

SLIP CONTINUITY ACROSS GRAIN BOUNDARIES  
IN ALUMINUM

by

KEITH GORDON DAVIS

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF  
THE REQUIREMENTS FOR THE DEGREE OF  
MASTER OF APPLIED SCIENCE

in the Department

of

MINING AND METALLURGY

We accept this thesis as conforming to the  
standard required from candidates for the  
degree of MASTER OF APPLIED SCIENCE.

Members of the Department of  
Mining and Metallurgy

THE UNIVERSITY OF BRITISH COLUMBIA

May, 1959

# ABSTRACT

A study of slip continuity across grain boundaries in high-purity aluminum has been carried out, both on bicrystals and on polycrystal strip. Metallographic methods have been used to show that slip continuity is not confined to the surface, but is a true continuity present within the metal. Orientations favouring continuity have been determined, which indicate that screw dislocations can pass through grain boundaries more easily than edge or mixed dislocations.

In presenting this thesis in partial fulfilment of the requirements for an advanced degree at the University of British Columbia, I agree that the Library shall make it freely available for reference and study. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by the Head of my Department or by his representatives. It is understood that copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Department of Mining and Metallurgy

The University of British Columbia,  
Vancouver 8, Canada.

Date 1<sup>st</sup> May 1959

#### ACKNOWLEDGEMENTS

The author is grateful for the advice and encouragement given by his research director, Dr. E. Teghtsoonian, and for technical assistance given by Mr. R.G. Butters and Mr. R. Richter. The work was carried out with the help of the Defence Research Board Grant Number 7510-29. Financial assistance was also received from the National Research Council and in the form of an Inco Fellowship.



## TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION . . . . .	1
The Deformation of Polycrystals . . . . .	1
Previous Work on Plasticity in Bicrystals . . . . .	3
The Problem . . . . .	5
EXPERIMENTAL PROCEDURE . . . . .	7
Bicrystal Work . . . . .	7
Preparation . . . . .	7
Orientation determination . . . . .	7
Electropolishing . . . . .	9
Testing . . . . .	10
Work on Thin Polycrystal Strip . . . . .	10
Methods Used to Try to see Slip Bands in the Interior as	
Revealed by Further Polishing After Testing . . . . .	12
(a) X-ray microscopy . . . . .	12
(b) Etching . . . . .	12
(c) Precipitation of a second phase . . . . .	12
BICRYSTAL ORIENTATIONS . . . . .	15
RESULTS . . . . .	17
Observations on Continuity . . . . .	17
Slip at the Boundaries . . . . .	19
(1) Where slip is continuous . . . . .	19
(2) Where there is no continuity . . . . .	19
Stress-Strain Curves . . . . .	20
Observations on Thin Polycrystal Strip . . . . .	20

## TABLE OF CONTENTS (continued)

Page

Slip Below the Surface . . . . .	20
(1) X-ray microscopy . . . . .	20
(2) Etching . . . . .	22
(3) Al - 4% Ag alloy strip . . . . .	22
Slip Continuity in Annealed Bicrystals . . . . .	23
Slip Continuity Observed on Anodised Surfaces . . . . .	23
<u>DISCUSSION</u> . . . . .	24
1. The Slip Systems Active in Symmetric Tilt Bicrystals . . . . .	24
2. Slip in the Interior . . . . .	24
3. Slip Band Continuity . . . . .	25
4. Possible Mechanisms for Slip Band Continuity . . . . .	25
(a) A theoretical treatment of dislocation source activation across a boundary . . . . .	27
(b) An examination of the possible movement of dislocations through a boundary . . . . .	30
5. Theoretical Considerations on the Effect on Continuity of the Angle of Dislocation Twist ( $\phi$ ) at the Boundary . . . . .	32
6. Theoretical Estimation of the Frequency of Continuous Slip Across Boundaries in a Polycrystal . . . . .	34
7. Further Evidence for the Transparency of Crystal Boundaries to Screw Dislocations . . . . .	34
8. Reasons Why Screw Dislocations Should Pass More Easily Through Grain Boundaries than do Edge or Mixed Dislocations . . . . .	37
9. Conclusions . . . . .	38

<u>TABLE OF CONTENTS</u> (continued)	<u>Page</u>
APPENDIX I . . . . .	40
Electropolishing Large Areas of Aluminum . . . . .	40
Introduction . . . . .	40
Experimental details . . . . .	40
Results . . . . .	41
Perchloric acid-ethyl alcohol solution . . . . .	41
An electropolish for rapid removal of metal. . . . .	42
Summary . . . . .	42
APPENDIX II . . . . .	43
The Shear Stresses Acting on Slip Planes Ahead of a Pile-up of Dislocations . . . . .	43
APPENDIX III . . . . .	45
An Analysis of the Energy Considerations in the Formation of a Stepped Dislocation at a Twist Boundary . . . . .	45
BIBLIOGRAPHY . . . . .	50
MICROGRAPHS . . . . .	52

## LIST OF FIGURES

	<u>Page</u>
1. Experiment of Bainbridge et al on a zinc bicrystal . . . . .	2
2. Orientations of the aluminium bicrystals used by Fleischer and Chalmers . . . . .	3
3. The crystal growing furnace . . . . .	8
4. The tensile testing apparatus . . . . .	11
5. Schematic diagram for Berg-Barret X-ray microscopy . . . . .	13
6. Orientation of the symmetric bicrystals . . . . .	13
7. Orientation of the twist boundary bicrystal . . . . .	16
8. Orientation of the bicrystals Q1, Q2 . . . . .	16
9. Slip traces on the symmetric tilt bicrystals . . . . .	18
10. Standard cubic stereographic plot showing the stress axis for the symmetric tilt bicrystals . . . . .	18
11. Work hardening slopes in the symmetric bicrystals . . . . .	21
12. The formation of a stepped dislocation in crystal <u>2</u> when a dislocation crosses from crystal <u>1</u> . . . . .	26
13. Values of <u>N</u> for bent continuity . . . . .	28
14. Values of <u>N</u> for straight continuity . . . . .	29
15. As in Fig. 12, when steps in the dislocation in crystal <u>2</u> join neighbouring slip planes . . . . .	26
16. Area of the boundary plane favourable to slip band continuity if $\phi \text{ max} \approx 15^\circ$ . . . . .	35
17. Dislocation structure for a low angle tilt boundary . . . . .	35
18. The passage of a dislocation through a boundary . . . . .	47

## LIST OF TABLES

	<u>Page</u>
1. Test for accuracy of orientation determination . . . . .	9
2. Orientation of the symmetric bicrystals . . . . .	15
3. Observations on bent continuity . . . . .	17
4. The angle $\alpha$ between slip direction and the boundary plane for the symmetric tilt boundary . . . . .	31
5. Angle of dislocation twist $\phi$ . . . . .	31

# SLIP CONTINUITY ACROSS GRAIN BOUNDARIES IN ALUMINUM

## INTRODUCTION

### The Deformation of Polycrystals.

Single crystals are weaker than polycrystals. One of the basic problems in physical metallurgy is to find out precisely why this is so. It is hoped that such knowledge will help in the development of polycrystal material with greater strength.

Two mechanisms strengthen polycrystals relative to single crystals. The first, and in many cases probably the most important mechanism, arises from the need for several slip systems to act in each grain in order to keep neighbouring grains in contact. Von Mises<sup>1</sup> has shown that unless at least five slip systems per grain are active, gaps will appear at the boundaries, a result that has never been challenged. Interaction between these slip systems will cause hardening. The question of which extra slip systems will be activated and how much slip each will give has been the subject of extensive research based on comparison between stress-strain curves for single crystals and for polycrystal specimens (see Refs. 2, 3, 4, 5). In later remarks the term 'complexity hardening' will be used to describe this type of mechanism.

The second mode of strengthening in polycrystals is the holding up of moving dislocations at grain boundaries. McLean<sup>6</sup> calls this 'barrier hardening', and his terminology will be followed. Where two slip systems of neighbouring crystals nearly coincide, lower barrier hardening might be expected. For F.C.C. and B.C.C. metals with many slip systems per grain,

barrier hardening will be less important than in C.P.H. metals (polycrystalline C.P.H. metals tend to have very rapid rates of work hardening compared with their single crystals). Alloys with a hard phase may be considered an extreme case of barrier hardening where the barrier virtually never breaks down.

The strength of a boundary as a dislocation barrier appears to be greatly enhanced by the presence of a solute with small atomic diameter which gives Cottrell locking. Sharp yield of the type observed in mild steel is now generally held to be caused, in the cases investigated, by the presence of such solutes: the yield is thought of as a catastrophic break through of dislocations across boundaries.

That even a low angle boundary can act as a dislocation trap has been demonstrated clearly by Bainbridge et al.<sup>7</sup> A low angle boundary zinc bicrystal was stressed as shown in Fig. 1. As the stress was increased, dislocations travelling along the basal planes were trapped in the boundary and the boundary angle increased. There must therefore be some degree of barrier hardening even from such low angle boundaries. To clarify the relative importance of complexity hardening and barrier hardening, studies have been carried out on the deformation characteristics of bicrystals.

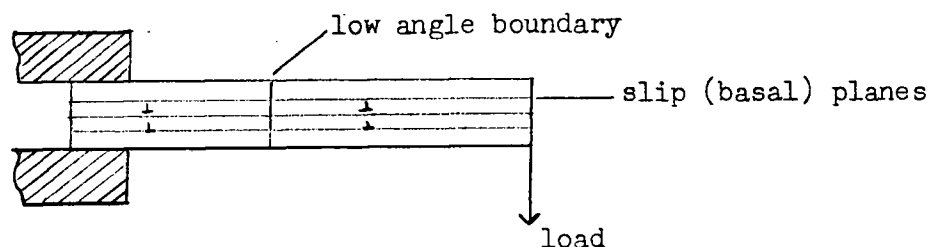


Fig. 1. Experiment of Bainbridge et al on a zinc bicrystal.

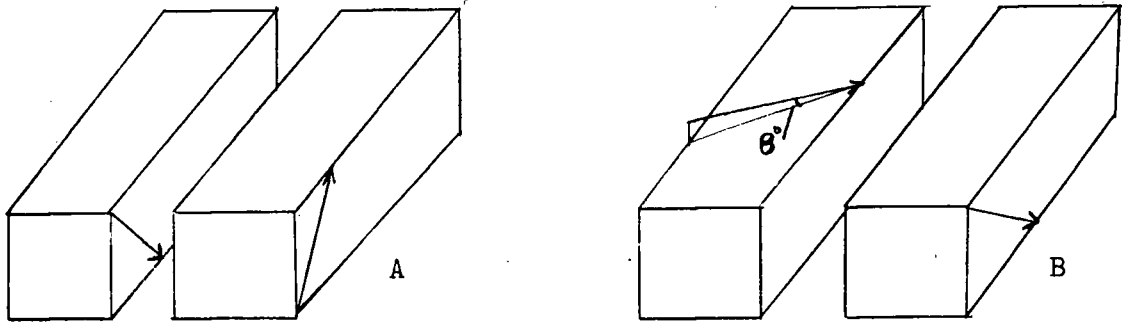


Fig. 2. Orientations of Al Bi-x's used by Fleischer and Chalmers. In A, slip directions are parallel to the boundary. In B, one crystal has its slip direction parallel and the other nearly normal to the boundary.

### Previous Work on Plasticity in Bicrystals.

Bicrystals of Zn, Sn and Al have been tested. In all the cases to be described the boundary was kept parallel to the stress axis.

Zn (a) Kawada<sup>8</sup> grew symmetric bicrystals with basal planes inclined at equal angles to the boundary. They showed the same strength as single crystals. A relative rotation about the stress axis gave some strengthening.

(b) Gilman<sup>9</sup> confirmed Kawada's results that in symmetric bicrystals the boundary gives no hardening. He also found a high degree of strengthening in bicrystals with a relative rotation about an axis normal to the boundary.

Sn Chalmers<sup>10</sup> grew symmetric bicrystals, varying the angle between the slip directions. The stress needed to produce a standard small extension was measured. He found that -

(1) the stress increased linearly with orientation difference between the two crystals from 0-90°.

(2) varying the orientation of the boundary so that it was inclined to the stress axis gave no difference in strength for the same relative orientation between the two crystals.



High-purity Al (a) Clark and Chalmers<sup>11</sup> carried out experiments on two crystals with a common  $\{111\}$  slip plane at  $45^\circ$  to the stress axis, rotations being made in these  $\{111\}$  planes to vary the angle  $\phi$  between favoured slip directions. They found that -

(1) the yield stress increases with  $\phi$  up to  $\phi = 30^\circ$ , and then remains steady.

(2) the initial work hardening rate increases linearly with  $\phi$  up to  $\phi = 60^\circ$ .

(b) Aust and Chen<sup>12</sup> grew symmetric bicrystals based on a seed crystal with a  $\langle 110 \rangle$  stress axis and a  $\{100\}$  plane facing upwards. Rotations were made about the  $\langle 110 \rangle$  axis. Yield stress and initial work hardening rate both increased with misorientation angle  $\theta$  from  $\theta = 5^\circ$  to  $\theta = 85^\circ$ , but the effect was less than that with Clark and Chalmers' bicrystals.

(c) Fleischer and Chalmers<sup>13</sup> grew two series of bicrystals. In one (A) the preferred slip direction was parallel to the boundary in both crystals. In the other (B) the preferred slip direction in one crystal was parallel to the boundary and in the other crystal nearly normal to the boundary (see Fig. 2). Series A bicrystals showed little strengthening due to the boundary. Series B showed a large boundary effect. Double slip near the boundary was observed only in series B bicrystals.

No clear conclusions about the relative importance of barrier-hardening and complexity hardening can be drawn from these data. The important factors that do emerge are -

(1) the strengthening effect of a boundary depends on the relative orientation of the component crystals in a fairly systematic manner.

(2) aluminum is strengthened only when double slip occurs near the boundary.

### The Problem.

When metals are polished and deformed, surface slip bands in general fade out near to the grain boundaries. In some cases, however, the slip bands pass right through a boundary. Ogilvie<sup>14</sup> has studied this in polycrystalline aluminum and  $\beta$ -brass. He found that continuity of slip occurred only -

(1) across straight boundaries.

(2) where the line of intersection with the boundary of the two active slip planes in neighbouring crystals had  $\langle 110 \rangle$ ,  $\langle 112 \rangle$  and  $\langle 123 \rangle$  directions (within  $2^\circ$ ). The directions were not necessarily the same in both grains.

Other observations of slip across grain boundaries have been published. Lacombe and Beaujard<sup>15</sup> observed slip across low angle boundaries in high-purity aluminum. Observations of slip continuous across twin boundaries have been made in several metals, and Gilman<sup>16</sup> has recently noted that slip is always continuous in symmetric zinc bicrystals.

It was thought that a further investigation of slip across boundaries may be useful. Information was sought on the following questions:

(a) Is slip band continuity as observed on polished surfaces a true continuity or purely a surface effect?

(b) What is the orientation dependence?

(c) What is the actual mechanism for slip through grain boundaries? Do dislocations pass through the boundary, or do pile-ups in one crystal activate dislocation sources in a neighbour?

(d) Is there a connection between barrier hardening and slip band continuity?

Answers to the first two questions should shed light on the third, which is of course the crux of the problem. Of interest is the investigation by Urie and Wain<sup>17</sup> on the deformation of polycrystalline aluminum. They deposited photographically a fine grid on the surface before deformation, and found that in general deformation was less at grain boundaries than near grain centres. However, where slip passed through, the grain boundary had no effect on deformation. Other interesting work was carried out by Kawada<sup>18</sup> on coarse polycrystal aluminum. Sensitive measurements of extension under load showed that, in the early stages of deformation, commercial aluminum between 100°C and 350°C showed sudden elongation 'jumps' of the order of a few microns. There were no jumps when slip was continuous across the boundary, and such jumps were never seen in high-purity aluminum.

## EXPERIMENTAL PROCEDURE

### Bicrystal Work.

#### Preparation

Bicrystals of 99.99% pure aluminum with approximate dimensions  $4\frac{1}{2}'' \times 3\frac{3}{4}'' \times 3\frac{3}{8}''$  were prepared by a modified Bridgman technique. Seed crystals were laid in a graphite boat in contact with a machined slug of aluminum which was covered by a lid. The boat was placed in a Vycor tube, through which helium was slowly passed. A resistor-type furnace was drawn along over the tube by a small electric motor (see Fig. 3). It was found necessary to join the seeds to the melt by mechanical agitation. The solid-liquid interface was approximately  $3''$  within the furnace. Some trouble was encountered with shrinkage on solidification. Shrinkage was reduced by providing a small reservoir of aluminum joined to the melt by a narrow hole. After solidification the aluminum remaining in the reservoir could be easily cut off.

Growth rate was controlled by the speed of furnace travel. This was kept at four cms. per hour, higher growth rates giving pronounced lineage structures. Stray crystals or changes in orientation were rare. About 20% of the crystals showed a few coarse lineage tilt boundaries with orientation differences of around  $1\frac{1}{2}^\circ$ . There rarely occurred more than two such boundaries per crystal, and in all cases they could be etched up easily.

#### Orientation determination.

Crystals were taken from the boat, etched in 'Tucker's etch' and oriented by the X-ray Laue back-reflection method. The accuracy of the method was tested by repeatedly mounting a crystal on the X-ray set, taking a picture, demounting the crystal, and repeating the procedure. A typical example of the

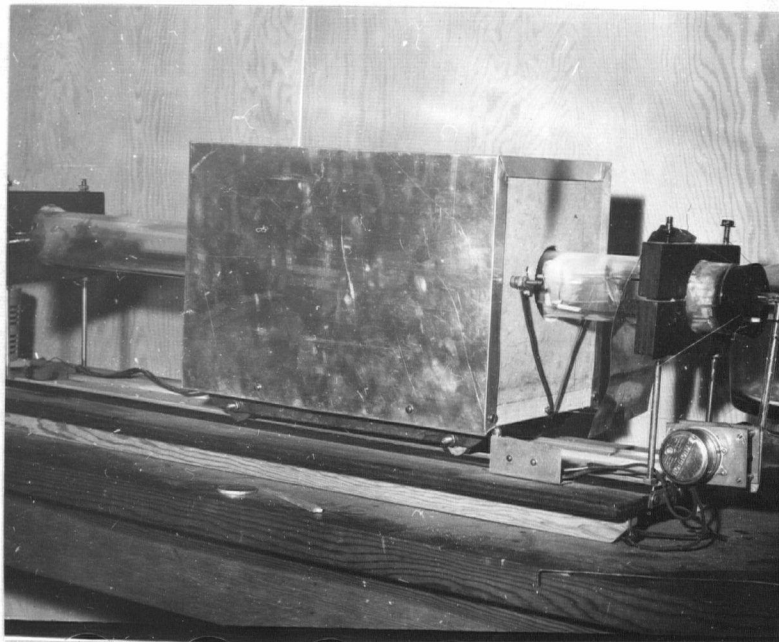


Fig. 3. The crystal growing furnace.

values obtained is given in Table 1. In this case the orientation desired had a {100} plane facing the film holder and a  $\langle 110 \rangle$  axis vertical. The values  $\theta$ ,  $\phi$  represent rotations about the vertical and horizontal axes in the film needed to give a perfect (100)  $\langle 110 \rangle$  orientation, and  $\alpha$  represents the rotation needed about an axis perpendicular to the film.  $\theta$ ,  $\phi$  and  $\alpha$  are all three needed to define the misorientation completely. The method appears accurate to within  $\pm 1^\circ$ . Traverses across a specimen surface were sometimes taken to test for low angle lineage boundaries. Except for the easily etched boundaries of some  $1/2$  to  $1\ 1/2^\circ$ , the structures were perfect within the limits of the method.

TABLE 1

Test for accuracy of orientation determination.

Test Number	$\theta^\circ$	$\phi^\circ$	$\alpha^\circ$
1	$1\ 1/2$	2	0
2	$1\ 1/2$	2	$1/4$
3	1	2	1
4	$1\ 1/2$	$1\ 1/2$	0
5	1	$2\ 1/2$	$1/2$
6	0	3	0

Electropolishing (for details see Appendix 1)

Several electropolishes were tried, the final choice being 15% perchloric acid in acetic acid. With a current of some  $3/4$  amp at 25-35 V. a good polish was obtained in about  $1\ 1/2$  hours. Cooling was effected by a jacket of cold running water. The best results were obtained when stirring was kept very slow, just sufficient to prevent the specimen from overheating. A region about 1" long in the middle of the specimen was polished, the rest being masked off by electrical tape. Preliminary treatment was restricted to grinding with a coarse-grade emery paper. It is believed that the worked

layer was totally removed. X-ray pictures of the polished surface showed no asterism, and annealing caused no recrystallisation.

### Testing.

Electropolished crystals were mounted in a Hounsfield tensometer (see Fig. 4). Simple V-block friction grips were machined from aluminum alloy, and the gripping surface covered with coarse emery paper. It was thought that the more usual serrated gripping surfaces would give unnecessary deformation at the ends of the specimens. Thin rubber was at first tried, but proved insufficiently strong. Even the light aluminum grips gave appreciable bending stresses from their own weight. The tensometer was therefore mounted in the vertical position from a wall bracket. To look for slip continuity, widely spaced slip bands were preferable. To observe the formation of slip bands a travelling microscope with magnification around 100x was placed in front of the specimen. The load was removed when slip bands were first seen to form, at 70 - 100 lbs. Stress-strain curves were recorded while testing.

The gripping system was not rigid, and some bending inevitably accompanied the tensile tests. For two bicrystals, SR 4 electrical resistance strain-gauges were cemented on to opposite faces. Both showed extension up to approximately 0.5% on one face, with either a slight compression or a small extension on the other. This was reflected in the frequent disparity between the density of slip bands on opposite faces.

### Work on Thin Polycrystal Strip.

Thin polycrystal strip, a few hundredths of a mm. thick was prepared to see if any correlation between slip bands on the two opposite surfaces could be found. Identification of the same boundary on both sides is much easier if the grains are large. The strip was therefore rolled, annealed at

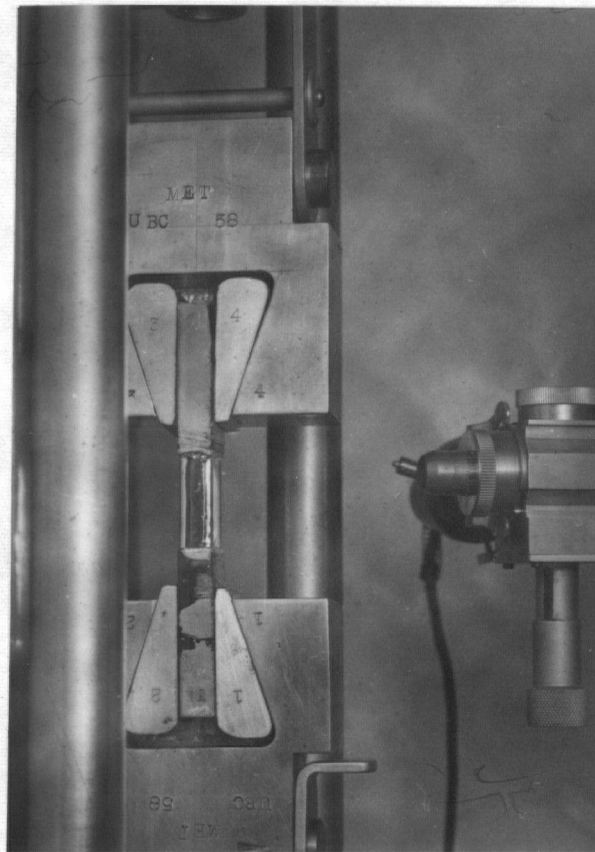


Fig. 4. The tensile testing apparatus.



640°C, given a further small reduction and re-annealed at 640°C for several days. Grains passing right through the strip with diameters of the order of a centimeter resulted. This strip was polished and tested in the same manner as the bicrystal.

Methods Used to Try to See Slip Lines in the Interior as Revealed by Further Electropolishing after Testing.

(a) X-ray microscopy. In this technique, devised by Berg<sup>19</sup> and applied to metals by Barrett,<sup>20</sup> a large area of the specimen surface is bathed in a diverging beam of characteristic X-rays. The photographic plate must be placed within a few mms. of the specimen surface (see Fig. 5). Each point on the plate represents a point on the surface, the plate being viewed, after development, on a metallograph. In this way, inhomogeneities in the structure such as deformation bands, low angle boundaries and scratches can be shown up.

(b) Etching. Two etches were tried. The first, due to Lacombe and Beaujard,<sup>15</sup> gives a very large number of small pits, which are believed to be nucleated at least in part by dislocations. The etch contains 46% HNO<sub>3</sub>, 50% HCl, 3% HF. It must be kept around 0°C by surrounding the dish with melting ice. The second etch, due to Barrett and Levenson,<sup>21</sup> has nine parts HCl, three parts HNO<sub>3</sub>, two parts HF., five parts H<sub>2</sub>O. This develops much larger pits. For both etches the specimen is immersed in the solution for a few seconds.

(c) Precipitation of a second phase. Failure of the first two methods led to the adoption of a precipitation technique. Age-hardening Al - 4% Ag alloys were prepared in the form of coarse-grained polycrystal strip. The strip was homogenised at 600°C, quenched, deformed, and aged at 200°C for 24 hours. Silver quenched in solution precipitated out preferentially in slip bands. The surface had to be anodised before slip bands could be detected.

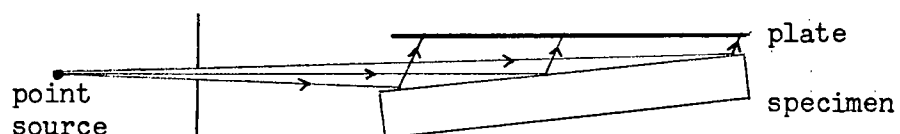


Fig. 5 Schematic diagram for Berg-Barret X-ray microscopy.

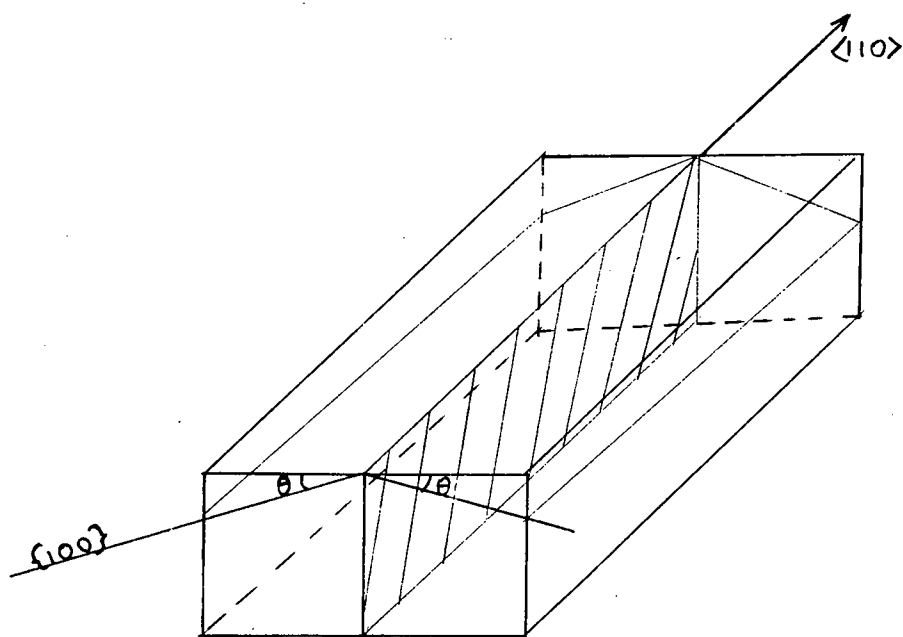


Fig. 6. Orientation of the symmetric bicrystals.

Several attempts to etch up the precipitate in the electropolished surface, removing the necessity for anodising, proved abortive. Perchloric acid-acetic acid electrolytes used in electropolishing pure aluminum were unsuitable for the aluminum-silver alloy, and a 20% perchloric acid in ethanol electrolyte at 40 V was used. This gave a high current, around 8A, accompanied by rapid heating.. After 20-30 seconds the current was switched off and the solution allowed to cool. It was found that if in the final polish the voltage was slightly reduced for a few seconds before the current was switched off a good anodised surface was given.

# BICRYSTAL ORIENTATIONS

A series of symmetric bicrystals was grown based on a seed crystal with a  $\{100\}$  face upward and a  $\langle 110 \rangle$  axis along its length. Seeds for the bicrystals were prepared by tilting this seed about its length axis by an angle  $\theta$  (Fig. 6). The two values of  $\theta$  for a bicrystal varied by up to  $3^\circ$ . At least three crystals were tested for each orientation. One twist boundary was prepared, with  $\theta = 9^\circ$  (see Fig. 7). Single crystals oriented for single

TABLE 2.

Orientation of the symmetric tilt bicrystals.

	Orientation Difference ( $2\theta^\circ$ )			
Code	1	2	3	4
A	5	6	8	
B	21	24	25	
C	26 1/2			
D	39	40	40	
E	75	70	74	73
F	92	93	93	
G	113	115	114	112
H	150	151	150	

slip were prepared, and two series of bicrystals grown from them. In the first, bicrystal Q 1, the preferred slip directions were parallel to the boundary surface. In the second, Q 2, the directions of easy slip lay in a plane nearly parallel to the top surface of the bicrystals (see Fig. 8).

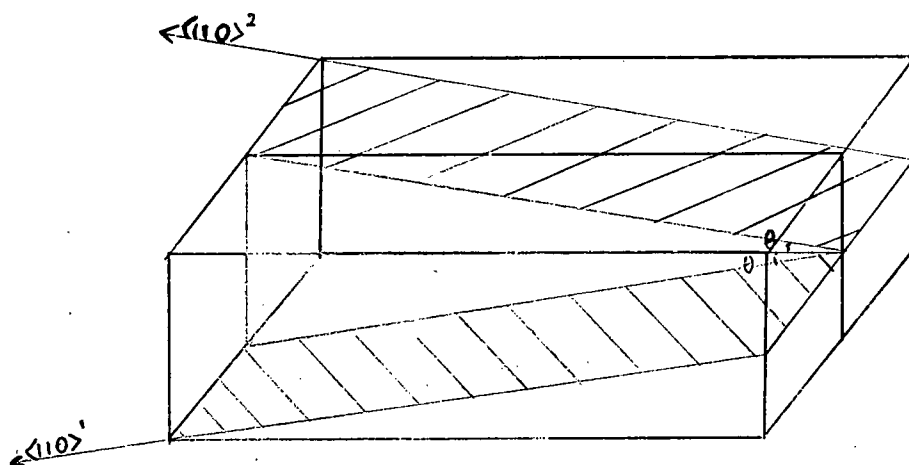


Fig. 7. Orientation of the twist boundary bicrystal. The shaded planes have  $\{100\}$  indices.

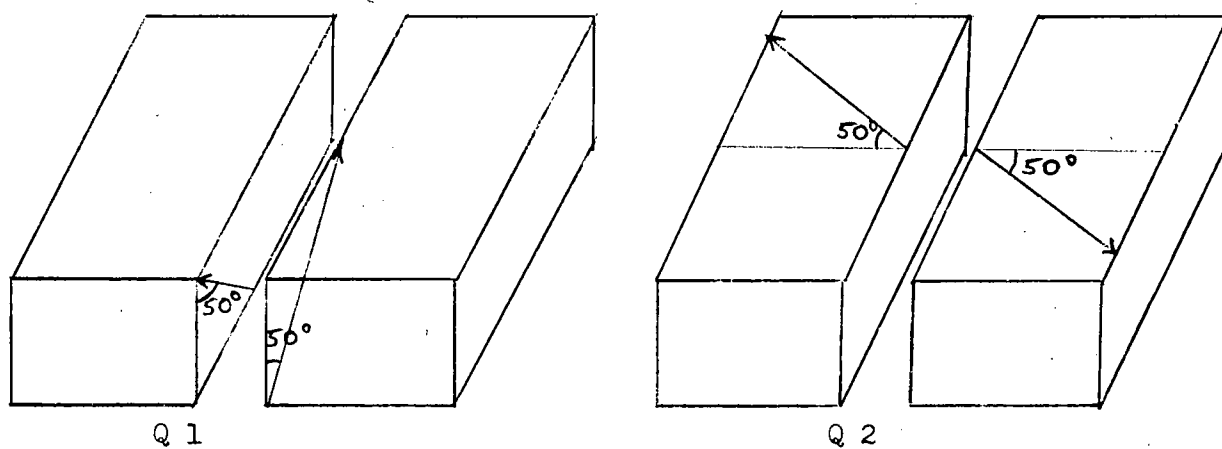


Fig. 8. Orientation of the bicrystals Q 1, Q 2. The slip directions are shown.

## RESULTS

### Observations on Continuity.

There is no clear cut definition between no continuity and continuity. It has been found convenient to use the following terms of description - 'excellent', 'good', 'fair', 'traces'. It is hoped that with the accompanying micrographs a fair description of what was observed will be accomplished. It should be emphasized that some of the specimens varied over their length, giving continuity at some places and not at others. The picture, therefore, cannot be wholly representative. Symmetric bicrystals have two possible sets of slip continuity, straight or bent (Figs. 9 and 10). Straight continuity was seen only once, and then faintly, in the symmetric bicrystals (in H 1, 2, 3). For bicrystal A, it was not possible to differentiate between the two cases. Observations on bent slip are summarised in Table 3 and micrographs 1 to 7. The series of pictures does not give a very true representation. Continuity in E was abundantly clearer than in the other bicrystals, and the straight continuity in H (Micros 11, 12) was clearer than could be shown. The twist boundary bicrystals and bicrystals Q 1, Q 2 showed no continuity. Both tricrystals showed good continuity.

TABLE 3

Observations on bent continuity

Code	Orientation difference ( $2\theta^\circ$ )	1	2	3
A	6	Good	Good	Good
B	23	Good	Fair	Traces
C	26 1/2	Fair	-	-
D	40	Good	Good	Good
E	73	Excellent	Excellent	Excellent
F	93	Good	Good	-
G	114	Fair	Traces	Fair
H	150	Traces	Traces	Traces

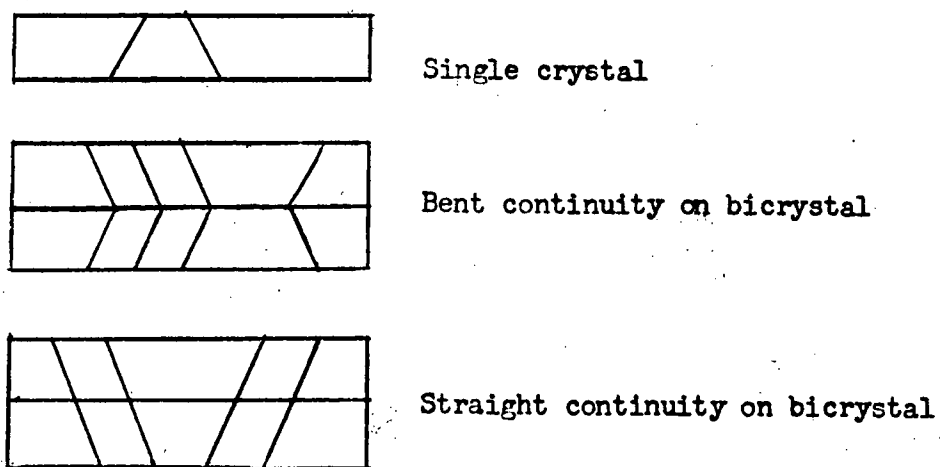


Fig. 9. Slip traces on the symmetric tilt bicrystals.

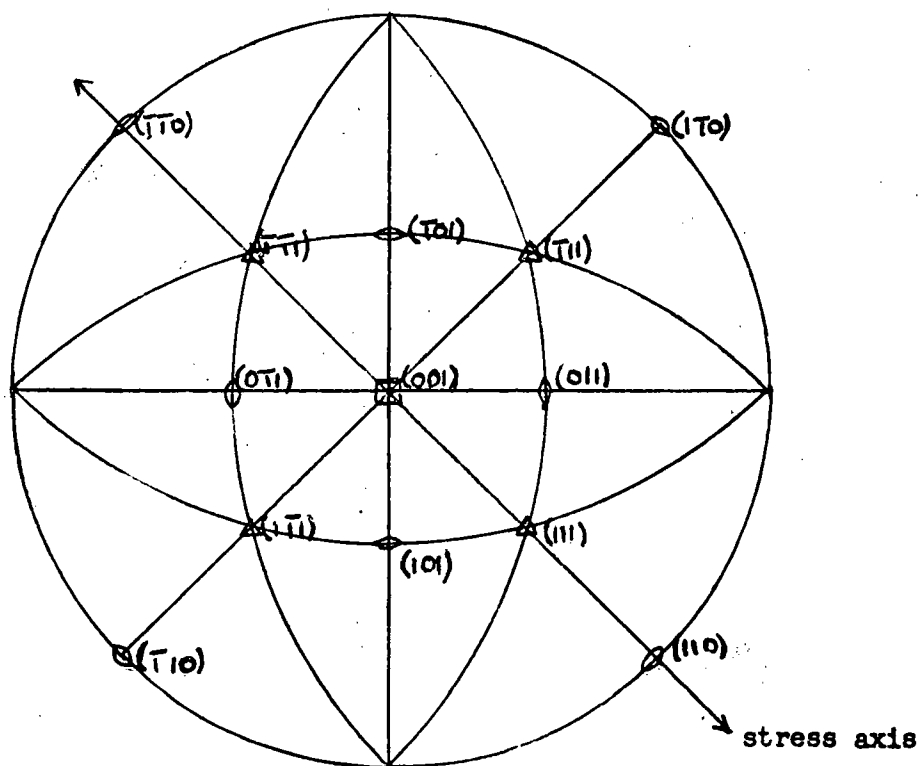


Fig. 10. Standard cubic stereographic plot showing the stress axis for the symmetric tilt bicrystals.

### Slip at the Boundaries

#### (1) Where slip is continuous.

When slip continuity was examined in detail, the following interesting facets were revealed.

(a) In several cases examined there was a displacement of the boundary where crossed (Micro 13). This displacement was of the same order of magnitude as the step height of a slip band.

(b) In bent continuity, the slip band sometimes crossed the boundary straight for a short way before joining a slip band in the second crystal (Micros 14, 15). Across the boundary slip bands sometimes became wavy for a short distance (Micro 16).

(c) Ogilvie<sup>14</sup> had stated that slip bands were continuous only over straight boundaries. This is not so (Micros 17, 18, 19). Further, the orientation conditions he stipulated were not followed here.

(d) Slip bands can turn through quite sharp angles, up to nearly 70° (see, for example, Micro 20).

#### (2) Where there is no continuity.

(a) The slip bands often faded out near to the boundary, giving way to wavy slip or a generally 'rumpled' surface (see Micros 21, 22, 23).

(b) The slip bands tended to run along parallel to the boundary when held up there (Micros 23, 24, 25), occasionally crossing before so doing (Micro 25).

(c) In bicrystals G ( $\sim 110^\circ$ ) the slip direction was very nearly parallel to the polished surface, making the observation of slip bands difficult. A wide region of very wavy slip occurred along the boundary (Micro 26).



### Stress-Strain Curves

The Hounsfield tensometer gave a direct load-extension curve. The early parts of the curve, up to around 30 lbs load, were found to be highly variable. Electrical strain-gauge measurements showed that up to this load there was no appreciable extension of the specimen, and it was assumed that any extension shown was caused by taking up slack at the grips. With few exceptions the load-extension curves were linear above 30 lbs. load; it was believed that the slope in this region indicated a true work-hardening of the bicrystals. Values were found from this slope for extensions at 125 lbs. load over a 3 cm. length, this being a measure of the inverse slope of the curve. Results were not of great precision, due to (1) bending stresses introduced, (2) inaccuracies in the load measuring device, and (3) rather crude estimates of cross-sectional area and distance between the grips. It is however felt that the values obtained were consistent enough to be significant. A plot is made in Fig. 11. The bicrystals E, which showed the best continuity, appear to be more easily deformed than the bicrystals D and F. Aust and Chen<sup>12</sup> gave values of the work hardening slope steadily increasing from 0 - 90° misorientation.

### Observations on Thin Polycrystal Strip

No correlation could be found between the slip band spacing on opposite faces of the strip. It was noted, however, that where continuity was present on a boundary, slip was also continuous over the same boundary in the other face when not obscured by secondary slip (Micros 27, 28).

### Slip Below the Surface.

(1) X-ray Microscopy. The method proved insufficiently sensitive to reveal clearly the slip band continuity, although slip bands could be detected.

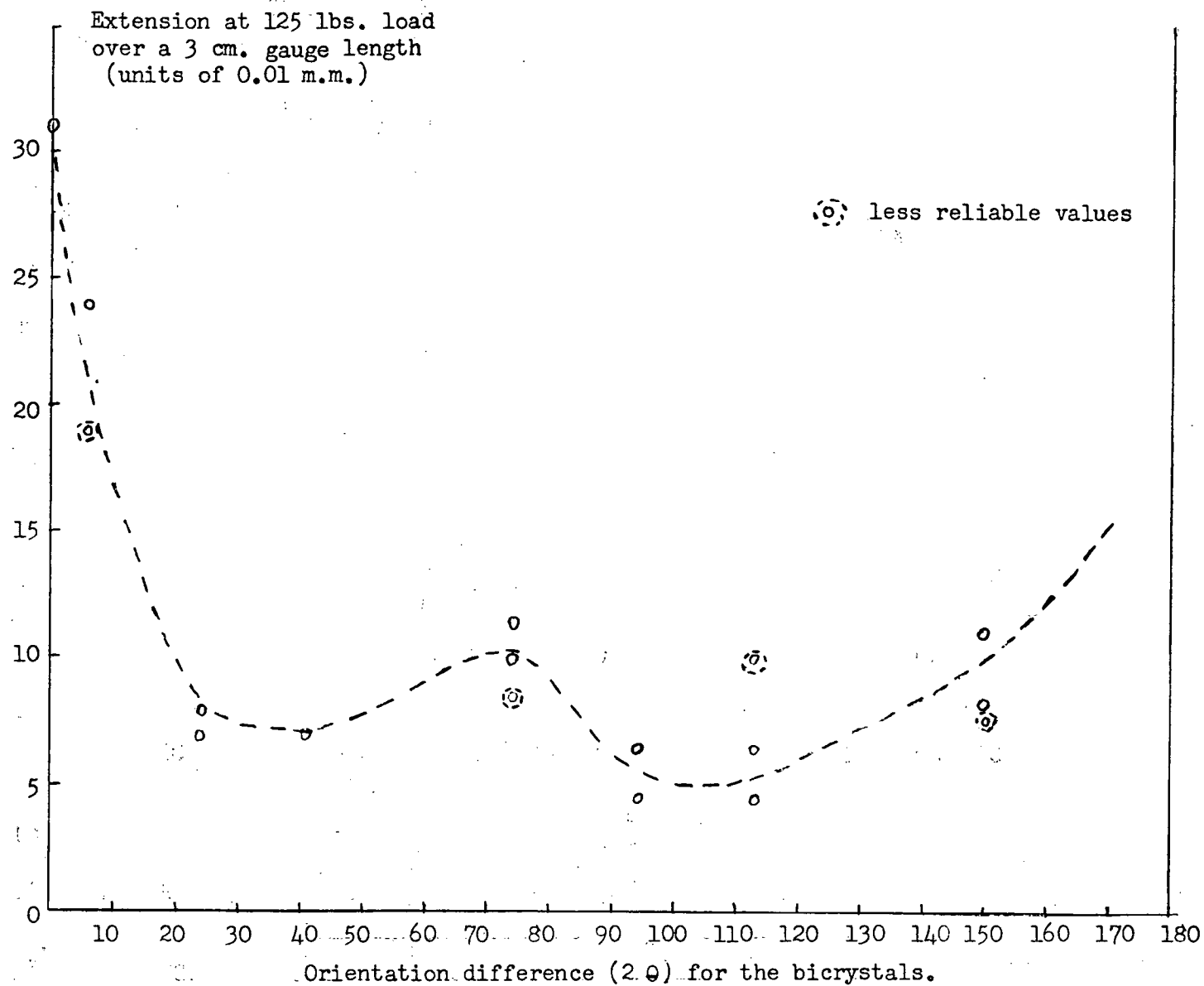


Fig. 11 Work hardening slopes in the symmetric bicrystals.

More suited to this technique would be the examination of sub-boundaries or deformation bands.

(2) Etching. In no case were slip lines revealed. Wyon and Marchin,<sup>22</sup> in an extensive research into the etching of dislocations in high purity aluminum, noted that "it was never found possible to reveal slip markings by this method." Their findings can only be echoed.

The etch-pit patterns are of interest in their own right. Micros 29 and 30 show the effect of the Lacombe and Beaujard etch on a bicrystal with a  $\{111\}$  plane very nearly parallel to the surface, annealed, after growth, at 600°C for several days. Micro 29 shows the surface after severe attack and Micro 30 reveals the dark patches to be overlapping etch pits. Two significant conclusions may be drawn. First, the boundary suffers very little preferential attack, and second, there is no sign of polygonisation or cell-structure. The electropolish has apparently removed completely any surface deformation.

Micros 31, 32, and 33 were taken from etched surfaces of crystals electropolished subsequent to deformation. In none do the etch pits align along slip bands. Micro 31 illustrates the difference in size between etch pits given by the two etches. The large triangular pits were given by Barrett and Levenson's etch and the small ones by Lacombe and Beaujard's. Micro 32 illustrates the very perfect equilateral triangle pits given by Lacombe and Beaujard's etch on a  $\{111\}$  surface, and Micro 33 shows the square pits given by this etch on a crystal near to the cube orientation.  $\{111\}$  surfaces were etched within a few seconds whereas  $\{100\}$  surfaces needed the order of a minute.

(3) Al - 4% Ag Alloy Strip. Pictures of the anodised surfaces taken under polarised light are shown in Micros 34, 35, 36 and 37. They show clearly

that slip band continuity can be present in the interior as well as on the surface. There is of course no guarantee that the same follows for high-purity aluminum with no silver addition, but there is no reason for there to be any difference in deformation mechanisms. The surface appearance of specimens of the Al - 4% Ag alloy electropolished and pulled is identical with that of the pure aluminum. The frequency of continuity in the interior of a polycrystal is not markedly different from that on the surface.

#### Slip Continuity in Annealed Bicrystals.

Two bicrystals, B3 and E4, were annealed at 600°C for several days before testing. No very significant difference in slip behaviour was noted, although bicrystal B3 did show poorer continuity than B1 or B2.

#### Slip Continuity Observed on Anodised Surfaces.

A few bicrystals and polycrystals were anodised before deformation. No difference in slip continuity characteristics could be detected.

## DISCUSSION

### 1. The Slip Systems Active in the Symmetric Tilt Bicrystals.

At room temperature aluminum slips in  $\langle 110 \rangle$  directions on  $\{111\}$  planes. There are four slip planes,  $(111)$ ,  $(\bar{1}11)$ ,  $(1\bar{1}1)$  and  $(\bar{1}\bar{1}1)$ , each with three possible slip directions, making twelve slip systems per crystal. The choice of crystals with a  $\langle 110 \rangle$  stress axis greatly simplifies the picture. A cubic stereogram is given in Fig. 11. Slip planes  $(\bar{1}11)$ ,  $(1\bar{1}1)$  are parallel to the applied stress, and having no shear stress component across them will not give slip. Slip direction  $[1\bar{1}0]$  (or  $[\bar{1}10]$ ) is perpendicular to the applied stress and therefore inoperative. The slip systems reduce to:

$$\begin{array}{lll} (111) & [\bar{1}01], & [0\bar{1}1] \\ (\bar{1}\bar{1}1) & [011], & [101] \end{array}$$

For symmetric bicrystals obtained by a rotation of the standard seed about its  $[110]$  axis, superscripts 1 and 2 will be used to represent slip elements in crystals 1, 2. It is easily seen that  $(111)^1$  and  $(\bar{1}\bar{1}1)^2$ , (or  $(111)^2$  and  $(\bar{1}\bar{1}1)^1$ ) will intersect the top surface in parallel lines, whereas  $(111)^1$  and  $(111)^2$ , or  $(\bar{1}\bar{1}1)^1$  and  $(\bar{1}\bar{1}1)^2$ , will intersect in lines at an angle, giving a herring-bone structure (see Fig. 9). The twist boundary will of course give slip bands normal to the length axis, and the bicrystal Q oriented for single slip will give bands parallel in the two crystals but inclined to the length axis.

### 2. Slip in the Interior.

The Al - 4% Ag alloys show conclusively that slip band continuity is not just a surface effect, but that slip bands join at the boundary throughout the metal. Further, were it simply a surface effect, a specimen with an anodised surface might possibly behave differently from an electro-

polished one, but no such difference was observed. This conclusion poses something of a problem. Unless the active slip planes in neighbouring crystals intersect in a line in the boundary, dislocations cannot pass directly from one crystal to the other. Consider the situation depicted in Fig. 12. The dislocation must take on a stepped form in order to lie completely on slip planes in crystal 2 and keep its continuity with the slip plane in crystal 1. On moving out into crystal 2 the steps are presumably removed by gliding out of the dislocation. Were this not so slip would, to an observer, be taking place on a non-octahedral plane. No record of such an observation is known.

### 3. Slip Band Continuity and Work Hardening.

The tensile tests indicate (Fig. 10) that the bicrystals E, where slip band continuity was very marked, showed rather less work hardening than expected. It appears, therefore, that slip band continuity is a true stress-relaxation, but too much credence should not be placed on this. The scatter in results shows that more reliable data, taking a greater number of tests per example and using a more sensitive machine, should be obtained before a detailed theoretical explanation is justified. It should be noted that Chen and Aust<sup>12</sup> tested no bicrystals with misorientation between  $55^\circ$  and  $85^\circ$ , and would therefore have missed a minimum in work hardening slope at around  $70^\circ$ . The observations of Urie and Wain<sup>17</sup> tie in with the data presented here.

### 4. Possible Mechanisms for Slip Band Continuity.

There are two basic mechanisms that could account for the observed continuity. Either the dislocations pile up against the boundary and their stress fields activate dislocation sources in the next crystal, or the dislocations in some way pass right through the boundary. The source activation theory will be treated first.

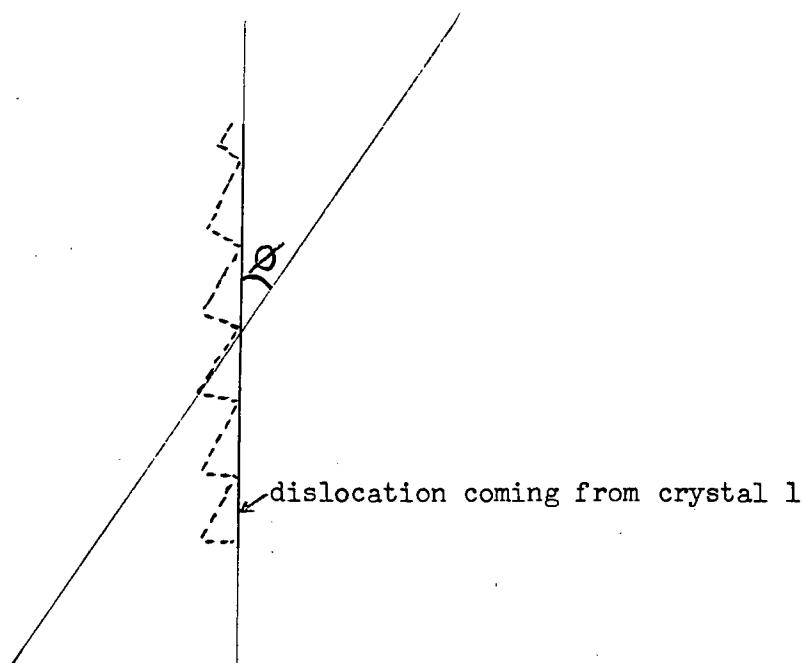


Fig. 12. Formation of a stepped dislocation in crystal 2 when a dislocation crosses from crystal 1. The plane of the diagram is the boundary plane. The dotted line represents the dislocation in crystal 2.

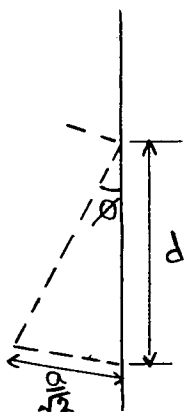


Fig. 15. As Fig. 12, where steps in the dislocation in crystal 2 join neighbouring slip planes.

- (a) A theoretical treatment of dislocation source activation across a boundary.

When  $M$  dislocations with Burgers vector  $b$  pile up against a boundary under an applied shear stress, they possess a stress field ahead of the pile-up with a value equivalent to that of a single dislocation of Burgers vector  $Mb$  at the centre of gravity of the pile-up.<sup>23</sup> There is therefore a stress concentration at the head of a slip band. This stress will be pure shear in the slip plane of crystal 1. If the crystal lattice is assumed elastically isotropic, and in aluminum this should lead to no serious inaccuracies, the stress in crystal 2 ahead of the slip plane in crystal 1 will also be pure shear. For a given shear stress  $\tau$  acting ahead of a slip system of crystal 1 in crystal 2, shear stresses  $N\tau$  acting on each of the four slip systems of crystal 2 can be found, where  $N$  is a geometrical factor. The shear stress from the applied tensile load will be the same on all eight slip systems (four per crystal). A method to calculate  $N$  is given in Appendix 2. If continuous slip is caused by dislocation source activation, high values of  $N$  between any two slip systems in the two crystals should lead to observed slip continuity.  $N$ -values between slip systems in  $(111)^1$  and  $(111)^2$  and between systems  $(111)^1$  and  $(\bar{1}\bar{1}1)^2$  have been calculated for symmetric crystals with misorientations from  $0 - 180^\circ$ ; high values for the first should lead to bent continuity and high values for the second to straight continuity. Plots are given in Figs. 13 and 14.

All slip systems being equally favoured, it is assumed that each is capable of giving rise to pile-ups at the boundary. It is seen that the best bent continuity would be expected at around  $40^\circ$  misorientation, and good straight continuity around  $130^\circ$ . Comparison of Figs. 13 and 14 with Table 3 shows there to be little correlation between these predictions and the observed continuity. For example, bicrystal B with misorientation  $23^\circ$  should give better



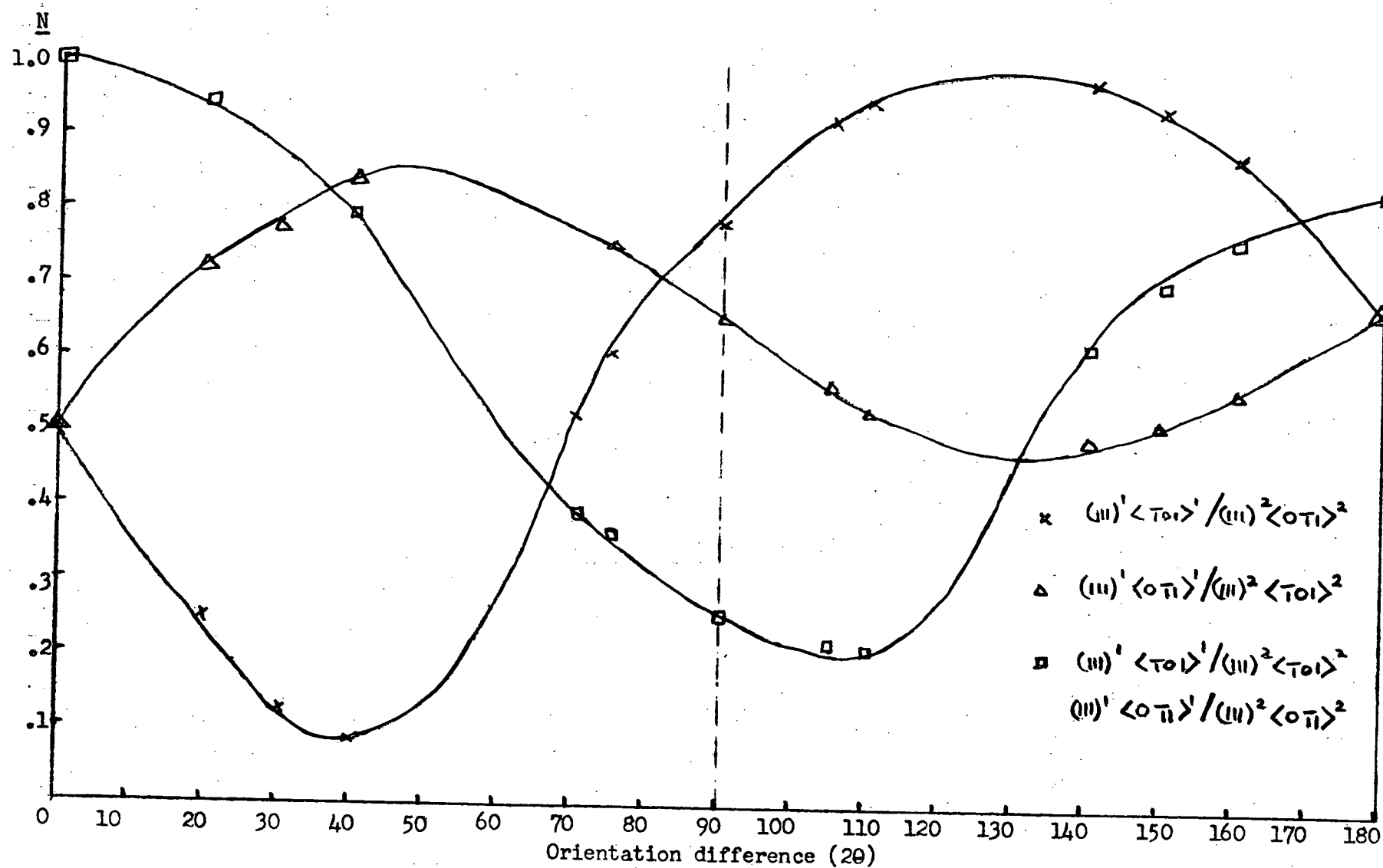


Fig. 13. Values of  $N$  for bent continuity.

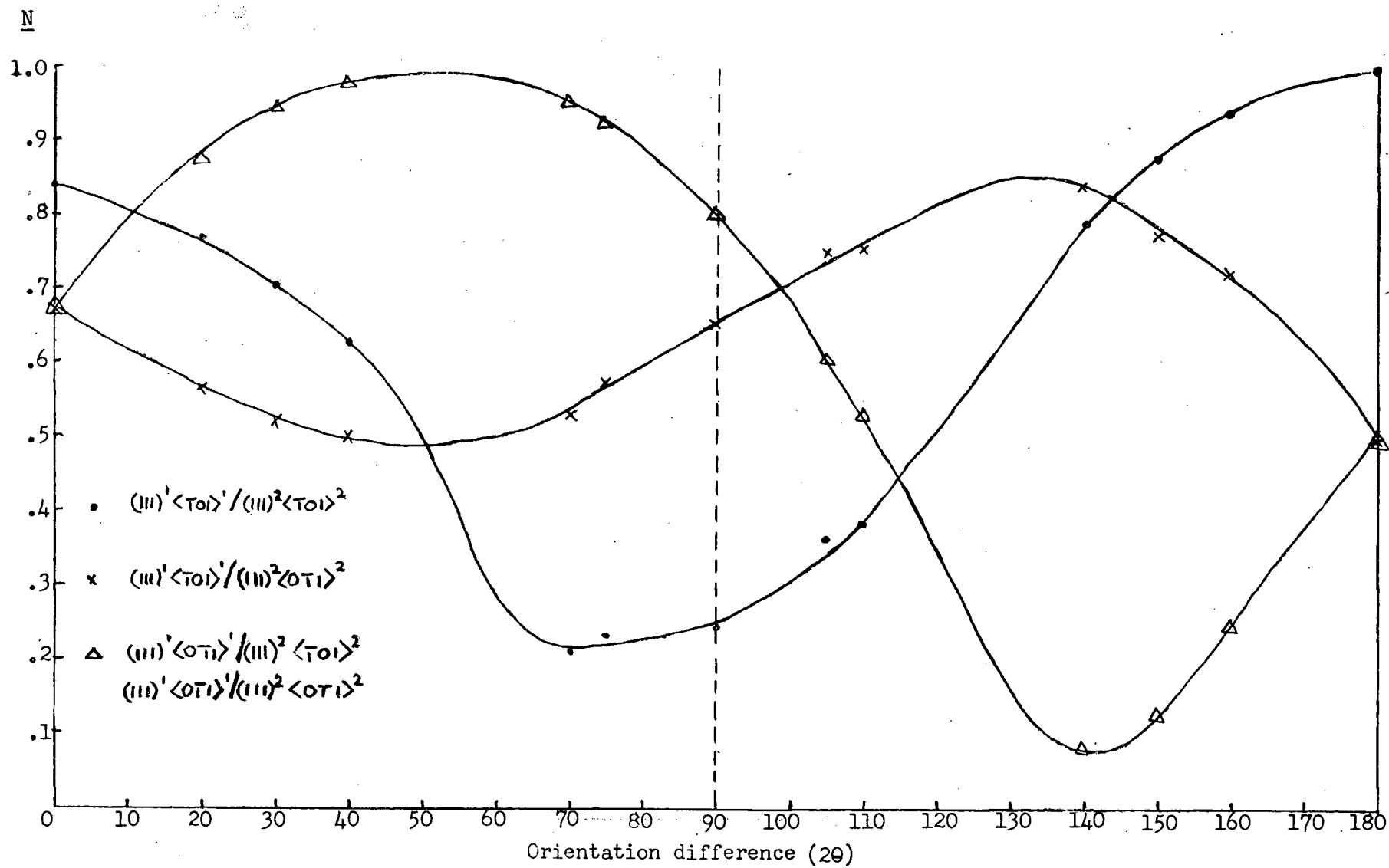


Fig. 14. Values of N for straight continuity.

continuity than bicrystal E with  $73^\circ$ ; the opposite is observed. The dislocation source activation idea appears invalid..

(b) An examination of the possible movement of dislocations through the boundary.

If dislocations are to pass through the boundary, three criteria would appear to be controlling:

- 1) the edge or screw character of the dislocations.
- 2) the nature of the boundary.
- 3) the angle  $\phi$  through which the dislocation must turn at the boundary (see Fig. 12), which is the angle between the lines of intersection of the active slip planes in the two crystals with the boundary plane.  $\phi$  will in future be referred to as 'the angle of dislocation twist' at the boundary.

Consider first the symmetric tilt bicrystals. They showed (1) best bent continuity at a misorientation angle around  $70^\circ$ , and (2) straight continuity only in bicrystals H with misorientation  $150^\circ$ , and then not very clearly. For bent continuity, the angle of dislocation twist is zero. It is instructive to investigate the nature of the dislocations approaching the boundary. Table 4 gives the angle  $\alpha$  between the slip direction and the boundary plane. Small values for  $\alpha$  mean that the slip direction is nearly parallel to the boundary plane, i.e. the dislocations coming up to the boundary are screw dislocations. Comparison of Tables 3 and 4 indicate that, except for the low angle boundary bicrystal A, good slip band continuity corresponds to low values for  $\alpha$ . Screw dislocations appear able to traverse the boundary better than edges.

Table 4.

The angle  $\alpha$  between slip direction and the boundary plane for the symmetric tilt boundary.

Misorientations	$\alpha^\circ$	
0	30	30
10	26	34
20	22	38
23(Bicrystal B)	20	40
30	17	42
50	8	49
70	0	55
90	8	59
110	17	63
114	19	
130	26	58
150	34	54

For criterion 3), values of the angle of dislocation twist at the boundary  $\phi$  have been read from a stereographic plot. The results are given in Table 5.

Table 5

Angle of dislocation twist  $\phi$  (for straight continuity only in the symmetric tilt bicrystals).

Symmetric tilt bicrystals orientation difference	$\phi^\circ$
0	70
24	68
40	66
60	62
80	54
100	48
120	38
140	28
150	20
18° twist boundary bicrystal	18
bicrystal Q1	78
" Q2	38
tricrystal 1 (13°)	1
" 2 (39°)	3

The symmetric bicrystals H (misorientation 150°) showed some slight

straight continuity, whereas the twist boundary bicrystal showed none. A critical  $\phi$ -value for continuous slip between  $15^\circ$  and  $20^\circ$  would appear likely. The remarkably small  $\phi$ -values in the tricrystals should be noted.

Tricrystal 2, where the outside crystals, tilted  $39^\circ$ , have slip nearly parallel to the boundary, showed exceptionally good continuity, rather better than tricrystal 1. It compared well with bicrystal E, where the seeds had nearly the same orientations as the outside crystals of tricrystal 2. The excellent continuity in bicrystal E is not, therefore, actuated by any peculiarity in its boundary misorientation of  $74^\circ$ , but simply by the screw character of the dislocations coming up against the boundary. Bicrystal Q1 also has slip direction parallel to the boundary, but continuity of the slip bands is prevented by a high angle of dislocation twist.

##### 5. Theoretical Considerations on the Effect on Continuity of the Angle of Dislocation Twist ( $\phi$ ) at the Boundary.

It has been shown that to pass through a boundary with a twist element a dislocation must take on a stepped form (Fig. 12). What will happen to such a dislocation when it expands into the slip planes of crystal 2? The long segments will probably bow out under the applied shear stress, and the short segments will act as drags on the movement. It is of interest to consider how strong this drag will be.

The situation is analogous to that for the movement of a jogged dislocation. Annealed aluminum will have the order of  $10^6 - 10^7$  dislocations per square centimeter. When one centimeter length of a dislocation moves one centimeter forward, it must pick up  $10^6 - 10^7$  jogs, i.e. one jog per  $2\frac{1}{2} - 25$  a where a is the lattice parameter. If  $\phi$  is as before the angle of dislocation twist at the boundary, and the steps produced join neighbouring

slip planes, then the distance  $d$  between these steps will be given approximately by  $d = \frac{a}{2 \sin \phi}$  (see Fig. 15 and Appendix 3).

When $\phi = 5^\circ$	$d = 8 a$
$\phi = 10^\circ$	$d = 4 a$
$\phi = 20^\circ$	$d = 2 a$

It can readily be seen that the number of jogs produced in a dislocation when passing through a low angle boundary will not be much greater than the number it would pick up in a normal slip process in annealed metal. In general, jogs are thought not to produce a large dragging force on dislocations in aluminum, (see for example A. Seeger, "Glide and Work Hardening in Metals", Lake Placid Conference, 1956, p.243). In many cases they can glide along easily with the rest of the dislocation, or can mutually annihilate.

It is possible that slip continuity can be prevented by the extra energy needed to give a stepped dislocation rather than by difficulty in moving such a dislocation once formed. An analysis of energy changes when a dislocation becomes stepped has been carried out in Appendix 3. Insufficient information about the location of the slip sources make it impossible to calculate a maximum  $\phi$ -value above which slip continuity would become impossible. It is, however, quite clear that applied forces can supply sufficient energy to give steps in the dislocation. Step formation can be eliminated as a mechanism for preventing slip continuity unless the slip sources are very near (less than 0.1 inches) from the boundary.

Elastic interactions between the strain fields of the moving slip dislocations and the dislocations in the boundary are likely to give stronger blocking. Dehlinger<sup>24</sup> has shown that such interactions can assume large values. The problem cannot be solved until exact models of boundary structure

have been developed.

6. Theoretical Estimation of the Frequency of Continuous Slip Across Boundaries in a Polycrystal.

Observations on polycrystal aluminum show that slip bands are continuous across a few percent of the grain boundaries. What proportion would be crossed if the maximum angle of dislocation twist  $\phi$  is taken as  $15^\circ$ ? Suppose that there is only one slip plane active in each crystal near the boundary. Then the problem is a very simple one. Take the intersection of the slip plane in crystal 1 with the boundary as a fixed arbitrary line. In a random aggregate, the slip plane from crystal 2 is equally likely, to cut the boundary in any line. The probability for a favourable relative orientation of the two grains is (see Fig. 16)  $\frac{30}{180} = \frac{1}{6}$ . With more than one slip plane active the proportion will rise. It has, therefore, been demonstrated that it is not unreasonable to suppose that this condition must be satisfied before continuity can occur.

7.. Further Evidence for the Transparency of Crystal Boundaries to Screw Dislocations.

Screw dislocations can easily change their plane of slip. Consequently, straight slip bands give way to wavy bands when slip is carried out by the movement of dislocations with nearly pure screw character. Examine Micro 16. It will be observed that the prominent slip band in the lower crystal is straight as far as the boundary. Once across the boundary it peters out into a wavy slip trace before joining a slip band in the second crystal. The slip across the boundary is not always wavy, as can be seen from Micros 15, 17, but this need cause no discrepancy in the argument that only screws penetrate. A screw dislocation will change its slip plane only if some barrier

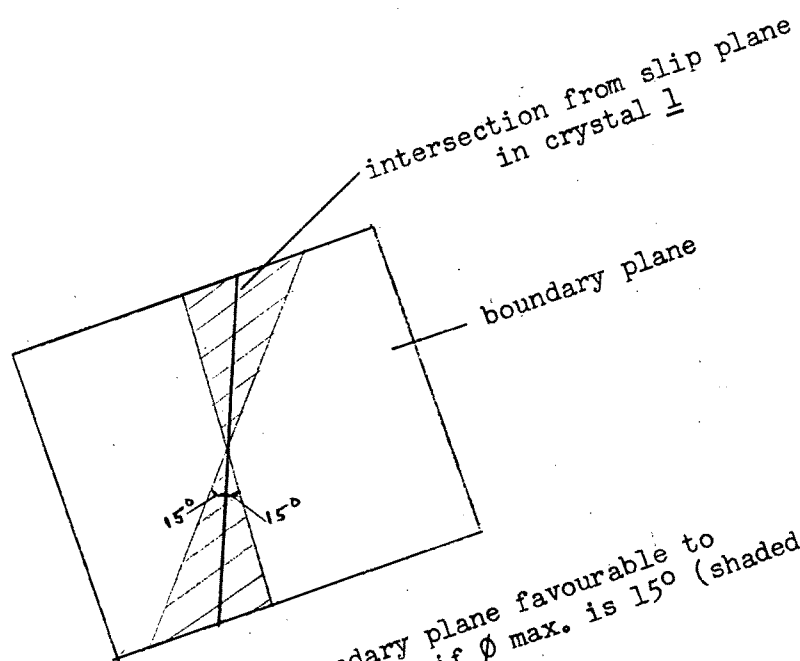


Fig. 16. Area of the boundary plane favourable to slip band continuity if  $\phi$  max. is  $15^\circ$  (shaded region).

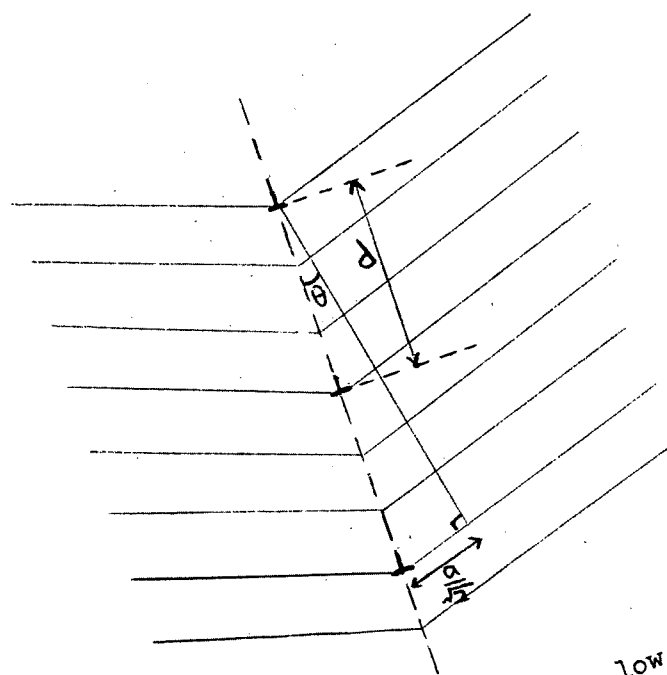


Fig. 17. Dislocation structure for a low angle tilt boundary.



to motion in the slip plane is encountered, such as a second active slip system near the boundary.

Micros 21, 22, 23, and 24 illustrate the prevalence of wavy slip in the boundary region, even when there is no continuity. In all these pictures the dislocations approaching the boundary will be of nearly pure screw orientation, but similar observations have been made on other bicrystals. Micro 23 makes it quite clear that the dislocations do not reach the boundary before being held up and so caused to cross slip. As soon as the slip bands reach the region of double slip near the boundary, short wavy lines start to branch out. At the boundary there is a very large obstacle to slip, and screw dislocations escape from the slip plane in large numbers giving rise to the short region of slip along the boundary seen in Micros 23, 24, and 25. In Micro 25 one of these slip bands parallel to the axis of stress occurs across the boundary from its generating slip band.

Micro 26 shows the effect of the boundary in the case where the slip direction is nearly parallel to the polished surface. Wavy slip is observed over quite a wide region around the boundary. The slip bands seen on a single crystal of such a specimen would be caused by dislocations of nearly screw orientation. Slip far from the boundary takes the normal form of closely-spaced bands, and it can be assumed that, to get wavy slip, dislocations of pure screw orientation are needed. In bicrystals of type Q2 there is a very large size effect and a large amount of double slip will be present at the boundary. This accounts for the wide regions over which screw dislocations are released by cross-slip.

Perhaps the best evidence for the generation of screw dislocations at boundaries comes from Micro 20, where the slip bands bend round to join slip bands in the neighbouring crystal after passing through the boundary.

The dislocations causing these bent slip bands cannot lie on one slip plane, and must therefore be screw dislocations.

To sum up, the properties of dislocations show that the movement of screw dislocations will lead to wavy slip. This is supported by the available evidence and there is no reason to doubt its validity. Observations of wavy and bent slip at the boundary support the view that those dislocations penetrating the boundary are pure screws.

#### 8. Reasons Why Screw Dislocations should Pass More Easily Through Grain Boundaries than do Edges or Mixed Dislocations.

For a low angle tilt boundary the position is fairly clear (see Fig. 17). An edge or mixed dislocation coming down its slip plane towards the boundary will enter the strain field of the boundary dislocations. It will pass through the boundary under a minimum applied stress when its slip plane bisects the space  $d$  between neighbouring boundary dislocations. The distance between such dislocations is given by:

$$\sin \theta = \frac{a}{\sqrt{2} \cdot 2d}$$

where  $a$  = lattice parameter

$2 \theta$  = boundary misorientation

The number of slip planes cutting the boundary in distance  $d$ ,

$$n = \frac{d}{\text{distance between the slip planes}}$$

$$= \frac{\sqrt{3}}{a} \frac{d}{1} = \frac{1/4 \sqrt{6}}{\sin \theta}$$

For misorientation $2 \theta =$	$5^\circ$	$10^\circ$	$15^\circ$	$20^\circ$
$n =$	14	7.2	4.7	3.6

For low angle boundaries,  $5^\circ$  or below, most dislocations will penetrate the boundary.

Consider now the situation for a screw dislocation. This can easily change its slip plane. The easiest path through the boundary will be in effect a valley in the energy field set up by reactions between the stress fields of the moving screw and the boundary dislocations; the dislocation will follow this valley. A screw dislocation will therefore always be in a favourable slip plane to penetrate the boundary.

For boundaries with misorientation above  $20^\circ$  the dislocation model is no longer satisfactory. A model for grain boundary structure used frequently is Mott's suggested 'islands' of good fit in a 'sea' of bad fit.<sup>25</sup> To this model similar arguments to those just put forward can be applied. The islands of perfect lattice will be easy to penetrate, and the screw dislocations will seek them out. The island must be large enough to allow a stable loop of dislocation to belly out through it. Having no way for estimating the diameter of the islands with a sufficient degree of accuracy this condition cannot be tested. The mechanism envisaged is that a screw dislocation moves up to the boundary and positions itself to be across the diameter of several of the islands of perfect fit. At such points it will pass into the next crystal and spread out on the slip plane, giving a segmented slip band. This is perhaps connected with the observation that where continuity is good the slip bands near the boundary tend to be wide.

## 9. Conclusions

- (a) Slip band continuity is a true bulk effect and is not confined to the surface.
- (b) Work hardening appears to be lowered by good slip band continuity.

(c) Dislocation source activation theory is incompatible with the results obtained here.

(d) Factors favouring continuity are:

- 1) a low angle boundary
- 2) a small angle of dislocation twist at the boundary, a limiting value of  $\phi_{\max} = 15 - 20^\circ$  being indicated.
- 3) slip direction nearly parallel to the boundary, i.e. screw dislocations pushed at the boundary.

(e) Detailed metallographic study of deformation markings at the boundary support the idea that screw dislocations are released by cross-slip near the boundary and that screw dislocations pass through the boundary.

## APPENDIX I

### Electropolishing Large Areas of Aluminum.

#### Introduction

There are many descriptions in the literature<sup>26</sup> of electropolishes for aluminum. Most of these refer to procedures for the preparation of specimens with small surface area for micrographic examination. Electropolishing large areas presents much greater problems in heat dissipation and electrolyte-flow.

#### Experimental details.

The following polishing solutions were tried:

Alcoa bright dip (chemical polish)

Perchloric acid-ethyl alcohol mixtures.

A phosphoric acid electrolyte

1 part nitric acid to four parts methyl alcohol.

15% perchloric acid in acetic acid.

Most electropolishes are effective only in a restricted temperature range, usually below 20°C. Efficient cooling is therefore essential. For these aluminum polishes, surrounding the bath with ice was generally ineffective (5 amps at 50 volts puts in enough energy to melt 12 grams of ice per second). In practice, the most successful arrangement utilised a stainless steel beaker as cathode, surrounded by cold running water, the liquid in the beaker being stirred. A glass beaker and aluminum cathode were sometimes used where cooling was less critical.

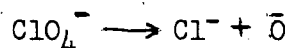
# Results.

Electrolyte	Current Density	Temperature	Quality of Finish
Alcoa bright dip	-	90-110°C	Good general surfaces with pitted areas, striations vertical in bath. Rather inconsistent.
70% orthophosphoric <sup>27, 28</sup> 2.5% water 26.51% "carbitol" 1% HF	0.3-0.5 amps/cm <sup>2</sup>	not critical 20-70°C	Some pitting. Tendency to get white deposit on the surface.
20% perchloric acid in ethyl alcohol	0.5-1.0 amps/cm <sup>2</sup>	10-18°C	Quite good in some cases. Greater C.D., better polish. See next section for details.
1 part HNO <sub>3</sub> <sup>29</sup> 4 parts CH <sub>3</sub> OH	0.5-0.7 amps/cm <sup>2</sup>	10-20°C	Good at low magnification. Some roughness shown up at x600 magnification and above.
15% perchloric in acetic acid	0.1-0.3 amps/cm <sup>2</sup>	14-20°C.	Good. Slower acting than the previous electrolytes.

## Perchloric acid-ethyl alcohol solutions.

These are the most widely used electropolishes on aluminum. For large surfaces, however, they have certain disadvantages. The best polish is obtained at high current densities, but this is accompanied by rapid heating of the solution. With the present arrangement, runs are limited to 30-45 seconds (this applies also to the HNO<sub>3</sub>-CH<sub>3</sub>OH electrolyte). A further drawback is a black deposit that frequently forms over part of the surface, under which there is no polish. This happens most frequently in solutions with 20% strength (S.G. 1.25) perchloric acid. A solution with concentrated (60%) perchloric never gave the deposit, but areas of severe pitting developed. In both solutions the trouble was minimised by obtaining a smooth flow of

electrolyte past the surface, but results were far from consistent. The following explanation has been put forward.<sup>30</sup> Turbulence at the surface leads to local overheating, and the following reactions:



With the strongest acid, the carbon deposit is further oxidised to CO, CO<sub>2</sub> leaving a pitted area. This suggestion is backed by the following observations:

- (1) Al or Al oxide can act as a strong catalyst for such reactions.
- (2) The HNO<sub>3</sub> CH<sub>3</sub>OH electrolyte can give a similar deposit if allowed to overheat.
- (3) At the same C.D., the specimen heats up very much faster in the electrolyte with the 60% perchloric than in that with the 20% perchloric. This is attributed to heat evolved at the specimen surface while the carbon is oxidised off.

#### An electropolish for rapid removal of metal

When it is desired to remove layers of aluminum several microns thick, e.g. to remove surface working, a 15% perchloric acid in ethyl alcohol solution is recommended. Temperature must be kept below 20°C for a polish. There must be no stirring.

#### Summary.

For electropolishing large areas of aluminum, the most consistent of the solutions tried is 15% perchloric acid in acetic acid. The polishing action is slow, needing 1-1 1/2 hours for a good finish. Grain boundaries are delineated by thin lines.

## APPENDIX II

### The Shear Stresses Acting on Slip Planes Ahead of a Pile-up of Dislocations.

The mathematics of the procedure to be described were developed by A.E. Love<sup>31</sup>; it has been applied to slip in metals in a paper by Davis et al.<sup>32</sup> The basic equation is:

$$P_i = P_N = P [(\underline{e}_i \cdot \underline{e}_1 \times \underline{l}_i \cdot \underline{l}_1) (\underline{e}_1 \cdot \underline{l}_i \times \underline{e}_i \cdot \underline{l}_1)],$$

where  $P_i$  is the shear stress on slip systems  $i$ , caused by an applied shear stress  $P$  on slip systems  $1$ . The vectors are unit vectors, such that their dot products are cosines.

$\underline{e}_i, \underline{e}_1$  are normal to the slip planes  $i$ ,

$\underline{l}_i, \underline{l}_1$  are slip directions.

Values of  $N$  are derived, with  $N = [\cos(\text{angle between slip plane normals}) \times \cos(\text{angle between slip directions})] + [\cos(\text{angle between normal to slip plane } 1 \text{ and slip direction } i) \times \cos(\text{angle between normal to plane } i \text{ and slip direction } 1)]$ .

For each bicrystal, a stereogram is plotted giving the slip planes and directions for both crystals. Angles between all active slip directions and slip plane normals are read off from the stereogram. For each slip system in crystal  $1$ ,  $N$ -values are found for all the slip systems in crystal  $2$ . If slip band continuity is caused by slip source activation across the boundary, high  $N$ -values should lead to good continuity. Figs. 13 and 14 show  $N$ -values for symmetric bicrystals with misorientation from  $0^\circ$  to  $180^\circ$ .

Each crystal has 12 slip systems. In the general case 144  $N$ -values would have to be calculated. The bicrystals grown for this investigation, however, had crystals with their  $\langle 110 \rangle$  axes parallel to the stress axis, and the applied stress will give appreciable shear stresses only on two slip



planes per crystal, each plane having two possible slip directions. There are therefore only four slip systems per crystal that need be considered, greatly reducing the labour involved.

### APPENDIX III

#### An Analysis of the Energy Considerations in the Formation of a Stepped Dislocation at a Twist Boundary.

Data for aluminum:<sup>33</sup>

shear modulus $\mu$	$= 2.7 \times 10^8 \text{ gms/cm.}^2$
critical shear stress $\sigma_0$	$= 5 \times 10^3 \text{ gms/cm.}^2$
energy for a jog in an edge dislocation	$= 0.4 \text{ e.v.}$
energy for a jog in a screw dislocation	$= 2/3 \times .4 \text{ e.v.}$
lattice parameter $a$	$= 2.5 \text{ \AA}$

A low angle twist boundary can be represented by dislocations with screw components. When a slip dislocation moves through such a boundary it will receive jogs where it cuts the boundary dislocations; the screw component of these dislocations will lead to those parts of the jogs that lie in the boundary plane. For simplicity, assume that the jogs are equally spaced along the slip dislocation and perpendicular to its line. Jogs will form that have a minimum energy, and those that join the slip planes in the shortest lines will be favoured. There are four slip planes in aluminum, and there will, except in special cases, be three possible jog directions. An angle fairly near to  $90^\circ$  between jog lines and the dislocation line is therefore likely. (The boundary grown had a  $\{100\}$  boundary plane; the dislocation line and the jogs will therefore be exactly perpendicular).

On this model (see Fig. 15),  $\frac{a}{\sqrt{2}} = d \sin \phi$

$$d = \frac{a}{\sqrt{2} \sin \phi}$$

for $\phi = 5^\circ$	$d = 8 a$
$\phi = 10^\circ$	$d = 4 a$
$\phi = 15^\circ$	$d = 2.7 a$
$\phi = 20^\circ$	$d = 2 a$
$\phi = 25^\circ$	$d = 1.7 a$
$\phi = 30^\circ$	$d = 1.4 a$

Variations from  $90^\circ$  in the angle between slip direction and jog will have little effect on  $d$  until  $d$  becomes greater than  $2 a$  (e.g. if this angle is  $70^\circ$ , at  $\phi = 20^\circ$   $d = 1.98 a$ ).

It is desirable to calculate the shear stresses needed to give the jog densities indicated. To do this the microscopic details of the process must be considered. The mechanism envisaged is illustrated in Fig. 18.

Increase in energy in the formation of each step in a dislocation with Burger's vector  $b$  is given by:

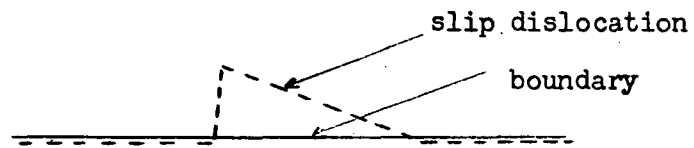
$$\begin{aligned} \Delta E &= \text{the energy for a jog} - \mu b^2 d (1 - \cos \phi) \\ \text{for } \phi &= 20^\circ, \mu b^2 d (1 - \cos \phi) = \mu b^2 d 0.06 \\ &= 0.06 \text{ e.v.} \end{aligned}$$

This is negligible compared to the energy for a jog ( $\sim 0.4$  e.v.) in the accuracy of these calculations. For all  $\phi$ -values considered,  $\Delta E \neq 0.4$  e.v.

Work done by the dislocation in forming each jog

$$\begin{aligned} &= \text{force} \times \text{distance} \\ &= 1/2 \text{ force} \times d \sin \phi \\ &= 1/2 \sigma b d \sin \phi \end{aligned}$$

$$\begin{aligned} \text{where } \sigma &= \text{shear stress at the dislocation} \\ &= N \sigma_0 \end{aligned}$$



(a) Formation of one step.



(b) Dislocation with the full number of jogs.

Fig. 18. The passage of a dislocation through a boundary.

with  $N$  = number of dislocations in a pile-up tending to push the dislocation through the boundary.

Now equating energy needed to cause the stepped dislocation with the work done in its formation,

$$\Delta E = \frac{1}{2\sqrt{2}} \sigma_0 N a d^2 \sin \phi$$

$$.4 \times 1.6 \times 10^{-12} = \frac{N \times 50 \times 10^6 \times 2.5^3 \times 10^{-24} \times K^2 \sin \phi}{2\sqrt{2}} \text{ ergs}$$

where  $d = Ka$

$$\text{now } N = \frac{2.3 \times 10^3}{K^2 \sin \phi}$$

for $\phi = 25^\circ$ ,	$K = 1.7$	$N = 1.9 \times 10^3$
$\phi = 20^\circ$ ,	$K = 2$	$N = 1.7 \times 10^3$
$\phi = 10^\circ$ ,	$K = 4$	$N = 0.85 \times 10^3$

These values are probably reliable to  $\pm 50\%$ .

The number of dislocations expected in a pile-up under shear stress  $\sigma_0$  is given by<sup>23</sup>

$$N = \frac{\pi L \sigma_0}{\mu b}$$

where  $L$  = the length of the pile-up.

$$\text{Thus } N = 3.2 \times 10^4 L$$

$$\text{for } L = 1 \text{ cm.} \quad N = 3.2 \times 10^4$$

$$L = \frac{1}{10} \text{ cm.} \quad N = 3.2 \times 10^3$$

Again, accuracy is probably about  $\pm 50\%$ .

When a stress sufficient to give slip lines is applied, pile-ups

of these orders will form at the boundary. Comparison with the N-values needed to give steps in a dislocation line, show that all slip sources distant  $1/10$  cm. or more from the boundary will be able to give these steps.

A factor not yet mentioned is the possibility of thermal fluctuations activating the formation of the steps. The activation would presumably have to push enough of the dislocation through the boundary to enable it to expand out into the second crystal. The critical radius for a dislocation in aluminum is of the order  $10^{-3}$  -  $10^{-4}$  cms.; a large number of steps would therefore have to be activated simultaneously. It is unlikely that thermal activation is a significant factor at room temperature.

BIBLIOGRAPHY

1. R. Von Mises, Z. Agnew. U. Mech., 8, 161, 1928.
2. G. Taylor, "Strains in a Crystalline Aggregate", from "Deformation and Flow of Solids": International Union of Theoretical and Applied Mechanics: Colloquium, Madrid, 1955.
3. J. Bishop and R. Hill, Phil. Mag. 42, 414, 1298, 1951.
4. B. Jaoul, J. Mech. Phys. Solids, 5, 2, 1957.
5. A. Kochendorfer, "Plastische Eigenschaften Von Kristallen", Springer 1941.
6. D. McLean, "Grain Boundaries in Metals", Oxford 1957.
7. D.W. Bainbridge, C.H. Li and E.H. Edwards, Acta Met. 2, 322, 1954.
8. T. Kawada, J. Phys. Soc. of Japan, 6, 363, 485, 1951.
9. J.J. Gilman, Acta Met. 1, 426, 1953.
10. B. Chalmers, Proc. Roy. Soc. A 162, 120, 1937.
11. R. Clark and B. Chalmers, Acta Met. 2, 80, 1954.
12. K.T. Aust and N.K. Chen, Acta Met. 2, 632, 1954.
13. R.L. Fleischer and B. Chalmers, Acta Met., 6, 265, 1958
14. G.J. Ogilvie, J. Inst. Metals 81, 491, 1952-53.
15. P. Lacombe and L. Beaujard, J. Inst. Metals 74, 1, 1948.
16. J.J. Gilman, Trans. A.I.M.E., 212, 783, 1958.
17. V.M. Urie and H.L. Wain, J. Inst. Metals, 81, 153, 1952.
18. T. Kawada, J. Phys. Soc. of Japan 7, 240, 1952.
19. W. Berg, Z. Krist. 89, 286, 1934. Naturwissenschaften 19, 391, 1931.
20. C.S. Barrett, Trans. A.I.M.E. 161, 15, 1945.
21. C.S. Barrett and L.H. Levenson, Trans. A.I.M.E., 137, 76, 1940.
22. G. Wyon and J.W. Marchin, Phil. Mag. 46, 1120, 1955.
23. A.H. Cottrell, "Dislocations and Plastic Flow in Crystals", Oxford 1952, pp. 106, 107
24. U. Dehlinger, Solvay Conference, 415, 1951.
25. N.F. Mott, Proc. Phys. Soc. 60, 391, 1948.

BIBLIOGRAPHY (continued)

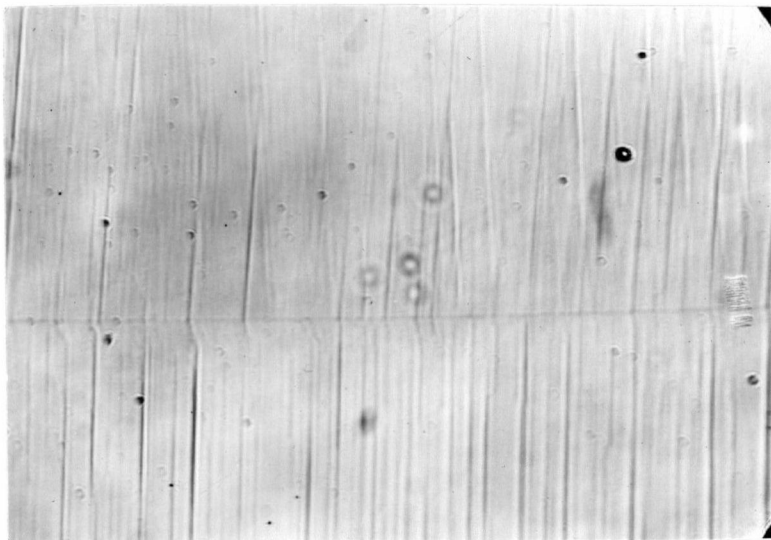
26. P.A. Jaquet, Metallurgical Reviews, 1, 157, 1956.
27. E.C. Pearson, G. Marchand, and R.H. Hay, Canadian Mining and Metallurgical Bulletin, 45, 598, 1952.
28. A. Hone and E.C. Pearson, Metal Progress, 53, 363, 1948.
29. G.E. Pellissier, H. Markus and R.F. Mehl, Metal Progress 38, 554, 1940.
30. D.R. Wiles, Private communication.
31. A.E. Love, "A Treatise on the Mathematical Theory of Elasticity", Cambridge University Press, 1927.
32. R.S. Davis, R.L. Fleischer, J.D. Livingstone and B. Chalmers, Journal of Metals, 7, 136, 1957.
33. A. Seeger, "Report of a Conference on Defects in Crystalline Solids", Physical Society of London, p. 391, 1955.



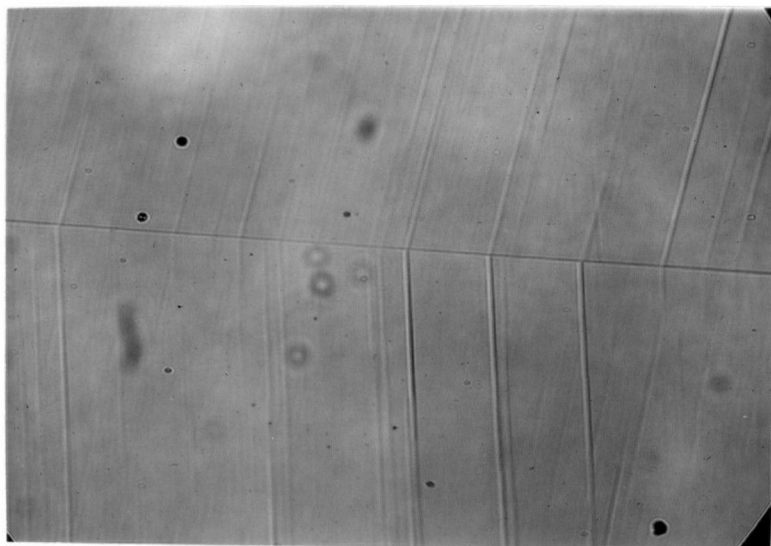
MICROGRAPHS

(The background pattern seen in some of these pictures was caused by interference effects within the metallograph, later corrected).

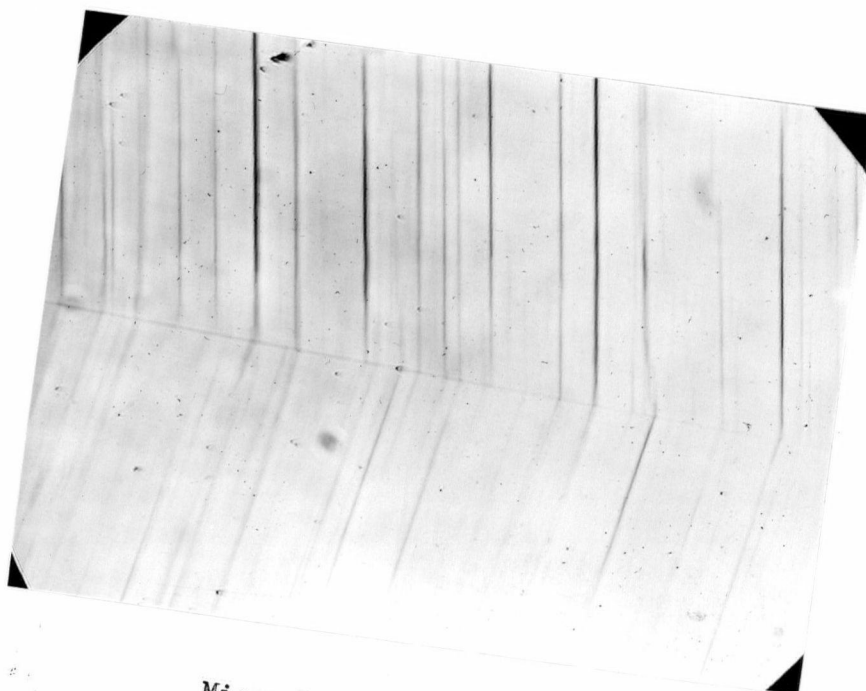
Bent Continuity in Symmetric Tilt Bicrystals. Mag: x100.



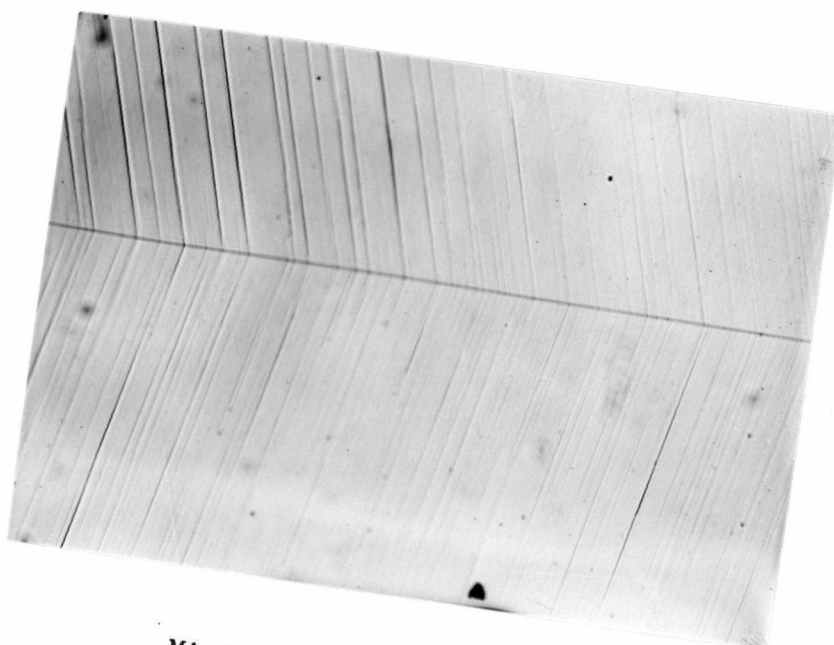
Micro 1. Bicrystal A.  
 $2\theta = 6^\circ$



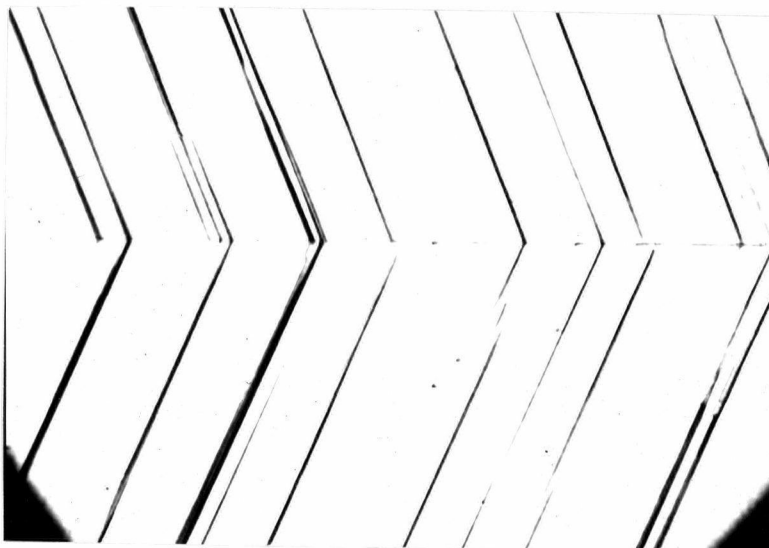
Micro 2. Bicrystal B  
 $2\theta = 23^\circ$



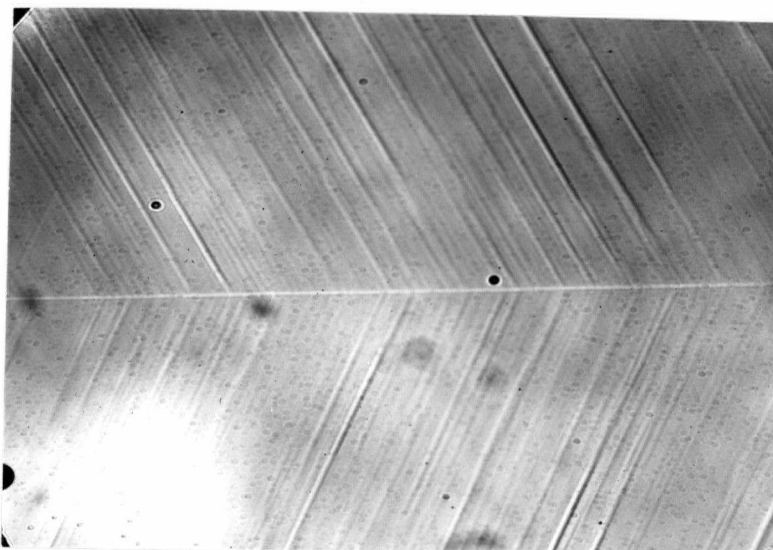
Micro 3. Bicrystal C.  
 $2\theta = 26 \frac{1}{2}^\circ$



Micro 4. Bicrystal D  
 $2\theta = 40^\circ$

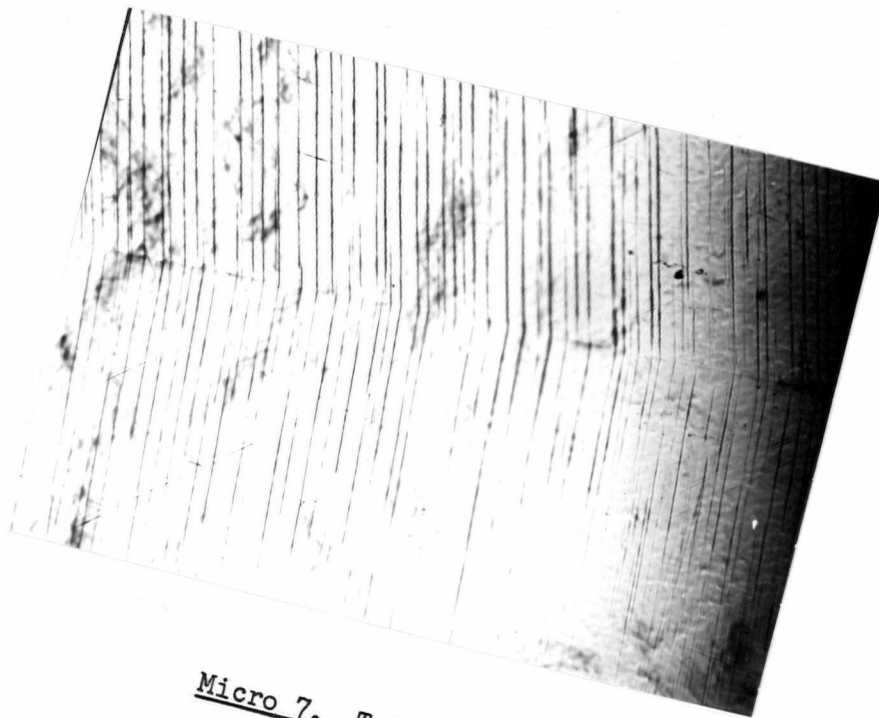


Micro 5. Bicrystal E  
 $2\theta = 73^\circ$



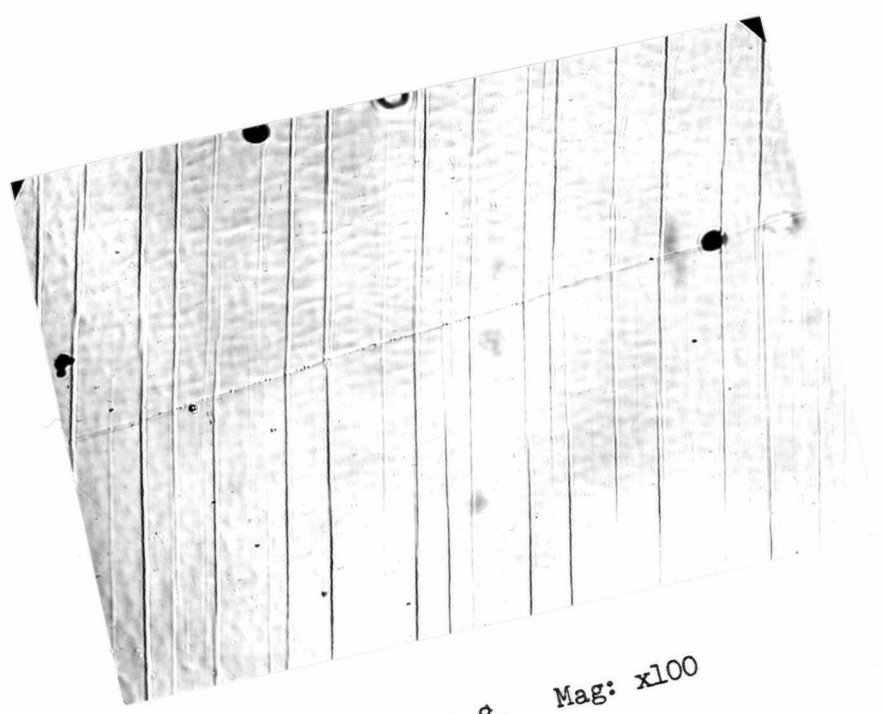
Micro 6. Bicrystal F  
 $2\theta = 93^\circ$

Bent Continuity in a Tricrystal



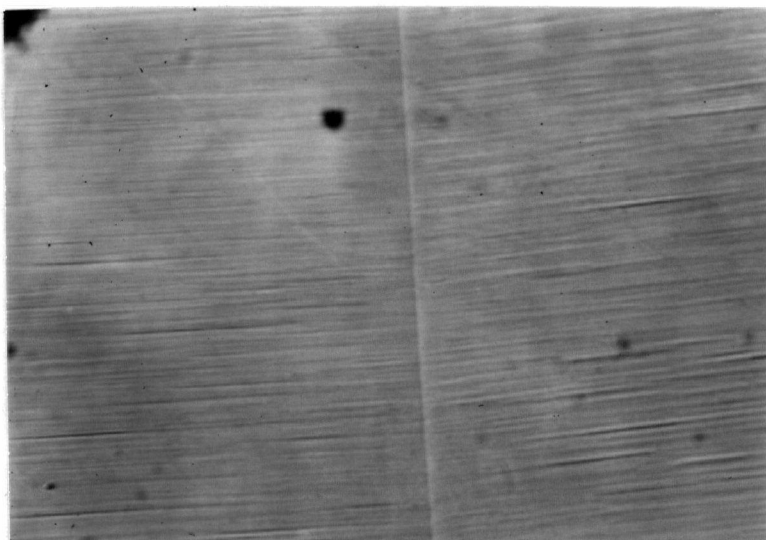
Micro 7. Tricrystal 1  
Mag: x100

Slip Continuous Across a Lineage Boundary



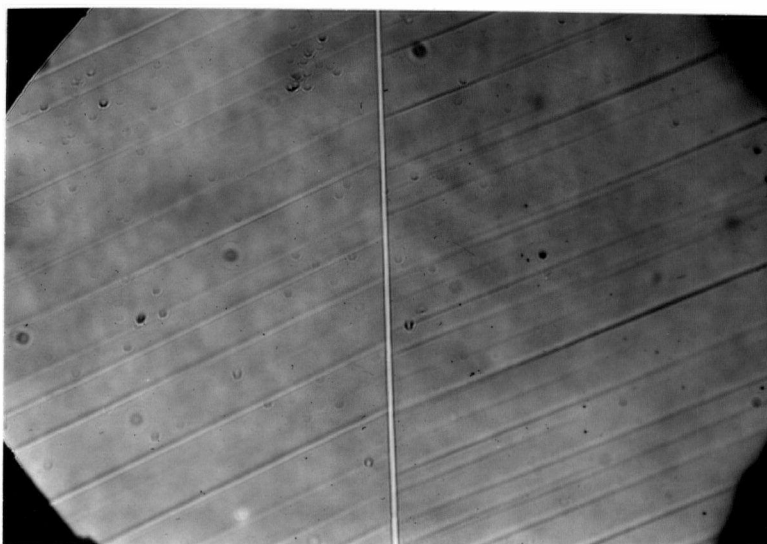
Micro 8. Mag: x100

Non-Continuity Across an  $18^\circ$  Twist Boundary



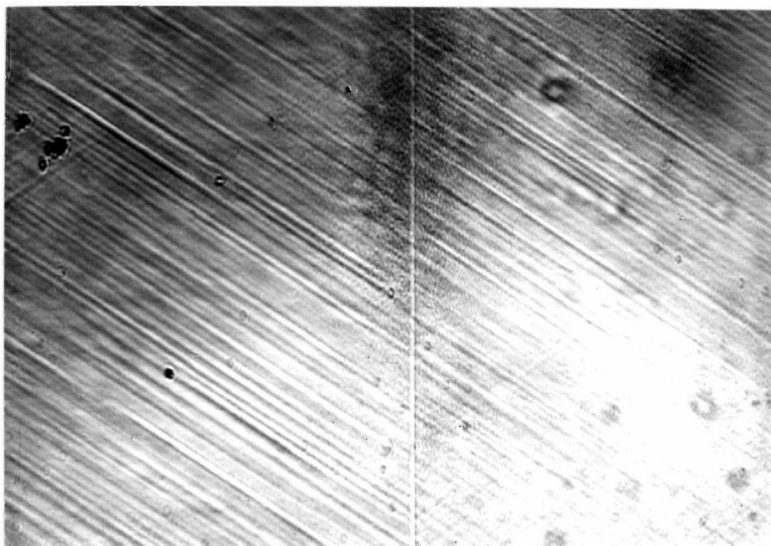
Micro 9. Mag: x100

Absence of Straight Continuity in Bicrystal E.

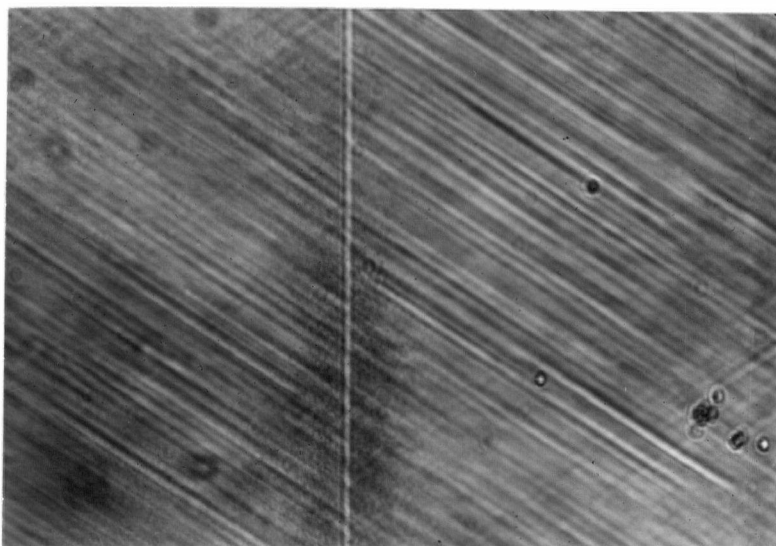


Micro 10. Mag: x100

Straight Continuity in Bicrystal H



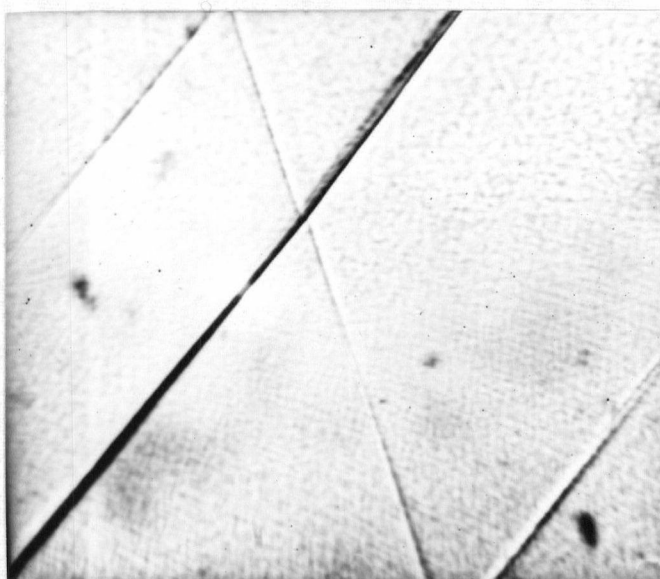
Micro 11. Mag: x100



Micro.12. Mag: x200



Displacement of a Boundary When Crossed by a Slip Band

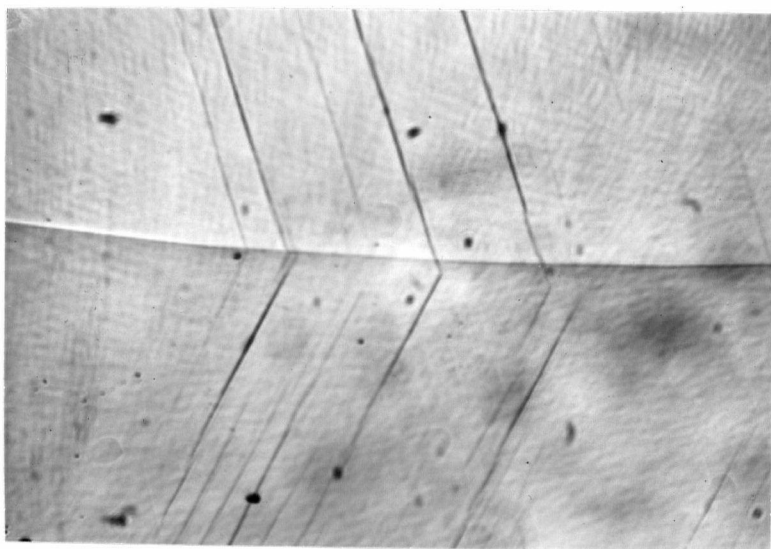


slip  
band

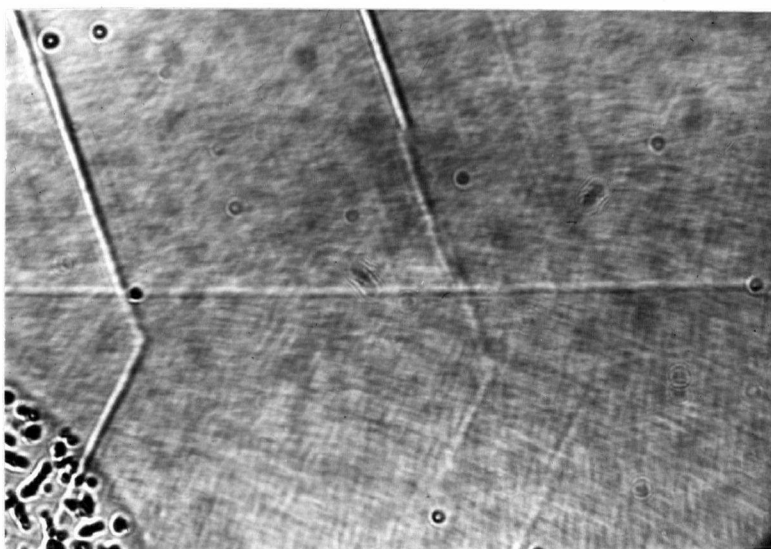
boundary

Micro 13. Mag: x2,500

Slip Bands Crossing a Boundary Straight for  
a short way before giving bent continuity.

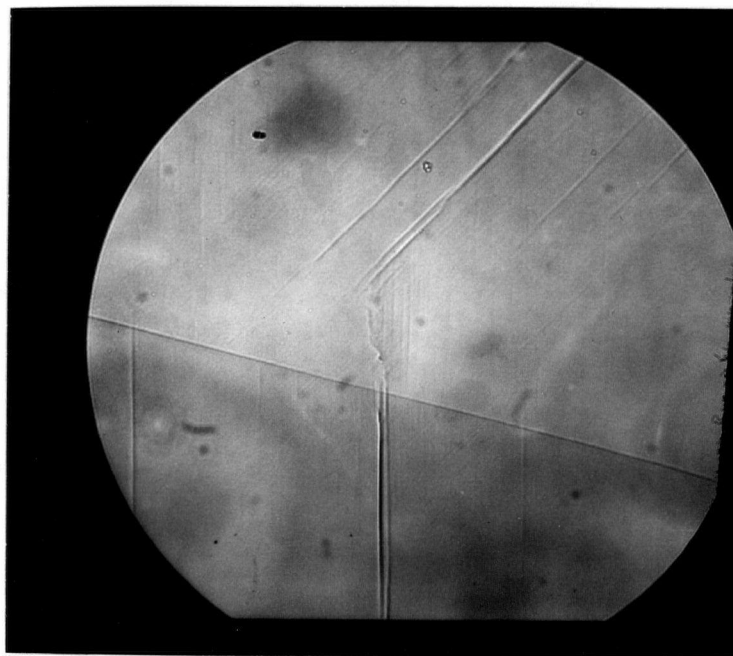


Micro 14. Bicrystal E  
Mag: x100



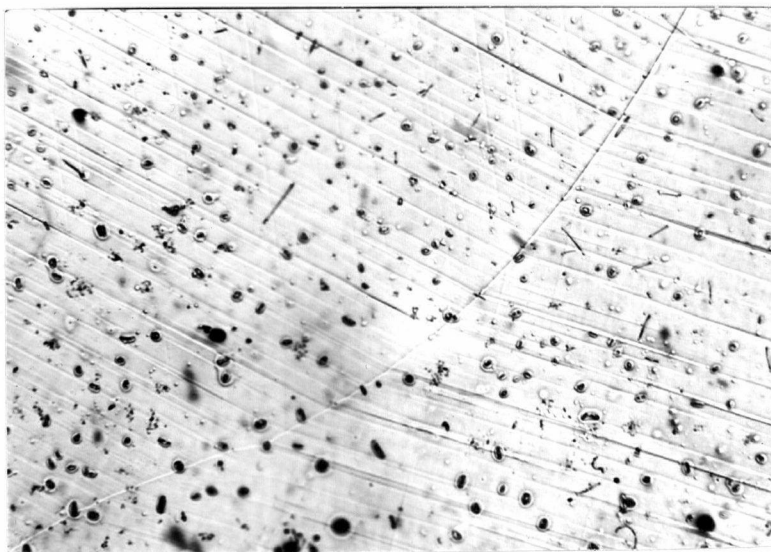
Micro 15. Same as Micro 14  
Mag: x200

Slip Band Becoming Wavy After Crossing the Boundary

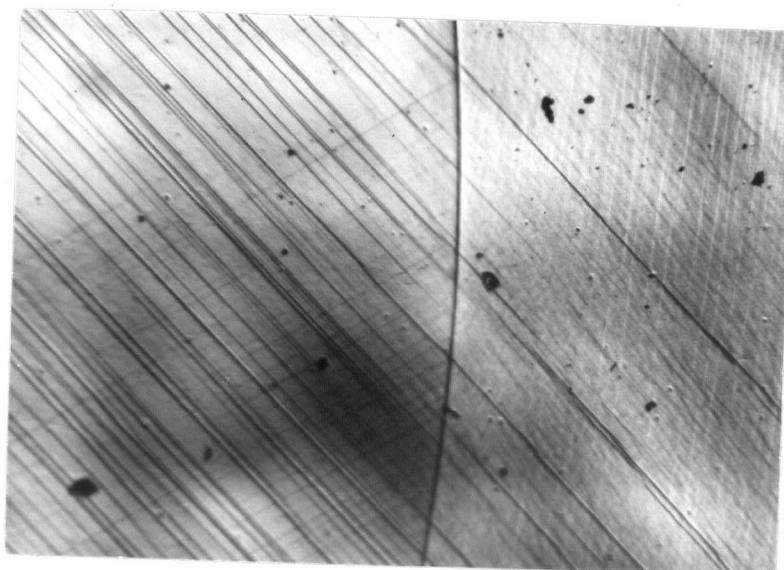


Micro 16. Bicrystal E2  
Mag: x200

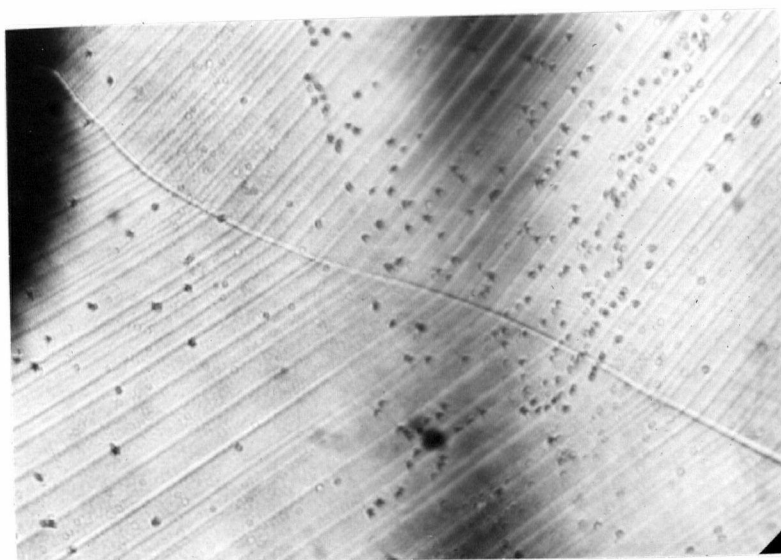
Slip Bands Continuous Through Bent Boundaries



Micro 17. Polycrystal strip  
Mag:xl00

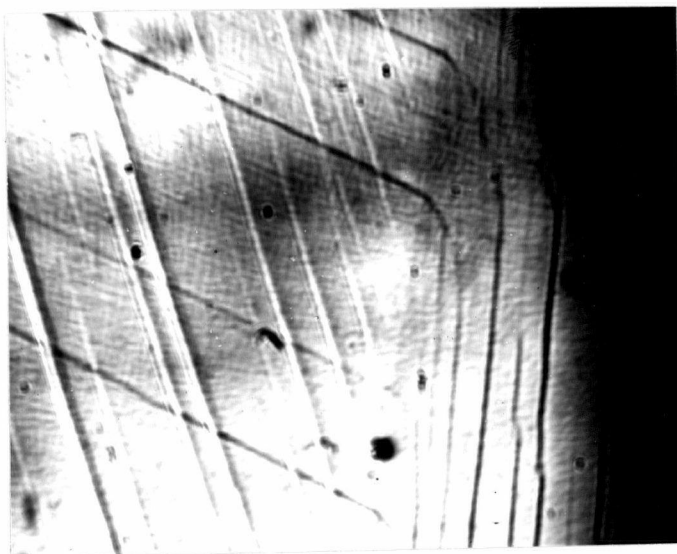


Micro 18. Polycrystal strip.  
Mag:xl00



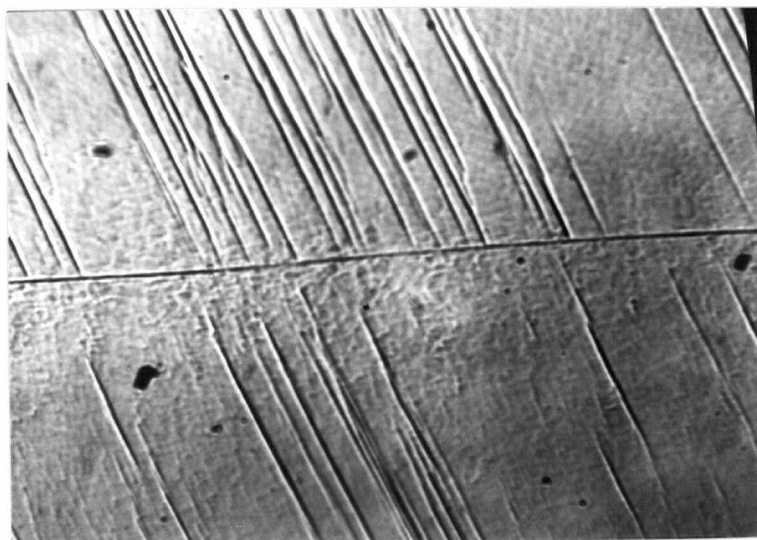
Micro 19. Al - 4% Ag Strip  
Mag: x100

Slip Bands Bent at the Boundary

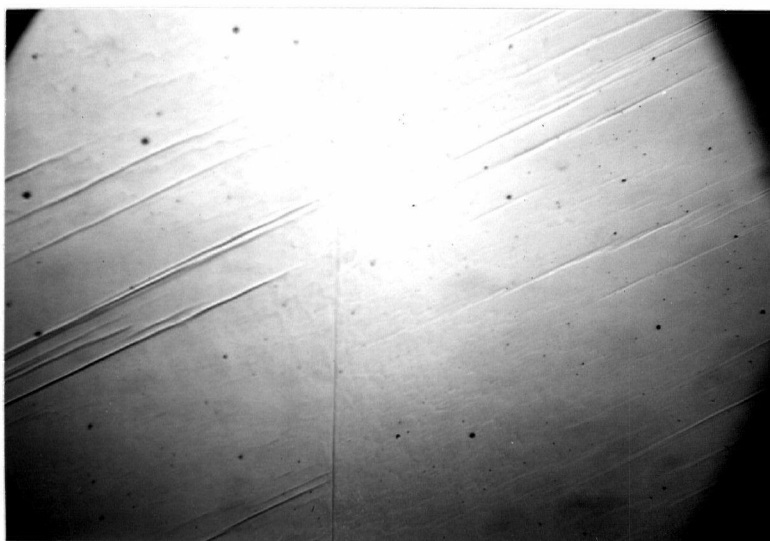


Micro 20. Polycrystal strip  
Mag: x200

Wavy Slip Near the Boundary

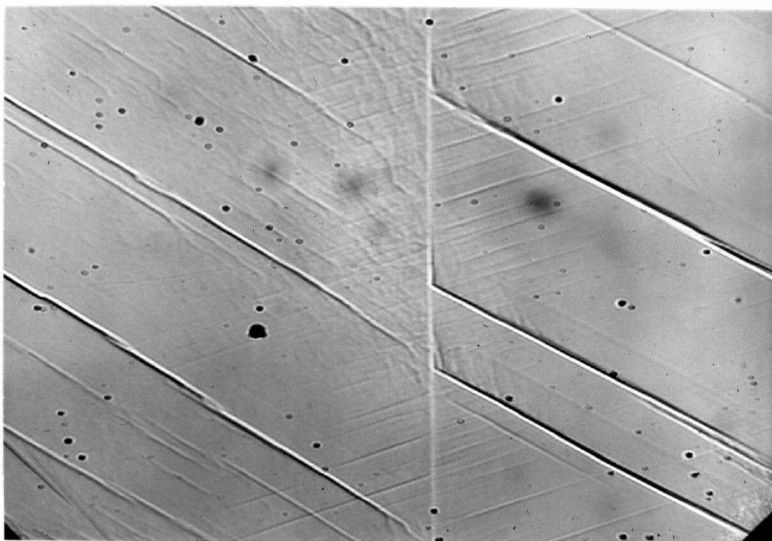


Micro 21. Bicrystal Q1  
Mag: x200



Micro 22. Bicrystal Q1  
Mag: x100

Slip Band Segments Parallel to the Boundary

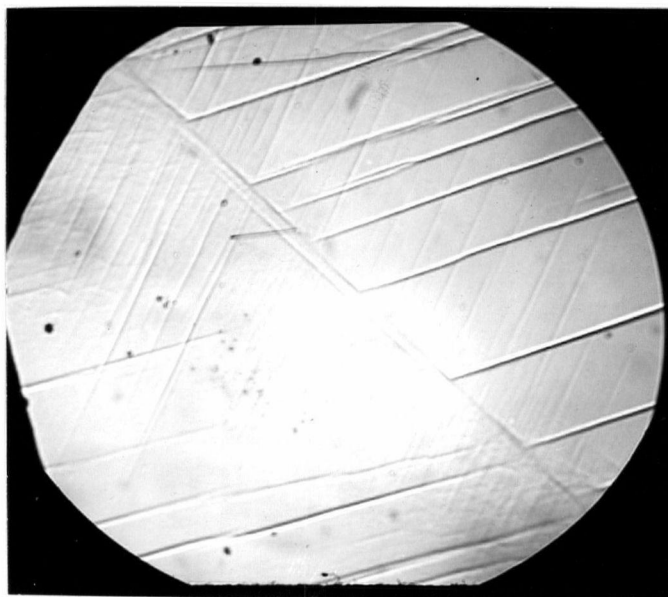


Micro 23. Bicrystal Q1  
Mag: x100



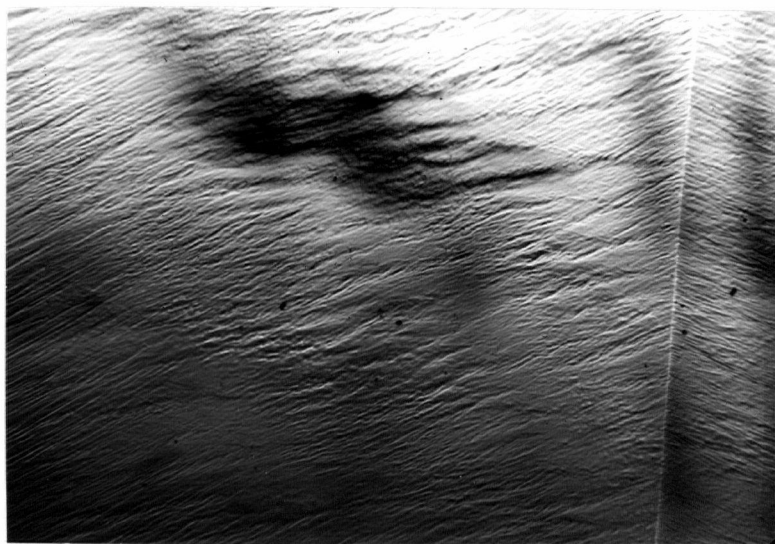
Micro 24. Bicrystal E2  
Mag: x100





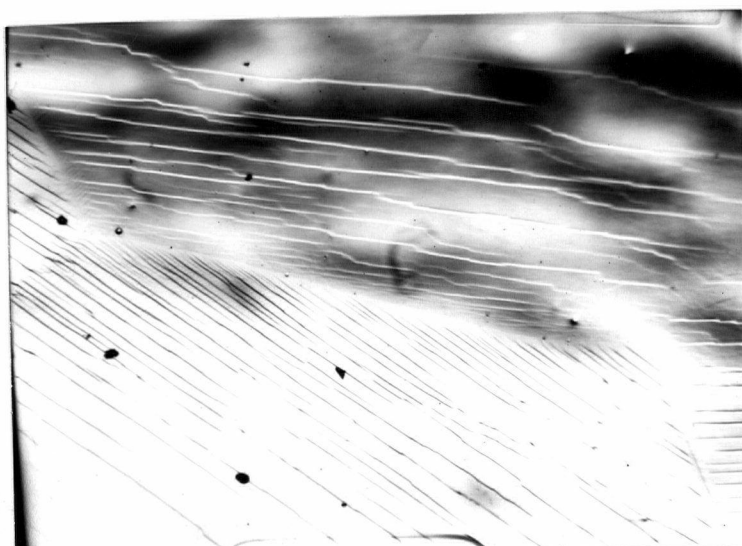
Micro 25. Bicrystal E2  
Mag: x100

Wavy Slip in a Wide Region Near the  
Boundary in Bicrystal G2.

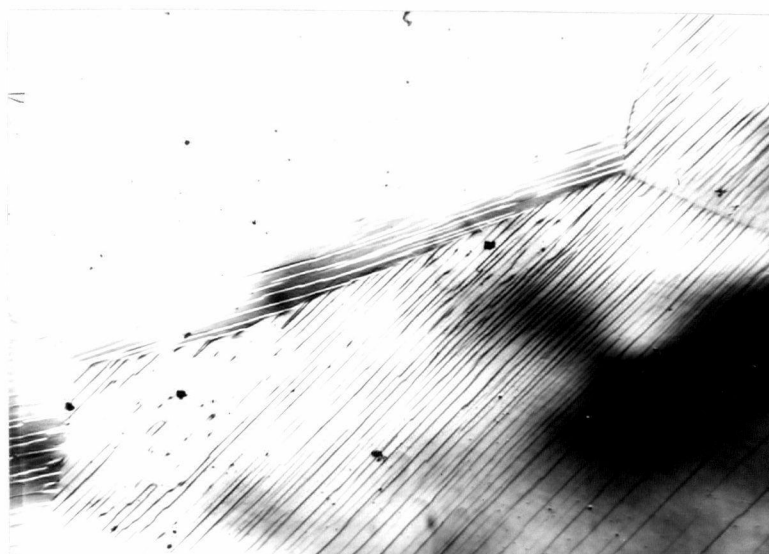


Micro 26. Mag: x30

The Same Boundary Viewed from Opposite Faces of Thin Strip

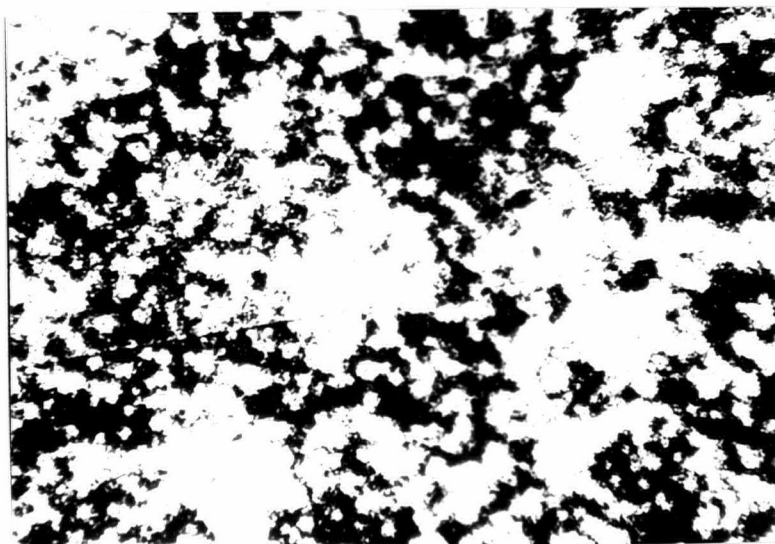


Micro 27. Mag: x100

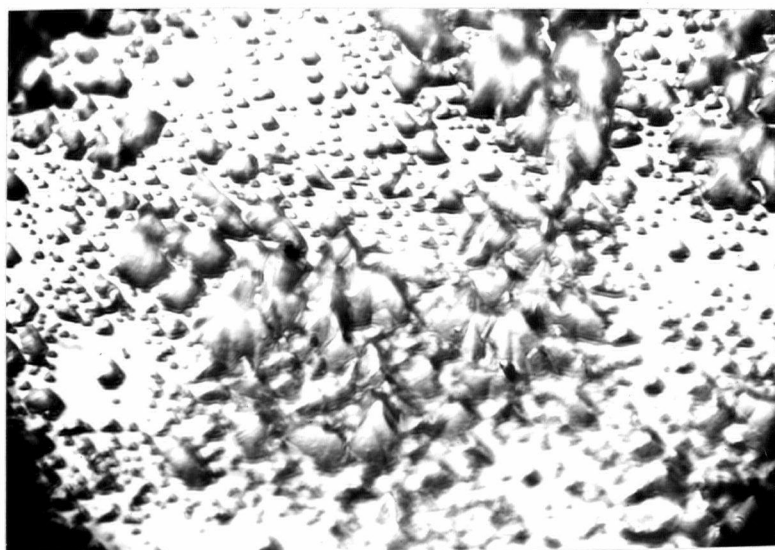


Micro 28. Mag: x100

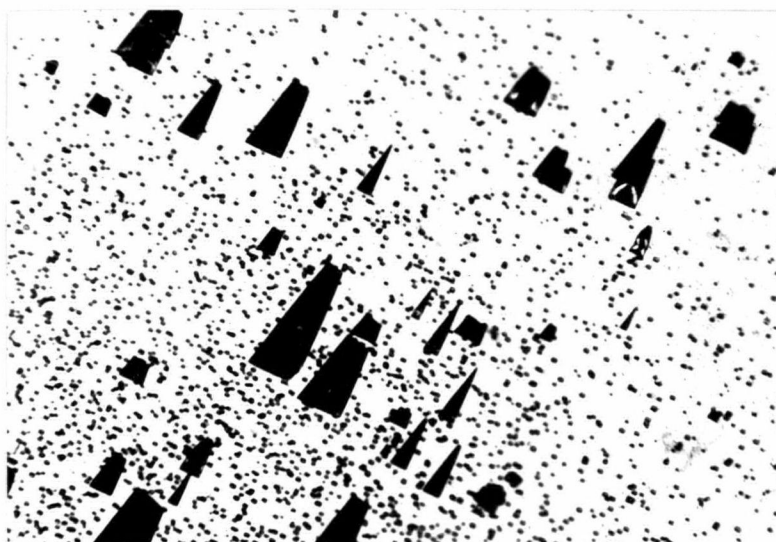
Etch Pits on High-Purity Aluminum



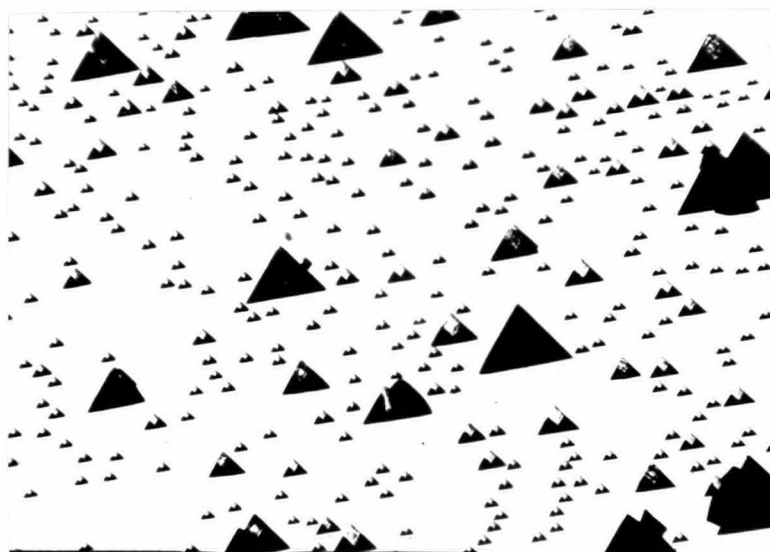
Micro 29. Bicrystal G. Etched  
in Lacombe and Beaujard's  
etch. Mag: x100.



Micro 30. As Micro 29  
Mag: x2,000



Micro 31. Etched consecutively in Lacombe and Beaujard's, and in Barrett and Levenson's reagents. Mag: x100.

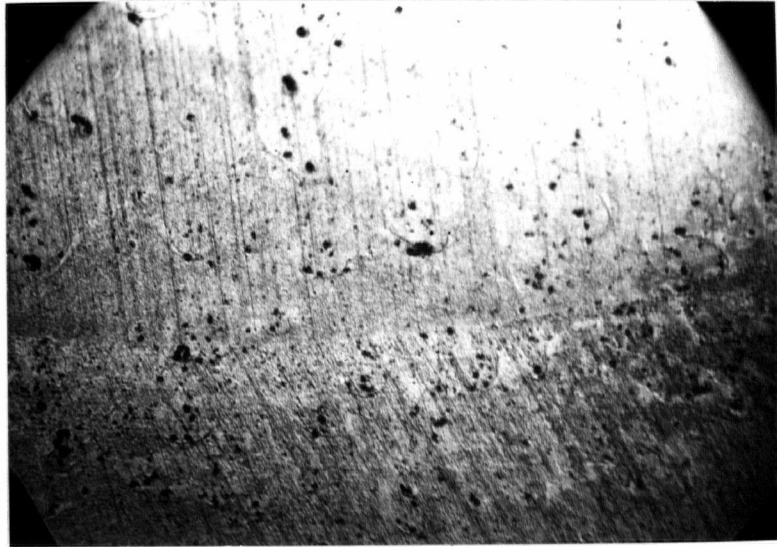


Micro 32. Etched in Barrett and Levenson's reagent. Surface near  $\{111\}$  orientation. Mag: x100.

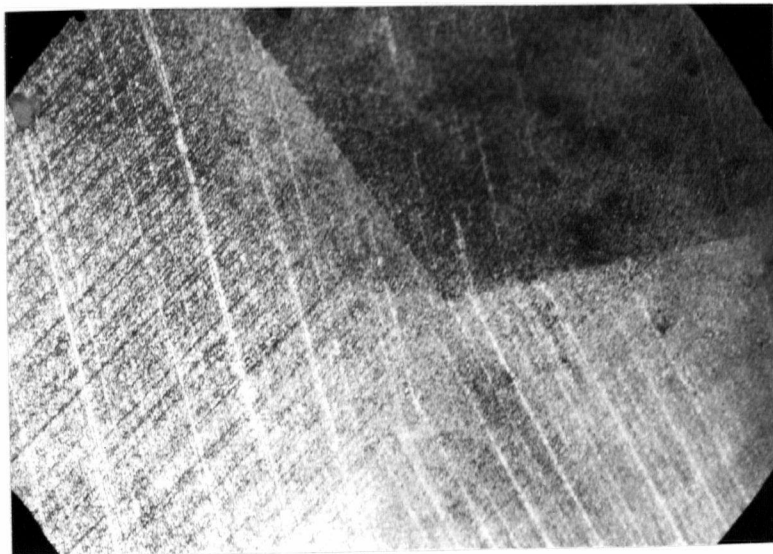


Micro 33. As Micro 32, but surface near  $\{100\}$  orientation. Mag:  $\times 100$ .

Slip Band Continuity in the Interior at Al - 4% Ag  
Alloys as Revealed by Electropolishing. Viewed on  
an anodised surface under polarised light. Mag: x100.



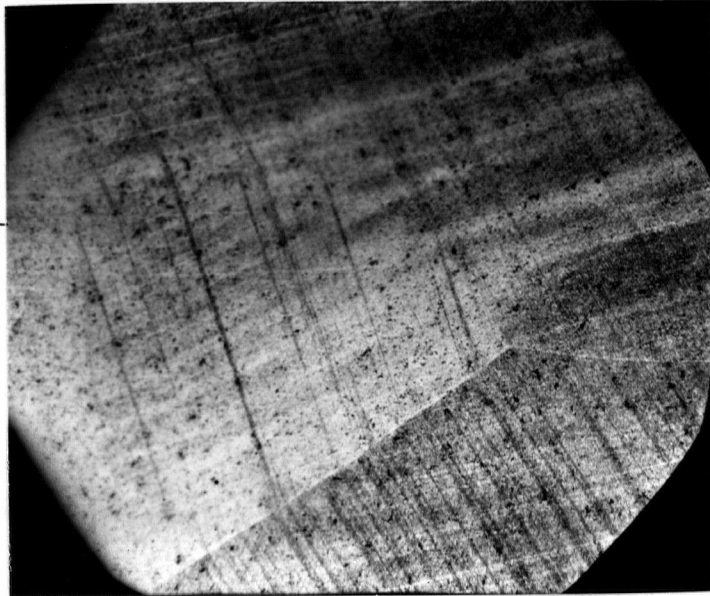
Micro 34.



| grain boundary

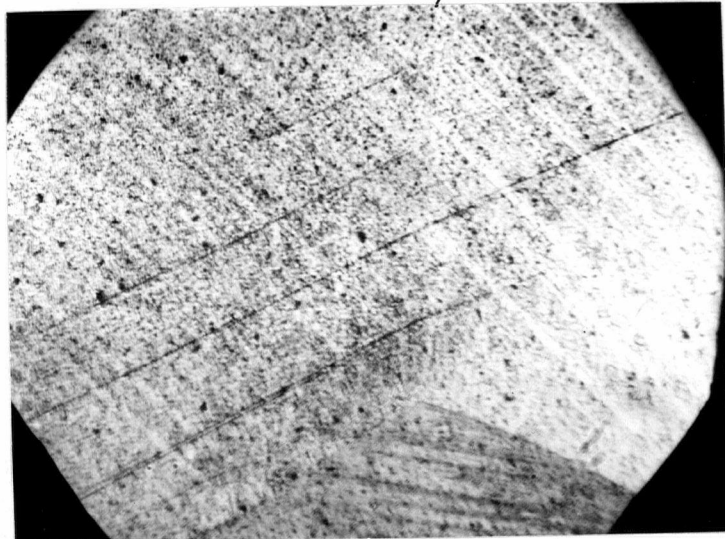
Micro 35.

grain  
boundary



Micro 36.

grain  
boundary



Micro 37.