 5.91: Stereo-pair of cluster structure forming from tangles after \( 5 \times 10^3 \) cycles, \( \epsilon_t = +1.4 \times 10^{-3} \) at 77K. \( B = [001]; \, g_{200}; \, \text{Marker} = 0.5 \, \mu m. \)
Fig. 5.92: Another example of cluster formation after $N = 5 \times 10^3$, $\varepsilon_t = \pm 1.4 \times 10^{-3}$ at 77K.

$B = [011]$; $g_{200}$; Marker = 5 µm.

Fig. 5.93: Area in Fig. 5.92 at higher magnification to show cluster details.

$B = [001]$; $g_{200}$; Marker = 1 µm.

Fig. 5.94: Example of dislocation tangles and wall formation near the cluster region in Fig. 5.92.

$B = [001]$; $g_{020}$; Marker = 2 µm.

Fig. 5.95: Optical micrograph of surface structure after $N = 5 \times 10^3$, $\varepsilon_t = \pm 1.4 \times 10^{-3}$ at 77K. A corresponds to cluster features in Fig. 5.92; B corresponds to matrix (Fig. 5.56). Marker = 100 µm.
Fig. 5.96: Area in Fig. 5.95 at high magnification to show details of regions A and B. Marker = 25 \( \mu \text{m} \).

Fig. 5.98: Area which has been found to correlate with cluster features in Fig. 5.92 at higher magnification.

Fig. 5.99: Area showing regions with dark contrast after \( N = 5 \times 10^3 \), \( \epsilon_t = +1.4 \times 10^{-3} \) at 77K. \( \mathbf{B} = [013] \); \( \mathbf{g}_{\overline{2}00} \); Marker = 3 \( \mu \text{m} \).

Fig. 5.100: Area in Fig. 5.99 at higher magnification, to show dislocation tangles. \( \mathbf{B} = [011] \); \( \mathbf{g}_{\overline{1}11} \); Marker = 1 \( \mu \text{m} \).

Fig. 5.101: Surface structure corresponding to the region shown in Fig. 5.100 marked A. Marker = 25 \( \mu \text{m} \).
Fig. 5.102 SEM of "cross-hatched" structure developed after $N = 5 \times 10^3$ at $\epsilon_t = \pm 1.4 \times 10^{-3}$ at 77 K.

Fig. 5.103 SEM of "rumpled bands" developed after $N = 5 \times 10^3$ at $\epsilon_t = \pm 1.4 \times 10^{-3}$ at 77 K.
Fig. 6.1 a: Optical micrograph of area near C7.3SU after N = 1.6 x 10^3 cycles, W_1.
Marker = 30 μm.

Fig. 6.1 b: Optical micrograph of PSBs developed near C7.3SU after N = 6.5 x 10^3 cycles, W_2.
Marker = 30 μm.

Fig. 6.1 c: Optical micrograph of C7.3SU at lower magnification after N = 6.5 x 10^3 cycles, W_2.
Marker = 150 μm.

Fig. 6.2 a: Optical micrograph of area near C7.6SU after N = 1.6 x 10^3 cycles, W_1.
Marker = 30 μm.

Fig. 6.2 b: Optical micrograph of PSBs developed near C7.6SU after N = 6.5 x 10^3 cycles, W_2.
Marker = 30 μm.

Fig. 6.2 c: Optical micrograph of C7.6SU at lower magnification after N = 6.5 x 10^3 cycles, W_2.
Marker = 150 μm.
Fig. 6.3 a: SEM micrograph of PSBs developed near C7.3SU after $N = 6.5 \times 10^3$ cycles, $W_2$.

Fig. 6.3 b: SEM micrograph of PSBs developed near C7.3SU after $N = 6.5 \times 10^3$ cycles, $W_2$. Crystal tilted to a higher angle to show PSB details.

Fig. 6.4 a: SEM micrograph of PSBs developed near C7.6SU after $N = 6.5 \times 10^3$ cycles, $W_2$.

Fig. 6.4 b: SEM micrograph of PSBs developed near C7.6SU after $N = 6.5 \times 10^3$ cycles, $W_2$. Crystal tilted to a higher angle to show PSB details.
Fig. 6.5 a: Optical micrograph of PSBs developed near C10.2SU after $N = 1.05 \times 10^4$ cycles, $W_2$. Marker = 150 $\mu$m.

Fig. 6.5 b: Optical micrograph of PSBs developed near C10.2SU after $N = 1.25 \times 10^4$ cycles, $W_2$. Marker = 150 $\mu$m.

Fig. 6.5 c: Optical micrograph of C10.2SU at higher magnification after $N = 1.25 \times 10^4$ cycles, $W_2$. Marker = 30 $\mu$m.

Fig. 6.6 a: Optical micrograph of area near C10.3SA after $N = 1.05 \times 10^4$ cycles, $W_2$. Marker = 150 $\mu$m.

Fig. 6.6 b: Optical micrograph of PSBs developed near C10.3SA after $N = 1.25 \times 10^4$ cycles, $W_2$. Marker = 150 $\mu$m.

Fig. 6.6 c: Optical micrograph, at higher magnification, of C10.3SA after $N = 1.25 \times 10^4$ cycles, $W_2$. Marker = 30 $\mu$m.
Fig. 6.7: SEM micrograph of PSBs developed near C10.2SU after $N = 1.25 \times 10^4$ cycles, $W_2$.

Fig. 6.8: SEM micrograph of PSBs developed near C10.3SA after $N = 1.25 \times 10^4$ cycles, $W_2$.

Fig. 6.9 a: Optical micrograph of PSBs developed near C11.5SU after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.9 b: Optical micrograph of PSBs developed near C11.7DU after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.9 c: Optical micrograph of PSBs developed near C11.4SA after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.9 d: Optical micrograph of PSBs developed near C11.6DA after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.
Fig. 6.10 a: Optical micrograph, at lower magnification, of PSBs developed near C11.5SU after $N = 2 \times 10^3$, $W_1$. Marker = 75 µm.

Fig. 6.11: Optical micrograph of area near C11.10SU after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 30 µm.

Fig. 6.12: Optical micrograph at lower magnification of area near C11.7DU after $N = 2 \times 10^3$, $W_1$. Marker = 60 µm.

Fig. 6.13 a: SEM micrograph of area near C11.1SU after $N = 2 \times 10^3$ cycles, $W_1$.

Fig. 6.13 b: SEM micrograph of area near C11.10SU after $N = 2 \times 10^3$ cycles, $W_1$. 
Fig. 6.14 a: Optical micrograph showing slip bands near C6.11SU after indenting.
Marker = 30µm.

Fig. 6.14 b: Optical micrograph showing slip bands near C6.12SU after indenting.
Marker = 30µm.

Fig. 6.15 a: SEM micrograph showing slip bands near C6.11SU after indenting.

Fig. 6.15 b: SEM micrograph showing slip bands near C6.12SU after indenting.
Fig. 6.16 a: Optical micrograph of area near C6.5SU after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.17 a: Optical micrograph of area near C6.11SU after $N = 2 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.16 b: Optical micrograph of PSBs developed near C6.5SU after $N = 4 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.17 b: Optical micrograph of area near C6.11SU after $N = 4 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.16 c: Optical micrograph of PSBs developed near C6.5SU after $N = 8 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.17 c: Optical micrograph of area near C6.11SU after $N = 8 \times 10^3$ cycles, $W_1$. Marker = 50 $\mu$m.
Fig. 6.18 a: Optical micrograph of area near C6.4SA after $N = 2 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.18 b: Optical micrograph of PSBs developed near C6.4SA after $N = 4 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.18 c: Optical micrograph of PSBs developed near C6.4SA after $N = 8 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.19 a: Optical micrograph of area near C6.6SA after $N = 2 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.19 b: Optical micrograph of PSBs developed near C6.6SA after $N = 4 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.19 c: Optical micrograph of PSBs developed near C6.6SA after $N = 8 \times 10^3$ cycles, $W_1$.
Marker = 50 $\mu$m.
Fig. 6.20 a: SEM micrograph of area near C6.5SU after $N = 2 \times 10^3$ cycles, $W_1$.

Fig. 6.20 b: SEM micrograph of PSBs developed near C6.5SU after $N = 6 \times 10^3$ cycles, $W_1$.

Fig. 6.21 a: SEM micrograph of area near C6.11SU after $N = 2 \times 10^3$ cycles, $W_1$.

Fig. 6.21 b: SEM micrograph of area near C6.11SU after $N = 6 \times 10^3$ cycles, $W_1$.

Fig. 6.22 a: SEM micrograph of area near C6.45A after $N = 2 \times 10^3$ cycles, $W_1$.

Fig. 6.22 b: SEM micrograph of PSBs developed near C6.45A after $N = 6 \times 10^3$ cycles, $W_1$. 
Fig. 6.23 a: SEM micrograph of area near C6.6SA after $N = 2 \times 10^3$ cycles, $W_1$.

Fig. 6.23 b: SEM micrograph of PSBs developed near C6.6SA after $N = 6 \times 10^3$ cycles, $W_1$.

Fig. 6.24 a: Optical micrograph showing slip bands near C3.1SA after indenting.
Marker = 30 $\mu$m.

Fig. 6.24 b: Optical micrograph showing slip bands near C3.5SA after indenting.
Marker = 30 $\mu$m.

Fig. 6.24 c: Optical micrograph showing slip bands near C3.3DA after indenting.
Marker = 30 $\mu$m.

Fig. 6.24 d: Optical micrograph showing slip bands near C3.7DA after indenting.
Marker = 30 $\mu$m.
Fig. 6.25: Optical micrograph of PSBs developed at narrow end of C3 after \( N = 2 \times 10^4 \), \( W_0 \). Marker = 50 µm.

Fig. 6.26: Optical micrograph of PSBs developed on two slip planes near edge \( t_1 \) of C3 after \( N = 7 \times 10^3 \), \( W_1 \). Marker = 50 µm.

Fig. 6.27: Optical micrograph of PSBs developed on C3 at \( l = 12.7 \text{ mm}, \Phi = 0.5^\circ \) after \( N = 8 \times 10^4 \), cycles, \( W_1 \). Marker = 50 µm.

Fig. 6.28: Optical micrograph of C3 at \( l = 31 \text{ mm}, \Phi = 1.2^\circ \), showing PSBs on two slip planes. \( N = 8 \times 10^4 \), \( W_1 \). Marker = 50 µm.

Fig. 6.29 a: SEM micrograph of PSBs developed near edge \( t_1 \) at wide end of C3 after \( N = 8 \times 10^4 \), \( W_1 \).

Fig. 6.29 b: SEM micrograph of part of Fig. 6.29 a at higher magnification to show PSB details.
Fig. 6.30 a: Optical micrograph of PSBs developed near C3.2SU after $N = 2.2 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.30 b: Optical micrograph of PSBs developed near C3.2SU after $N = 4.2 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.30 c: Optical micrograph of PSBs developed near C3.2SU after $N = 8 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.31 a: Optical micrograph of PSBs developed near C3.6SU after $N = 2.2 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.31 b: Optical micrograph of PSBs developed near C3.6SU after $N = 4.2 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.

Fig. 6.31 c: Optical micrograph of PSBs developed near C3.6SU after $N = 8 \times 10^4$ cycles, $W_1$.
Marker = 50 $\mu$m.
Fig. 6.32 a: Optical micrograph of PSBs developed near C3.1SA after N = 2.2 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).

Fig. 6.32 b: Optical micrograph of PSBs developed near C3.1SA after N = 4.2 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).

Fig. 6.32 c: Optical micrograph of PSBs developed near C3.1SA after N = 8 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).

Fig. 6.33 a: Optical micrograph of PSBs developed near C3.5SA after N = 2.2 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).

Fig. 6.33 b: Optical micrograph of PSBs developed near C3.5SA after N = 4.2 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).

Fig. 6.33 c: Optical micrograph of PSBs developed near C3.5SA after N = 8 x 10^4 cycles, \( W_1 \). Marker = 50 \( \mu m \).
Fig. 6.34 a: Optical micrograph of PSBs developed near C3.4DU after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 50 µm.

Fig. 6.34 b: Optical micrograph of PSBs developed near C3.4DU after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 50 µm.

Fig. 6.34 c: Optical micrograph of PSBs developed near C3.4DU after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 50 µm.

Fig. 6.35 a: Optical micrograph of PSBs developed near C3.8DU after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 50 µm.

Fig. 6.35 b: Optical micrograph of PSBs developed near C3.8DU after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 50 µm.

Fig. 6.35 c: Optical micrograph of PSBs developed near C3.8DU after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 50 µm.
Fig. 6.36 a: Optical micrograph of PSBs developed near C3.3DA after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.36 b: Optical micrograph of PSBs developed near C3.3DA after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.36 c: Optical micrograph of PSBs developed near C3.3DA after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.37 a: Optical micrograph of area near C3.7DA after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.37 b: Optical micrograph of PSBs developed near C3.7DA after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.

Fig. 6.37 c: Optical micrograph of PSBs developed near C3.7DA after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 50 $\mu$m.
Fig. 6.38 a: Optical micrograph of area near C3.6SU after \( N = 3 \times 10^3 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).

Fig. 6.38 b: Optical micrograph of area near C3.6SU after \( N = 2.2 \times 10^4 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).

Fig. 6.38 c: Optical micrograph of PSBs developed near C3.6SU after \( N = 4.2 \times 10^4 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).

Fig. 6.39 a: Optical micrograph of PSBs developed near C3.5SA after \( N = 3 \times 10^3 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).

Fig. 6.39 b: Optical micrograph of PSBs developed near C3.5SA after \( N = 2.2 \times 10^4 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).

Fig. 6.39 c: Optical micrograph of PSBs developed near C3.5SA after \( N = 4.2 \times 10^4 \) cycles, \( W_1 \).
Marker = 30 \( \mu m \).
Fig. 6.38 d: Optical micrograph of PSBs developed near C3.6SU after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.39 d: Optical micrograph of PSBs developed near C3.5SA after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.40 a: Optical micrograph of PSBs developed near C3.4DU after $N = 3 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.41 a: Optical micrograph of area near C3.3DA after $N = 3 \times 10^3$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.40 b: Optical micrograph of PSBs developed near C3.4DU after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.41 b: Optical micrograph of PSBs developed near C3.3DA after $N = 2.2 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.
Fig. 6.40 c: Optical micrograph of PSBs developed near C3.4DU after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.41 c: Optical micrograph of PSBs developed near C3.3DA after $N = 4.2 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.40 d: Optical micrograph of PSBs developed near C3.4DU after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.

Fig. 6.41 d: Optical micrograph of PSBs developed near C3.3DA after $N = 8 \times 10^4$ cycles, $W_1$. Marker = 30 $\mu$m.
Fig. 6.42 a-h: SEM micrographs showing details of PSBs developed near unannealed and annealed indentations in C3.
Fig. 6.43: SEM micrograph to show details of the PSBs developed near C3.2SU after $N = 8 \times 10^4$ cycles, $W_1$.

Fig. 6.44a: Optical micrograph of PSBs developed near an etch pit at $l = 12.7 \text{mm}$, $\Phi = 12.8^\circ$ after $N = 8 \times 10^4$, $W_1$. Marker $= 30 \mu\text{m}$.

Fig. 6.44b: SEM micrograph of PSBs developed near the etch pit above after $N = 8 \times 10^4$, $W_1$. A PSB is apparent within the pit.
Fig. 7.1: Optical micrograph of unannealed indentation in F71 to show areas examined by TEM. Marker = 10 μm.

Fig. 7.2: Dislocation structure observed in region A within the indentation in Fig. 7.1. B = [011]; g200; Marker = 0.4μm.

Fig. 7.3: Dislocation structure observed in region B within the indentation in Fig. 7.1. B = [011]; gIII; Marker = 0.5μm.

Fig. 7.4: Dislocation structure observed in region C within the indentation in Fig. 7.1. B = [011]; gIII; Marker = 0.3μm.
Fig. 7.5: Optical micrograph of unannealed indentation in F_{13}1 show areas examined by TEM.
Marker = 20 \mu m.

Fig. 7.6: Dislocation structure observed in region A within the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{020} \); Marker = 0.2\mu m.

Fig. 7.7: Dislocation structure observed in region B within the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{020} \); Marker = 0.25\mu m.

Fig. 7.8: Dislocation structure observed in region C within the indentation in Fig. 7.5.
B = [013]; \( \varepsilon_{151} \); Marker = 0.2\mu m.

Fig. 7.9: Dislocation structure observed in region D within the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{020} \); Marker = 0.25\mu m.
Fig. 7.10: Dislocation structure observed in region E within the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{[020]} \); Marker = 0.2 \( \mu \)m.

Fig. 7.11: Dislocation structure observed in region F within the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{[200]} \); Marker = 0.2 \( \mu \)m.

Fig. 7.12: Dislocation structure observed in region G within the indentation in Fig. 7.5.
B = [011]; \( \varepsilon_{[111]} \); Marker = 0.4 \( \mu \)m.

Fig. 7.13: Dislocation structure observed in region H near the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{[020]} \); Marker = 1 \( \mu \)m.

Fig. 7.14: Another example of microstructure in region H near the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{[020]} \); Marker = 0.5 \( \mu \)m.

Fig. 7.15: Dislocation structure observed in region I near the indentation in Fig. 7.5.
B = [001]; \( \varepsilon_{[020]} \); Marker = 1 \( \mu \)m.
Fig. 7.16 a: Schematic diagram to show the positions of TEM areas at 3.3d (A), 2.3d (B), 1.2d (C) and 0.5d (D) in the grain containing the unannealed indentation in F133. E corresponds to the area examined in an adjoining grain, close to the grain boundary.

Fig. 7.16 b: Optical micrograph of the unannealed indentation in F133 shown schematically in Fig. 7.16 a. Marker = 20 μm.

Fig. 7.17: Dislocation structure observed at position A shown in Fig. 7.16 a above. B = [001]; g = 020; Marker = 1 μm.
Fig. 7.18: Dislocation structure observed at position B shown in Fig. 7.16 a above. 
\( \mathbf{B} = [001]; \mathbf{g} = 020; \) Marker = 1 \( \mu m \).

Fig. 7.19: Dislocation structure observed at position C shown in Fig. 7.16 a above. 
\( \mathbf{B} = [001]; \mathbf{g} = 020; \) Marker = 1 \( \mu m \).

Fig. 7.20: Dislocation structure observed at the indentation edge denoted D in Fig. 7.16 a above. 
\( \mathbf{B} = [013]; \mathbf{g} = 200; \) Marker = 0.5 \( \mu m \).

Fig. 7.21: Dislocation structure in grain adjoining the indented grain, denoted E in Fig. 7.16 a. 
\( \mathbf{B} = [001]; \mathbf{g} = 200; \) Marker = 1 \( \mu m \).
Fig. 7.22: Optical micrograph of unannealed indentation in \( F_{266} \) to show areas examined by TEM. Marker = 20 \( \mu \text{m} \).

Fig. 7.23: Dislocation structure observed near the indentation edge denoted A in Fig. 7.22.
\( B = [001] \); \( g_{200} \); Marker = 0.4\( \mu \text{m} \).

Fig. 7.24 a: Microstructure observed in region B at edge of indentation shown in Fig. 7.22.
\( B = [001] \); \( g_{020} \); Marker = 0.5\( \mu \text{m} \).
Fig. 7.24 b: Area shown in Fig. 7.24 a at higher magnification to show microstructure details. $B = [001]; \xi_{200}; Marker = 0.4\mu m.$

Fig. 7.25 a: Microstructure observed in region C at edge of indentation shown in Fig. 7.22. $B = [013]; \xi_{200}; Marker = 0.5\mu m.$

Fig. 7.25 b: Area in Fig. 7.25a above under a different operating reflection. $B = [001]; \xi_{200}; Marker = 0.5\mu m.$

Fig. 7.25 c: Area in Fig. 7.25 a above under a different operating reflection. $B = [013]; \xi_{200}; Marker = 0.5\mu m.$

Fig. 7.25 d: Area in Fig. 7.25 a above under a different operating reflection. $B = [013]; \xi_{131}; Marker = 0.5\mu m.$

Fig. 7.25 e: Area in Fig. 7.25 a above under a different operating reflection. $B = [013]; \xi_{131}; Marker = 0.5\mu m.$
Fig. 7.26 a: Microstructure observed in region D within the indentation shown in Fig. 7.22. $B = [001]; g_{200}; Marker = 0.5\mu m$.

Fig. 7.26 b: Area in Fig. 7.26 a at higher magnification to show microstructure details. $B = [001]; g_{200}; Marker = 0.3\mu m$.

Fig. 7.26 c: Microstructure in area adjoining region D shown in Fig. 7.26 a above. $B = [001]; g_{200}; Marker = 0.5\mu m$.

Fig. 7.26 d: Weak beam dark field image ($g, 3g$) of part of the area shown in Fig. 7.26 c. $B = [001]; g_{200}; Marker = 0.25\mu m$.

Fig. 7.25 f: Area in Fig. 7.25 a above under a different operating reflection. $B = [\bar{1}4]; g_{\bar{3}1\bar{1}}; Marker = 0.5\mu m$. 
Fig. 7.27: Optical micrograph of unannealed indentation in single crystal material showing area examined by TEM. Marker = 20 μm.

Fig. 7.28 a: Microstructure observed near the indentation edge denoted A in Fig. 7.27. $B = [011]$; $g_{111}$; Marker = 0.4 μm.

Fig. 7.28 b: Weak beam dark field image ($g$, 3$g$) of area in Fig. 7.28 a above. $B = [011]$; $g_{111}$; Marker = 0.4 μm.
Fig. 7.29: Optical micrograph of annealed indentation in F274 to show areas examined by TEM. Marker = 20\(\mu\)m.

Fig. 7.30: Low magnification TEM micrograph showing areas denoted in 7.29 around the indentation perforation. Marker = 2\(\mu\)m.

Fig. 7.31 a: Microstructure observed in region A within the indentation in Fig. 7.29.
\[ B = [001]; \ \vec{g}_{020}; \ \text{Marker} = 0.5\mu\text{m}. \]

Fig. 7.31 b: Area in Fig. 7.31 a under a different operating reflection.
\[ B = [001]; \ \vec{g}_{200}; \ \text{Marker} = 0.5\mu\text{m}. \]

Fig. 7.31 c: Area in Fig. 7.31 a at higher magnification to show microstructure details.
\[ B = [001]; \ \vec{g}_{020}; \ \text{Marker} = 0.3\mu\text{m}. \]

Fig. 7.32: Microstructure observed in region B within the indentation shown in Fig. 7.29.
\[ B = [\bar{1}12]; \ \vec{g}_{1\bar{1}1}; \ \text{Marker} = 0.5\mu\text{m}. \]
Fig. 7.33 a: Microstructure observed in region C within the indentation shown in Fig. 7.29. \( B = [\bar{1}14] \); \( g_{220} \); Marker = 0.5\( \mu \)m.

Fig. 7.33 b: Area in Fig. 7.33 a above under a different operating reflection. \( B = [\bar{1}14] \); \( g_{3\bar{1}1} \); Marker = 0.5\( \mu \)m.

Fig. 7.33 c: Area in Fig. 7.33 a above under a different operating reflection. \( B = [\bar{1}12] \); \( g_{1\bar{1}1} \); Marker = 0.5\( \mu \)m.
Fig. 7.34: Schematic diagram of annealed indentation in F$_{272}$ to show areas examined by TEM. Marker = 10 μm.

Fig. 7.35 a: Microstructure observed in region A within indentation shown in Fig. 7.34. B = [001]; g = 200; Marker = 0.5 μm.

Fig. 7.35 b: Area in Fig. 7.35 a above under a different operating reflection. B = [114]; g = 220; Marker = 0.5 μm.
Fig. 7.35 c: Area shown in Fig. 7.35 a under a different operating reflection. 
$B = [011]_z$; $g_{111}$; Marker = 0.5μm.

Fig. 7.35 d: Area shown in Fig. 7.35 a under a different operating reflection. 
$B = [011]_z$; $g_{111}$; Marker = 0.5μm.

Fig. 7.35 e: Area shown in Fig. 7.35 a above under a different operating reflection. 
$B = [\bar{1}12]; g_{\bar{1}13}$; Marker = 0.5μm.

Fig. 7.35 f: Area shown in Fig. 7.35 a above under a different operating reflection. 
$B = [\bar{1}14]; g_{1\bar{3}1};$ Marker = 0.5μm.

Fig. 7.35 g: Area shown in Fig. 7.35 a above under a different operating reflection. 
$B = [\bar{1}12]; g_{311};$ Marker = 0.5μm.

Fig. 7.35 h: Area shown in Fig. 7.35 a above under a different operating reflection. 
$B = [011]; g_{31\bar{1}};$ Marker = 0.5μm.
Fig. 7.36 a: Microstructure observed in region B within indentation shown in Fig. 7.34. 
B = [011]; $g_{200}$; Marker = 0.5μm.

Fig. 7.36 b: Area shown in Fig. 7.36 a under a different operating reflection. 
B = [011]; $g_{022}$; Marker = 0.5μm.

Fig. 7.36 c: Area shown in Fig. 7.36 a above under a different operating reflection. 
B = [112]; $g_{131}$; Marker = 0.5μm.

Fig. 7.36 d: Area shown in Fig. 7.36 a above under a different operating reflection. 
B = [011]; $g_{311}$; Marker = 0.5μm.

Fig. 7.37 a: Microstructure observed in region C within indentation shown in Fig. 7.34. 
B = [011]; $g_{200}$; Marker = 0.5μm.

Fig. 7.37 b: Area shown in Fig. 7.37 a under a different operating reflection. 
B = [112]; $g_{220}$; Marker = 0.5μm.
Fig. 7.37 c: Dark field image 
\((g, -g)\) of area shown in Fig.
7.37 b.
\(B = [\bar{1}12]; \ g_{220}; \ \text{Marker} = 0.5\mu m.\)

Fig. 7.37 e: Area shown in Fig.
7.37 a above under a different 
operating reflection.
\(B = [\bar{1}12]; \ g_{111}; \ \text{Marker} = 0.5\mu m.\)

Fig. 7.37 g: Dark field image 
\((g, -g)\) of area shown in Fig.
7.37 f.
\(B = [\bar{1}12]; \ g_{1\bar{3}1}; \ \text{Marker} = 0.5\mu m.\)

Fig. 7.37 d: Area shown in Fig.
7.37 a under a different 
operating reflection.
\(B = [011]; \ g_{11\bar{1}}; \ \text{Marker} = 0.5\mu m.\)

Fig. 7.37 f: Area shown in Fig.
7.37 a above under a different 
operating reflection.
\(B = [\bar{1}12]; \ g_{1\bar{3}1}; \ \text{Marker} = 0.5\mu m.\)

Fig. 7.37 h: Area shown in Fig.
7.37 a above under a different 
operating reflection.
\(B = [\bar{1}12]; \ g_{311}; \ \text{Marker} = 0.5\mu m.\)
Fig. 7.38: Schematic diagram of TEM areas near unannealed (I1) and annealed (I2) indentations within a single grain in F2711. Marker = 10 μm.

Fig. 7.39 a: Microstructure in region A near the unannealed indentation in Fig. 7.38. B = [013]; g200; Marker = 1 μm.

Fig. 7.39 b: Microstructure in region B near the unannealed indentation in Fig. 7.38. B = [001]; g200; Marker = 1 μm.

Fig. 7.40 a: Microstructure in region A near the annealed indentation in Fig. 7.38. B = [001]; g200; Marker = 1 μm.

Fig. 7.40 b: Microstructure in region B near the annealed indentation in Fig. 7.38. B = [001]; g200; Marker = 1 μm.
Fig. 7.39 c: Microstructure in region C near the unannealed indentation in Fig. 7.38. 
\( B = [001] \); \( g_{200} \); Marker = 1 µm.

Fig. 7.39 d: Microstructure in region D near the unannealed indentation in Fig. 7.38. 
\( B = [001] \); \( g_{200} \); Marker = 1 µm.

Fig. 7.40 c: Microstructure in region C near the annealed indentation in Fig. 7.38. 
\( B = [001] \); \( g_{200} \); Marker = 1 µm.

Fig. 7.40 d: Microstructure in region D near the annealed indentation in Fig. 7.38. 
\( B = [001] \); \( g_{200} \); Marker = 1 µm.

Fig. 7.41 a: Microstructure in region A/B near unannealed indentation shown in Fig. 7.38. 
\( B = [011] \); \( g_{111} \); Marker = 0.5 µm.

Fig. 7.42 a: Microstructure in region A/B near annealed indentation shown in Fig. 7.38. 
\( B = [011] \); \( g_{200} \); Marker = 0.5 µm.
Fig. 7.41 b: Microstructure in region B/C near unannealed indentation shown in Fig. 7.38.
\( B = [011]; \ g_{200}; \ \text{Marker} = 0.25\mu m. \)

Fig. 7.41 c: Microstructure in region C/D near unannealed indentation shown in Fig. 7.38.
\( B = [001]; \ g_{200}; \ \text{Marker} = 0.5\mu m. \)

Fig. 7.41 d: Microstructure in region D/A near unannealed indentation shown in Fig. 7.38.
\( B = [011]; \ g_{200}; \ \text{Marker} = 0.5\mu m. \)

Fig. 7.42 b: Microstructure in region B/C near annealed indentation shown in Fig. 7.38.
\( B = [011]; \ g_{111}; \ \text{Marker} = 0.25\mu m. \)

Fig. 7.42 c: Microstructure in region C/D near annealed indentation shown in Fig. 7.38.
\( B = [011]; \ g_{111}; \ \text{Marker} = 0.5\mu m. \)

Fig. 7.42 d: Microstructure in region D/A near annealed indentation shown in Fig. 7.38.
\( B = [011]; \ g_{111}; \ \text{Marker} = 0.5\mu m. \)
**Fig. 7.43 a:** Optical micrograph of an unannealed indentation in Fe₄fatigued for \( N = 5 \times 10^3 \) at \( \varepsilon_t = \pm 6 \times 10^{-4} \). Marker = 100 \( \mu m \).

**Fig. 7.43 b:** Indentation in Fig. 7.43 a at higher magnification with area examined by TEM denoted A. Marker = 30 \( \mu m \).

**Fig. 7.44 a:** Microstructure in region A within the fatigued indentation shown in Fig. 7.43a. \( B = [001] \); \( \xi_{220} \); Marker = 0.5 \( \mu m \).

**Fig. 7.44 b:** Area shown in Fig. 7.44 a above under a different operating reflection. \( B = [001] \); \( \xi_{220} \); Marker = 0.5 \( \mu m \).

**Fig. 7.44 c:** Area shown in Fig. 7.44 a above under a different operating reflection. \( B = [001] \); \( \xi_{220} \); Marker = 0.5 \( \mu m \).
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APPENDIX A

SCHMID FACTOR CALCULATIONS

If the cyclic stress axis of a crystal makes an angle $\psi$ with the normal to the slip plane, and the angle between the axis and the slip direction is $\lambda$ (see Fig. A.1) then the resolved shear stress amplitude $\tau$ is given by,

$$\tau = s \cos \psi \cos \lambda$$

where $s$ is the cyclic stress amplitude.

The factor $\cos \psi \cos \lambda$ which is used for converting the cyclic stress amplitude to the resolved shear stress amplitude is called the Schmid factor. In a crystal of f.c.c. structure there are twelve possible slip systems, each of which is a $\{111\}$ plane combined with a $<110>$ direction that lies in it. When a stress is applied to a crystal, the Schmid factor will in general be different for the various slip systems. The values depend on the orientation of the crystal in relation to the cyclic stress axis.

The Schmid factors for polycrystalline specimens for which the direction of the tension-compression axis has been determined (see Figs. 5.11 and 5.17) are given in Table A.1.
### Table A.1: Schmid Factor Determination for Polycrystalline Specimens

<table>
<thead>
<tr>
<th>Slip Plane</th>
<th>Slip Direction</th>
<th>Schmid Factors for Tension-compression axis in Fig. 5.11</th>
<th>Schmid Factors for Tension-compression axis in Fig. 5.17</th>
</tr>
</thead>
<tbody>
<tr>
<td>(111)</td>
<td>[011]</td>
<td>0.25</td>
<td>0.29</td>
</tr>
<tr>
<td>(111)</td>
<td>[101]</td>
<td>0.49</td>
<td>0.07</td>
</tr>
<tr>
<td>(111)</td>
<td>[110]</td>
<td>0.25</td>
<td>0.36</td>
</tr>
<tr>
<td>(111)</td>
<td>[011]</td>
<td>0.08</td>
<td>0.48</td>
</tr>
<tr>
<td>(111)</td>
<td>[101]</td>
<td>0.16</td>
<td>0.12</td>
</tr>
<tr>
<td>(111)</td>
<td>[110]</td>
<td>0.25</td>
<td>0.36</td>
</tr>
<tr>
<td>(111)</td>
<td>[011]</td>
<td>0.08</td>
<td>0.48</td>
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<tr>
<td>(111)</td>
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<tr>
<td>(111)</td>
<td>[110]</td>
<td>0.25</td>
<td>0.36</td>
</tr>
</tbody>
</table>
Fig. A.1: Resolved shear stress on a slip system.
CONDENSED DISLOCATION STRUCTURES IN POLYCRYSTALLINE ALUMINIUM FATIGUED AT 77K.

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(Received November 1, 1986)

Introduction
The configurations of dislocations in fatigued metal have been studied in detail in recent years. Most of the published work is on face-centred cubic metals, particularly Cu and Ni fatigued at room temperature. This work has been reviewed in several papers by Mughrabi (1, 2, 3) and most recently by Laird, Charsley and Mughrabi in the context of low energy dislocation structures (4). A significant feature of this work is the formation of a matrix structure at low fatigue amplitudes which is replaced, either wholly or partially, by a more compact or 'condensed' dislocation structure at larger values of cumulative plastic strain. The condensed structures are typical of stress saturation. Such structures, in crystals of easy glide orientation, have a direct relationship to the persistent slip bands (PSB's) which are seen on the surface. They are seen as 'ladder structures' formed from primary dislocation dipoles which are stacked in walls perpendicular to their Burgers vectors. In multiple slip orientations a common feature is the formation of walls on 2 sets of (100) planes - the so-called labyrinth structure.

In some recent work on Al, fatigued at room temperature, we have been unable to observe any features similar to those found in Cu even when the total strain amplitude is as low as \( \varepsilon = \pm 4 \times 10^{-4} \). The literature is also devoid of reports of such structures in the case of Al, and for this reason we have investigated Al fatigued at 77K. We are unable to maintain the specimen temperature at this low value during the preparation of thin foils for transmission electron microscopy (TEM), but we have made every attempt to maintain as low a temperature as possible at all stages. We have observed structures similar to all of those outlined above together with a type of dislocation structure which has not been reported before in any fatigued metal.

Experimental Techniques
The surfaces of 90 \( \mu \)m thick, 99.99\% purity polycrystalline Al specimens were prepared by electro-polishing using a 35% nitric acid/methanol solution, held at \( \sim -10^\circ \)C. Discs of 3 mm diameter were cut from this material by spark erosion, and were annealed for 1 h at 425\(^\circ\)C giving a grain diameter of \( \sim 130 \mu \)m. The Al specimens were then fatigued using a technique previously developed in which foils are glued to a thick Al alloy beam, which is cycled in reverse-bending (5) so that the foil on the surface undergoes fatigue which approximates closely to push-pull. Total strain amplitudes in the range \( \pm 4 \times 10^{-4} \) to \( \pm 1.4 \times 10^{-3} \) were used, and specimens were maintained at 77K throughout fatigue by directing a jet of liquid nitrogen on to them. Acetone cooled with liquid nitrogen to a temperature of \( \sim -50^\circ \)C was used to dissolve the glue in order to remove the specimens from the beam. The discs were then prepared for TEM by jet thinning using the electropolishing conditions outlined above. To enable the dislocation structures observed to be related to surface features, a number of specimens were electropolished from one side only, thus retaining the deformed specimen surface as one surface of the foil. Prior to TEM examination using a JEOL 200 CX microscope operated at 200 kV, specimens were stored in liquid nitrogen to maintain their temperature at a low value. A Cambridge Stereoscan 250 scanning electron microscope was used for surface observations.

Results
Figs 1 and 2 show examples of two types of dislocation configuration observed after fatigue at \( \varepsilon = \pm 1.4 \times 10^{-3} \) for 5000 cycles. In fig.1 matrix structure can be seen, consisting of dense irregular regions of dislocation loop patches with narrow irregular channels which have a relatively low dislocation density. The second structure, apparent in fig.2, has regions which approximate to the 'ladder structure' observed in Cu at room temperature. The dislocations which cross the channels of the structure have a single value of the Burgers vector and are close to a screw orientation; the walls, which are not completely planar, are approximately normal to the active Burgers vector. Near the centre of this micrograph is a region which can be interpreted as uncondensed matrix structure.
The separation of the walls is \( \approx 2.8 \ \mu\text{m} \) ie \( \approx \) four times the wall separation for PSBs in Cu at 77K (6) and \( \approx \) twice the separation for Cu fatigued at room temperature (7).

For values of \( e_0 \) between \( \pm 8 \times 10^{-4} \) and \( \pm 1.4 \times 10^{-3} \) after a cumulative plastic strain of \( \approx 24 \), a large fraction of the grains contained the labyrinth structure based on dislocation walls close to \( \{100\} \). A typical example is shown in Fig 3. Unlike the most usual observations of structures based on \( \{100\} \) walls in Cu fatigued at room temperature, we have frequently observed large areas dominated by a single set of \( \{100\} \) walls in Al fatigued at 77K. An example of this feature is shown in Fig 4. The observations of Jin and Winter on the room temperature fatigue of Cu crystals with a \{001\} orientation reveal a similar structure (see Fig 1(b), (8)), however in Al at 77K the dominance of a single set of walls is much more extensive than that reported for Cu. In Al the separation of the walls in the labyrinth structure is approximately 1.6\( \mu\text{m} \), and in the single set of \( \{100\} \) walls the separation is \( \approx 1.7 \mu\text{m} \) (ie \( \approx 2.2 \times \) the separation observed for similar structures in Cu at room temperature (9).

Other orientations of dislocation walls have been observed particularly at larger strain amplitudes at 77K, and also in Al fatigued at room temperature. These structures will be discussed in detail in a later publication.

An unexpected type of dislocation configuration is shown in Fig 5. This specimen was fatigued at \( e_0 = 1.4 \times 10^{-3} \) at 77K for 5000 cycles, and in this case the specimen contained the surface. This structure can be described as a square 'lattice' of dislocation dense regions with side parallel to \( \langle 110 \rangle \) directions and with a 'lattice parameter' \( \approx 1.9 \mu\text{m} \). A magnification TEM micrograph in Fig 6 shows how extensive this structure can be. A SEM micrograph Fig 7, of a part of the area shows a complementary arrangement of circular surface features which are below the surrounding surface.

Thus the dark contrast in Fig 5 corresponds to thinner regions of the foil and is predominantly strain contrast. This contrast corresponds to regions with a high density of dislocations which have Burgers vectors parallel to \( \{011\} \), \( \{101\} \), \( \{110\} \) and \( \{110\} \) in approximately equal numbers. Between the circular regions of dark contrast are areas relatively free of dislocations.

Discussion

The technique which has been used for studying Al, fatigued at 77K, followed by TEM observations involving only short periods of time at room temperature, would appear to provide good evidence for the dislocation structures produced by fatigue at the low temperatures. The fact that some important dislocation configurations are present which are closely similar to those observed in Cu fatigued at room temperature supports this view. There is expected to be some dislocation loss and rearrangement, but there is no a priori reason to believe that this will be greater than in other studies of fatigue using TEM, except for the possibility of enhanced dislocation movement due to climb caused by the condensation of point defects.

The observations of Ceresara (10) on resistivity changes in Al fatigued in torsion at 77K show that a large decrease in defect density, corresponding to a reduction of \( \approx 50\% \) in the fatigue induced resistivity, occurs on annealing up to 300K. It is likely that a major contribution to this resistivity change arises from point defect loss. An examination of the channels between regions of high dislocation density in the present work (eg in Figs.1 and 3) reveals a very high density of small loops - a higher density than observed in Cu fatigued at room temperature. It is suggested that many of these small loops may be from vacancies formed during fatigue at 77K which become mobile at a higher temperature. The condensation of point defects may partly act to stabilize the large scale dislocation structures by pinning. The dislocation structures which we have observed by this technique are not observed when Al is fatigued at room temperature.

A more complete discussion of the dislocation structures in Al, fatigued at 77K will be published elsewhere. For the present purposes the main aim is to show that the dislocation structures observed in fatigued pure Al are very similar to other fcc metals of high stacking fault energy providing the fatigue temperature is lowered. In the experiments reported here the fatigue temperature was \( \approx 0.06 \text{Tm} \) whereas in the case of Cu fatigued at room temperature the testing temperature is \( \approx 0.22 \text{Tm} \), where Tm is the appropriate melting temperature. Measurements made by Basinski, Korbel and Basinski on the dislocation microstructure in Cu after fatigue at different temperatures suggests that the spacing in the wall structures decreases with decreasing temperature (6). The results on Al suggest that the spacing between condensed dislocation structures is approximately four times as large as in Cu at equivalent temperatures.

The lattice type of structure has not been reported in Cu or Ni at the present time. It is possible that it would be more unlikely to form in these metals if we take into account the observed differences in spacing of the dislocation structures and suppose that the dislocation clusters themselves would remain relatively constant in size. If the clusters begin to overlap the structure would be expected to be unstable.
Figs. 1 to 6 are TEM micrographs of dislocation configurations in polycrystalline Al fatigued at 77K at a constant total strain amplitude of ±1.4.10⁻³. Figs. 1 and 2 show matrix and PSB ladder structure. Fig. 3 shows a well-defined {100} labyrinth wall structure and fig. 4 a region containing a single set of {100} walls. Figs. 5 and 6 show the lattice-type arrangement.
The occurrence of the lattice type structure would appear to depend on the presence of significant numbers of dislocations with 4 distinct Burgers vectors, whereas the labyrinth structure can be formed with only 2. It seems likely that a tensile axis with an orientation very close to [001] is required. The observed surface structure can be understood as the result of an extrusion process in which the regions surrounding the clusters are relatively soft. Thus a square lattice of approximately circular depressions is observed on the surface. A similar, but less well defined, surface structure has been reported for Al fatigued at room temperature at low strain by King and Teer, and by Forsyth (11, 12). It is not clear, at the present, whether a regular arrangement of circular depressions (as in Fig 7) is formed in Al when fatigued at room temperature.

It is not possible to determine whether the approximately circular patches of dislocations correspond to spherical or cylindrical groupings. In either case the regular arrangement on a lattice strongly suggests that a low energy dislocation configuration is formed in which a repulsive interaction between the clusters plays a prominent role.

Acknowledgements

This work has been carried out with the support of the Procurement Executive of the Ministry of Defence.

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TEM of dislocation structures in Al fatigued at 77 K

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Abstract

High purity Al has been fatigued at 77K and precautions taken to reduce subsequent ageing to a minimum. Under these conditions dislocation structures, not seen after room temperature fatigue, are observed similar to those seen in Cu and Ni fatigued at room temperature. In addition a new 'lattice-type' configuration is observed. The scale of the structures are about 4 times larger than those observed in Cu at 77K.

Introduction

Extensive TEM studies on several fcc metals and alloys fatigued at room temperature have revealed a consistent pattern of dislocation arrangements. These highly reproducible configurations depend upon orientation, amplitude and stacking fault energy and have recently been summarized in an extensive review (Laird et al 1986). For medium to high values of stacking fault energy, at moderate or low fatigue strain amplitudes, an increase in the number of cycles results successively in the formation of a matrix structure, a persistent slip band (PSB) ladder structure or, where there is multiple slip near [001], a labyrinth or maze structure. These structures are built up from edge dislocation dipoles, either loosely, in the case of the matrix structure or tightly packed, parallel to well-defined planes, in the case of the ladder or labyrinth structures.

Pure Al fatigued at room temperature shows none of these well-developed structures, only an irregular cell structure is formed. Because this might result from the fatigue testing temperature being relatively high in comparison with the melting temperature it is important to attempt to study dislocation configurations in Al at temperatures much lower than room temperature. Liquid nitrogen temperature is ideal for this purpose since the ratio T/T_m of the fatigue test temperature (T) to the melting temperature (T_m) is 0.08 for Al at 77K. For Cu and Ni at room temperature the ratios are 0.22 and 0.17 respectively. To reduce recovery effects we have designed the experimental work so that the fatigued Al has a very small ageing time at temperatures above 77K.

Experimental Techniques

Polycrystalline, annealed Al specimens were fatigued in the form of 3 mm
discs of 90 µm thickness so that they could very rapidly be thinned by jet polishing for TEM. The fatigue cyclic straining was accomplished by cementing the discs onto a 3 mm thick hardened Al alloy beam which was cycled in reversed bending. The discs being very thin and attached to the surface of the beam underwent cyclic strain which was to a good approximation tension-compression between constant total strain limits ($\varepsilon_t$). Cyclic straining was carried out under a flow of liquid nitrogen. After a given number of cycles (N) the glue was dissolved in a solvent at ~ - 50°C and then TEM specimens were prepared by jet thinning using an electrolyte at ~ - 10°C. The specimens were examined in a 200 CX microscope operated at 200 KV. At all other times specimens were stored in liquid nitrogen.

Experimental Results & Discussion

A matrix structure very similar to that seen in Cu fatigued at room temperature was found in many grains examined for amplitudes between $\pm 4.10^{-4}$ and $1.4.10^{-3}$, Figure 1. When compared with Cu the 'clear regions' in Al, between the dipole clusters contain many more small loops and the volume occupied by the dense clusters is a smaller fraction of the whole. Some of the loops may be a result of point defect condensation since the high mobility of point defects produced in Al fatigued at 78K and isochronally annealed up to room temperature has been established by Ceresara (1969). In some other grains structures which were similar to the PSB ladder structures were observed, Figure 2. In this area a significant number of non primary dislocations are in the channels; in the centre of the micrograph is a remnant of the original matrix structure.

**Fig. 1.** Al fatigued at 77K.
$\varepsilon_t = \pm 1.4.10^{-3}$. $N = 5.10^3$

**Fig. 2.** $\varepsilon_t = \pm 1.4.10^{-3}$ $N = 5.10^3$
High resolution electron microscopy

It is probable that the relatively ill-defined fragmented ladder structure is due to a greater amount of secondary slip compared with Cu oriented for single slip. We have observed similar behaviour in Cu fatigued at room temperature near surface indentations (Abdur-Razzaq and Charsley, unpublished) where secondary slip is enhanced. In the case of Al the wall separation (~ 2.8µm) is about four times the wall separation in Cu fatigued at the same temperature (Basinski et al 1980).

In many of the grains large areas were seen of well-defined walls, on a single set of {100} planes, as shown in Figure 3. However, in many regions of this micrograph there is a strong tendency for an individual wall to become split and, at the top of the micrograph a partly formed labyrinth can be seen. Figure 4 shows a part of Figure 3 at higher magnification, the splitting of walls can be seen clearly. Again there is a high density of loops in the channels and screw dislocations cross between the walls. The contrast on either side of some of these dislocations shows that screw dislocations with Burger's vectors of opposite sign are present within the same channel moving in opposite directions. The screw dislocations are expected to carry the major part of the fatigue plastic strain. Insofar as the high loop density is representative of the as-fatigued material it is to be expected that there will be a large frictional stress opposing the movement of the screws. Again the wall separation is much larger than in Cu but a determination of {100} wall separation for Cu fatigued at this temperature has not been made.

Fig. 3. $\varepsilon_t = \pm 1.4 \times 10^{-3}$, $N = 5 \times 10^3$

Fig. 4. Part of Figure 3 at higher magnification
In Figure 5 is shown a region of an extensive array of approximately circular dislocation clusters. This 'lattice-type' of dislocation configuration, aligned accurately along <110> directions, has not been reported for any other fatigued fcc metals. The dislocations in the channels would appear to be readily mobile and correspond to surface extrusions (observed in SEM) which are aligned along these directions. In the regions of the clusters at least 4 different Burger's vectors are found.

In conclusion there are many similarities between dislocation configurations in Al fatigued at 77K and in Cu fatigued at room temperature or at lower temperatures. There are however, many differences in detail and in the sizes of the structures some of which can be related to the ease of secondary slip and differences in the stacking fault energy.

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TEM of dislocation structure near regions of surface damage in Al

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1. Introduction

Previous work on copper fatigued in reverse bending has shown that persistent slip bands (PSBs) form much earlier near regions of surface damage (indentations) than for regions well away from indentations (Charsley, Puttick and White 1981, White 1984). The observed PSB distribution cannot be explained by surface topography changes or by residual stresses due to the indent, however it may be attributed to the dislocation arrangements produced by indenting (Charsley and White 1985). The work on copper was confined to surface studies of PSB formation. In order to examine the associated dislocation structures work has begun on aluminium, and preliminary observations of the microstructure near indents which have not been fatigued are presented here. The main experimental difficulties are the production of TEM specimens which enable the dislocation structure to be studied at known and various positions near indents.

2. Experimental

The surfaces of polycrystalline aluminium of 99.99% purity (90 μm thick) and a single crystal of high purity aluminium (2 mm thick) grown by the Bridgeman technique, were prepared by mechanical polishing and electro-polishing in a nitric acid/methanol solution. The orientation of the single crystal was ~5° from [013] and had a shape shown in Fig. 1a which provided a region of uniform surface cyclic strain during reverse bending, suitable for the study of PSB formation near indents. After annealing for 1 hr at 425°C, a diamond pyramid indenter was used to produce indents of diagonal lengths (d) 56 μm (30 gm load) and 36 μm (10 gm load), in the single crystal and polycrystalline specimens respectively. Before cutting discs around these indents by spark erosion, a section was spark eroded from the indented region in the single crystal and its thickness was reduced to ~0.2 mm by mechanically polishing the non-indented side. Discs were prepared for TEM by jet thinning from the non-indented side only. This generally gave rise to a perforation which covered the major part of the indent, but enabled the dislocation structure in the resulting thin areas to be related to the indent edges by using appropriate low magnifications. Specimens were examined using a 200 CX microscope operated at 200 kV using both bright field and weak beam techniques.
3. Results

An example of the PSB distribution near an indent in the fatigued single crystal is shown in Fig. 2. PSBs have not nucleated within the pit or directly at its edges. The distance from the indent centre to the closest PSBs is d (i.e. ~ 60 μm); a second group of PSBs has formed at 2.1d (i.e. ~ 120 μm). The dislocation structure around an indent within a single grain in polycrystalline material has been studied at the following distances from the indent centre, 0.5d (i.e. at the edge of the indent), 1.2d, 2.3d and 3.3d (see Fig. 1b). In the single crystal material the dislocation structure has been studied at 0.5d only.

Fig. 1(a) Shape and dimensions of the single crystal. (b) Positions of areas at 3.3d(A), 2.3d(B), 1.2d (C) and 0.5d(D) relative to the indent in the polycrystalline material.

Fig. 2 Optical micrograph of PSBs developed near an indent in a fatigued Al single crystal. The direction t is the specimen axis.

At both 3.3d (Fig. 3) and 2.3d(Fig. 4) the dislocation arrangement consists of loose tangles of dislocations with areas of low dislocation density between the tangles, however the number of dislocations present in the tangles appears higher at 2.3d. At 1.2d (Fig. 5) dislocation tangles are again evident but the density of dislocations is much higher than at 3.3d and 2.3d mainly because the dislocation free areas are much smaller. Examination of micrographs taken using different operating reflections (g) show that in all three areas dislocations with at least 4 Burgers vectors (b = ± [110], b = ± [110], b = ± [011] or ± [011] and b = ± [101] or ± [101]) are present. The majority (> 75%) of dislocations at 3.3d have Burgers vectors b = ± [110] with the vertical grouping of dislocations (indicated by the arrow) mainly of this type, and those approximately parallel to [110] mainly with b = ± [011] or ± [011]. At 2.3d the majority...
Metals and semiconductors

(\textasciitilde 70\%) of dislocations which are approximately parallel to [\textit{110}] have Burgers vectors \(b = \pm [110]\); in the arrowed vertical grouping approximately 70\% have Burgers vectors \(b = \pm [110]\). Slip traces parallel to [110] and [110] indicating that movement of dislocations has occurred are apparent both at 3.3d and 2.3d. At 1.2d the majority (\textasciitilde 80\%) of dislocations have Burgers vectors \(b = [110]\) or \(\pm [110]\) and there is some grouping of dislocations along the directions indicated by the arrows. At the corner of the indent (0.5d) an elongated cell structure lying approximately parallel to [110] is apparent (Fig. 6). Dislocations present in this area have the following Burgers vectors: \(b = \pm [110]\); \(b = \pm [10\bar{1}]\); \(b = \pm [110]\) or \(\pm [101]\) and \(b = \pm [011]\) or \(\pm [01\bar{1}]\).

Cell structures are also formed at 0.5d in the single crystal (Figs. 7 & 8). The cells are elongated and are of variable size. Typically the cell width is \(\approx 0.5 \text{ \textmu m}\). Comparison of images taken using different values of \(g\) showed that the cell walls were made up of a number of different Burgers vectors. At E in Fig. 7 dislocations present have Burgers vectors \(b = \pm [011]\).

Generally the areas between the cell walls are relatively free of dislocations. Misorientation between the cells is indicated by the change in contrast across the cell walls. The presence of internal stress is indicated by these misoriented cells and by the point that dislocation walls appear curved. The absence of slip traces at 0.5d is an indication that dislocations cannot readily move in these areas.
4. **Conclusion**

The results presented show the following:

(i) The dislocation structure in areas near an unannealed indent where PSBs are observed to form during fatigue in reverse bending, appears to consist of dislocation tangles, the density of dislocations decreases with distance from the indent centre.

(ii) Near the edge of an unannealed indent where no PSBs are observed to form during fatigue testing, the dislocation arrangement consists of cell structures, and evidence of internal stress is apparent.

5. **Acknowledgements**

The authors would like to acknowledge financial support by the Ministry of Defence (R.A.E., Farnborough).

6. **References**
