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DISLOCATION ARRANGEMENTS IN DEFORMED COPPER POLYCRYSTALS

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(Masters Thesis)

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DISLOCATION ARRANGEMENTS IN DEFORMED COPPER POLYCRYSTALS

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ABSTRACT

The very early stages of plastic deformation were studied by means of etch pit techniques in large grained copper polycrystals deformed in compression. Special attention was given to regions near grain boundaries in an attempt to clarify the role of grain boundaries in plastic deformation. Their role as barriers to slip during the early stages of plastic deformation was clearly demonstrated. One of the first indications that plastic deformation had taken place was the operation of Frank Read sources. The resulting dislocation loops frequently traversed the entire cross-section of the grain and piled up against the grain boundary.

I. INTRODUCTION

Grain boundaries are believed to contribute to the increased strength of polycrystalline metals by acting as barriers to slip. Slip lines terminating at grain boundaries can be readily observed in the light microscope. Also, dislocations piling up along the slip planes at the grain boundaries can be directly observed by means of special etch pit techniques or transmission electron microscopy of thin films. These pile ups may activate sources close to the boundary on new slip systems both in the same grain and in the neighboring grain. There is, further, the possibility that grain boundaries may themselves act as sources of dislocations. According to Li¹ as long as grain boundaries are not atomically flat there will be ledges along the boundary and the stress necessary to emit dislocations from these ledges is only of the order of the yield stress. Ledges or steps have also been suggested by the coincidence lattice model of a high angle boundary developed by Brandon et al.² Some direct evidence for their existence has been obtained from field ion microscope observations. Although ledges have been observed there is yet no direct evidence that dislocations are emitted from grain boundaries.

The purpose of the present work was to follow, by means of etch pit techniques, the changes in the dislocation arrangements with increasing strain in large grained copper polycrystals. Particular attention was given to regions near grain boundaries, in an attempt to get new information on the role of grain boundaries during the very early stages of plastic deformation. One difficulty in the application

of the etch pit technique to polycrystalline metals is the fact that dislocations can be revealed by etching only when a low index plane is parallel to the surface of observation. Also, there are many variables that cannot be controlled such as the relative orientations of the various neighboring grains, the spatial arrangement of grain boundaries, etc. However, it was felt that etch pit observations, though not amenable to quantitative interpretation, might at least be helpful to get a better qualitative picture of the behavior of dislocations in polycrystalline metals, particularly during the pre-yield range of plastic deformation.

The effect of temperature on the general nature of the dislocation arrangements was studied by carrying out deformations at three different temperatures, viz. liquid nitrogen temperature, room temperature and 600°C.

II. EXPERIMENTAL PROCEDURES

Specimens (3 cm X 2 cm X 2 cm) of OFHC copper machined out from a larger block were heated to about 1060°C in a vacuum of less than 10^{-5} mm of Hg and were held at that temperature for 48 hours to produce large grains with an average size of 5 mm.

Since successful etching requires a surface orientation within 2 to 3° of {111} the specimens were first visually examined to find a grain having a {111} plane nearly parallel to the external surface. The presence of a large number of twins in the annealed specimens was very helpful in quickly detecting grains having a {111} plane within 10 to 15° of being parallel to a side surface of the specimen. Since

the twinning plane in copper is of the {111} type the straight twin traces visible on the surface of the specimens are parallel to the {111} planes in the individual grains. Hence, the problem of finding a grain having a {111} plane nearly parallel to the external surface reduces to finding a grain having two twin traces at an angle close to 60 degrees to each other. Since most of the grains showed non-parallel twin traces it was relatively easy to find the required type of grain. Whenever possible, grains in the central region of the external surface were chosen so that grain boundaries all around the selected grain could be observed.

The exact orientation of the grain selected as above was then determined by the back reflection Laue method and a slice (about 1 cm in thickness) with one face parallel to and containing the {111} plane of the particular grain obtained by cutting the specimen on an acid saw.³ A goniometer was used to hold the specimen and tilt it to the exact {111} orientation. The resulting slice was further suitably cut on the acid saw to get specimens approximately 2 cm X 1 cm X 1 cm with a central grain on one of the 2 cm X 1 cm faces having a {111} plane parallel to the face. The orientation of the grain was again checked by X-rays and then polished to within 2° of {111} on a chemical polishing wheel³ using a solution of the following composition: -

50% nitric acid, 25% acetic acid and 25% phosphoric acid.

The original dislocation substructure in the grain was observed by electropolishing the surface in a 60% phosphoric acid - 40% water solution (at a cell voltage of 1.5V and a current density of 0.1 amps/cm²)

and etching in a solution of the following composition⁴ for about 10 seconds.

Etchant composition: -

1 part bromine

15 parts acetic acid

25 parts hydrochloric acid and

90 parts water

The observations of etch pits were made with an optical bench metallograph.

The specimens were then compressed across the 1 cm X 1 cm faces using an Instron machine at the three different temperatures, viz. liquid nitrogen temperature, room temperature and 600°C. The applied stresses ranged from 50 to 1000 g/mm². (At stresses higher than these the density of dislocations near the boundaries often became too high and their arrangement too complex to be studied profitably). After each successive deformation the specimens were electropolished just enough to get a plain surface and then etched to follow the nature of the dislocation distribution. Unless otherwise stated, the amount of material removed between successive etchings was always of the order of 5 microns. Low temperature deformation was accomplished by keeping the specimen immersed, during compression, in liquid nitrogen in a styrofoam container. An ordinary split-core air furnace was employed for the high temperature compression.

III. RESULTS AND DISCUSSION

1. General Arrangement of Dislocations

The distribution of etch pits observed after the liquid nitrogen temperature and room temperature compressions corresponded closely to those observed in tension experiments on copper single crystals by Basinski and Basinski⁵ at liquid helium temperature and by Young⁶ and Livingston⁷ at room temperature. There was no marked difference in the dislocation arrangements whether the compression was carried out at liquid nitrogen temperature or room temperature. Figures 1(a) to 1(d) are photomicrographs of an interior region in a grain after successive compressions to stresses of 300, 400, 500 and 600 g/mm², respectively, at room temperature. At low stresses the distribution of dislocations showed some alignment along directions parallel to the trace of the primary slip planes. With increasing stress such alignment became increasingly evident. Occasionally, regions of high dislocation density were found to form even in the early stages of deformation. Figure 2, which is another area of the same grain shown in Fig. 1 after loading to 400 g/mm² is a good example. The thick dense bands always were parallel to the trace of a secondary slip system. These bands apparently formed in regions where early in the deformation a source on the secondary system had sent out loops. Even though no further activity on the secondary system had occurred, the dislocation interactions represented enough of a barrier to dislocations of the primary system to start the growth of tangles that continued to develop as total strain on the primary system increased. In a few cases there

was evidence for equally prominent slip occurring on more than one slip system. Figure 3 shows such a region of fine multiple slip in a sample also deformed to about 400 g/mm^2 compressive stress at room temperature. A cell structure as shown in Fig. 4 (0.2% compressive strain at liquid nitrogen temperature) was only occasionally evident after low temperature compressions. However, a cell structure was the most prominent feature of the dislocation arrangements after high temperature compressions. Figure 5 (200 g/mm^2 compressive stress at 600°C) is a typical example. Another distinguishing feature at the high temperature was that at stresses more than about 400 g/mm^2 the few slip traces observed were always very short, wavy and diffuse (Fig. 6) as against the comparatively longer, straighter and more distinct slip traces observed at lower temperatures (e.g. Figs. 1(a) to 1(d)). At high temperature the slip traces were long and distinct only at stresses less than about 100 g/mm^2 (Fig. 7(a)). Alignment along the trace of the active slip plane disappeared almost completely at high stresses. Figure 7(b) shows the same region after the stress has been raised to only 200 g/mm^2 . The slip traces are much less distinct, particularly near the grain boundary.

2. Dislocations Near Grain Boundaries

One particularly striking feature observed at the smallest stresses for all the three temperatures was small dislocation pile-ups at the boundaries. Figure 8 shows such a group of dislocations near a twin boundary in a sample compressed to only about 100 g/mm^2 at 600°C . Figures 9 and 10 show similar arrangements after compression to about 400 g/mm^2 at liquid nitrogen temperature. Figure 11 is an example

of a case in which such groups were seen in a sample which had not been intentionally deformed at all. The formation of these pile-ups shows that dislocation loops emanating from the first sources to become active more across a substantial portion of the entire cross section of the grain without leaving any trace. Their operation can be detected only because of the grain boundary barriers. High sensitivity strain measurements have also suggested that the first dislocation to move glide over long distances.⁸ It is interesting to note that in single crystal etch pit experiments operation of these first sources might not be detected at all because in the absence of any barriers to stop them the dislocation loops might traverse the entire crystal and pass out at the surfaces.

An explanation for cases like Figure 8 where "pile-ups" are seen at one boundary, but not at the opposite one could be that slip has been transmitted across the boundary. Such a transmission of slip should be particularly easy if the boundary happens to be a coherent twin boundary (as is perhaps the case in Fig. 8), since an active Burger's vector could then be common to both the grains. Another possibility is that a considerable amount of plastic deformation has taken place in the neighboring grain (which is not in the etching orientation), and that pile-ups at the boundary have activated sources in the etched grain near the boundary.

The operation of a Frank Read source at low stresses is clearly indicated in Figure 12(a) which shows both sets of pile-ups at the opposite boundaries in a specimen compressed to about 100 g/mm^2 at room

temperature. It is interesting to note that one set of dislocations appears as dark pits while the other appears as light pits. It has been previously shown by Livingston⁹ that edge dislocations of one sign are revealed as dark pits and those of opposite sign are revealed as light pits under appropriate lighting conditions. This particular source in Fig. 12(a) was apparently a surface source, viz. one that operated very close to the external surface since the pile-ups completely disappeared after two successive polishings (to remove about 50 microns of material each time) and re-etching (Figs. 12(b) and 12(c)). A calculation of the back stress at the source due to the dislocation pile-ups in Fig. 12(a) using the formula

$$\text{Stress} = \frac{G nb}{2\pi(1-\nu) r}$$

with G = shear modulus = 4.22×10^6 g/mm²

n = number of dislocations in pile-up = 9

b = Burger's vector = $\frac{a}{2} \langle 110 \rangle = 2.56\text{\AA}$

ν = Poissons ratio = 0.34

and r = distance from source to pile-up = 100 μ

gave a value of 21.96 g/mm² in close agreement with the resolved shear stress of 22.13 g/mm² on the particular slip system. It should,

however, be mentioned that the actual stress on the slip system might be considerably affected by the unknown deformations of the neighboring grains. The agreement between the two values cannot be taken too seriously.

Operation of a Frank Read source deep within the grain is shown in

Figs. 13(a) to 13(d). Figure 13(a) shows an isolated group of dislocations apparently piled up at a twin boundary after compression of the specimen to about 75 g/mm^2 at room temperature. Figure 13(b) shows a similar group of dislocations in the interior of the grain but apparently along the same slip plane as those dislocations near the boundary in Fig. 13(a). However, there was no pronounced alignment of etch pits along the same slip plane in the region in between these groups. Removing about 50 microns of material by chemical polishing and re-etching showed many more dislocations aligned along the particular slip plane and the operation of a Frank Read source became quite evident (Fig. 13(c)). This observation can be explained by the fact that on removal of material more and more dislocation loops already generated from the source intersected the external surface. Figure 13(d) shows both pile-ups at a lower magnification.

The apparent pile-ups near the grain boundaries in Figs. 8 to 11 and the increase in the number of etch pits observed along the slip trace in Fig. 13(c) could also be explained by assuming that the dislocation sources were at points along the grain boundaries. However, there was no evidence to support the idea of grain boundary sources whereas in some cases, like that shown in Fig. 12, there was strong indication that the source had been located at the surface. The increased spacing of the dislocations in the pile-up in Fig. 12(b) and their disappearance in Fig. 12(c), strongly suggest a surface source. Also, because the grown-in dislocations are highly mobile in copper, it might be expected that the dislocation sources having the

lowest critical stresses for operation would be single ended surface sources.

Although the pile-ups were mostly observed at twin boundaries regular grain boundaries, particularly when approximately perpendicular to the slip plane trace, also acted as barriers to dislocations. Figures 14 and 15 show pile-ups at such boundaries in specimens compressed to about 300 g/mm^2 at room temperature and liquid nitrogen temperature, respectively. A particularly interesting feature of Fig. 14 is the pronounced "glide polygonization"⁴ near the boundary. The plastic strain associated with a structure like that of Fig. 15, can be calculated to be about 5×10^{-5} if one assumes that the dislocations have traversed through the entire grain. It is interesting that even at these rather high strains the dislocation density in the bulk of the grain remains practically the same as in the undeformed sample, the only regions of higher dislocation density being those near the boundaries. This tendency is found to persist even at higher strains, the regions of maximum dislocation density still being those near the boundaries. Figure 16 shows a region in a sample deformed to about 0.05% strain at room temperature already having developed a much higher dislocation density near a twin boundary than in the central region of the grain.

In a few instances where two neighboring grains of different orientations could be simultaneously etched there was a pronounced difference in the density and arrangement of dislocations in the two grains (Fig. 17, for example). This can be due to the fact that one

grain is oriented for duplex slip while the other is oriented for single slip. The operative slip system in a given grain will not only depend on the orientation of the grain with respect to the compressive axis, but also on the orientation of all the surrounding grains. Because of this fact different slip systems sometimes operate in different regions of the same grain.

IV. CONCLUSIONS

1. Motion and multiplication of dislocations begins in polycrystalline copper at resolved shear stresses below 20 g/mm^2 as in single crystals.
2. Dislocation loops that emanate from the first operating Frank Read sources glide easily through the grain and form small pile-ups of three to ten dislocations at the grain boundaries.
3. The motion of the first five or ten loops across a given glide plane leaves few defects behind that can be detected by etching. Only the pile-ups at the grain boundaries show that a Frank Read source has operated.
4. As plastic strain increases the dislocation arrangement develops differently at low and high temperature. At -196°C and at 20°C the active slip plane become marked with increasing clarity by lines of pits. In specimens compressed at 600°C alignment of pits along active glide planes disappears after small strains in favor of a network of dislocation tangles forming a cell structure.
5. A small amount of slip on a secondary system occurring during the earliest stage of deformation can very greatly increase the

local density of dislocations left in the crystal after a given strain even when essentially all of the total strain has been on the primary system.

6. In copper the primary role of the grain boundaries during the early stages of slip is to act as barriers to dislocations and not as sources of dislocations.

ACKNOWLEDGMENTS

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References

1. J. C. M. Li, *J. Aust. Inst. Metals*, 8, 206 (1963).
2. D. G. Brandon et al., *Acta Met.*, 12, 813 (1964).
3. F. W. Young, Jr., and T. R. Wilson, *Rev. Sci. Inst.*, 32, 559 (1961).
4. J. D. Livingston, *J. Appl. Phys.*, 31, 1071 (1960).
5. Z. S. Basinski and S. J. Basinski, *Phil. Mag.*, 9, 51 (1964).
6. F. W. Young, Jr., *J. Appl. Phys.* 33, 963 (1962).
7. J. D. Livingston, *Acta Met.*, 10, 229 (1961).
8. R. F. Tinder and J. Washburn, *Acta Met.*, 12, 129 (1964).
9. J. D. Livingston, Direct Observation of Imperfections in Crystals, Interscience Publishers, New York (1961).

FIGURE CAPTIONS

- Fig. 1 a, b, c, d: Photomicrographs showing changes in dislocation arrangements in the interior region of a grain after successive compressions of the specimen to stresses of 300, 400, 500 and 600 g/mm², respectively, at room temperature.
- Fig. 2 A particularly good region showing dense bands of dislocation along traces of a secondary slip system in the same specimen as in Fig. 1 after loading to 400 g/mm² at room temperature.
- Fig. 3 Fine duplex slip in a specimen loaded to about 400 g/mm² at room temperature.
- Fig. 4 Unusual area showing a cell structure in a specimen deformed to about 0.2% compressive strain at liquid nitrogen temperature.
- Fig. 5 Typical area showing cell structure in a specimen loaded to about 200 g/mm² at 600°C.
- Fig. 6 Short, wavy and diffuse slip traces after loading of a specimen to about 600 g/mm² at 600°C. Operation of two slip systems can be noticed.
- Fig. 7 (a) Long distinct slip traces in a specimen loaded to about 100 g/mm² at 600°C.
(b) Same region as in Fig. 7(a) after increasing the loading to 200 g/mm². Note that slip traces and pile-ups have become much less obvious.
- Fig. 8 Groups of dislocations near a twin boundary in a specimen loaded to 100 g/mm² at 600°C.
- Fig. 9 and 10 Similar dislocation arrangements as in Fig. 8 in a specimen

loaded to about 400 g/mm^2 at liquid nitrogen temperature.

Fig. 11 Piled up dislocations at twin boundary in a specimen which had not been subjected to any intentional deformation at all.

Fig. 12 (a) Dislocation pile-ups of opposite sign held up at opposite boundaries in a specimen loaded to 100 g/mm^2 at room temperature.

(b) and (c) Same region as in Fig. 12(a) after successive removals of about 50 microns of material each time and re-etching. Note that the dislocation loops have been completely polished away in Fig. 12(c).

Fig. 13 (a) Group of dislocations piled up at a twin boundary after loading of a specimen to about 75 g/mm^2 at room temperature.

(b) Group of dislocations found near the center of the grain on the trace of the same glide plane as the group.

(c) Same region as in Fig. 13(a) after removing about 50 microns of material and re-etching.

(d) Same area as Fig. 13(c) showing both groups at lower magnification.

Fig. 14 Dislocations piled up at a boundary approximately perpendicular to the slip trace in a specimen loaded to 300 g/mm^2 at room temperature.

Fig. 15 Pile-up and glide polygonization at a boundary perpendicular to the slip trace in a specimen loaded to 300 g/mm^2 at liquid nitrogen temperature.

Fig. 16 Photomicrograph showing regions of high dislocations density

near the boundaries after compression to 0.05% strain at room temperature.

Fig. 17 . Difference in density and arrangement of dislocations in two grains of different orientations after about 0.1% strain at liquid nitrogen temperature.



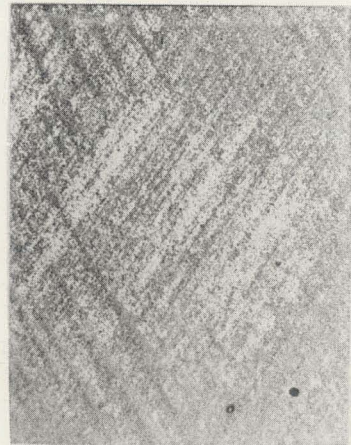
(a)



(b)



(c)



(d)

Fig. 1

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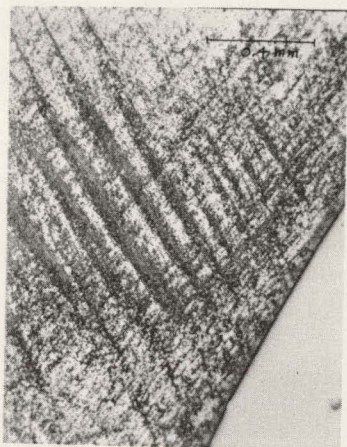


Fig. 2



Fig. 3



Fig. 4

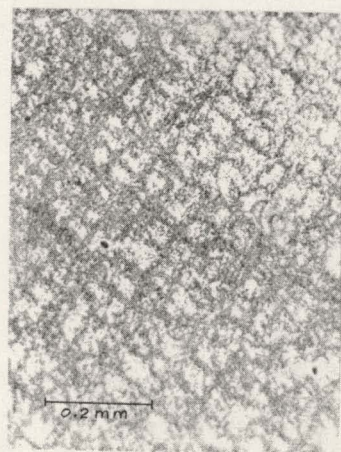


Fig. 5

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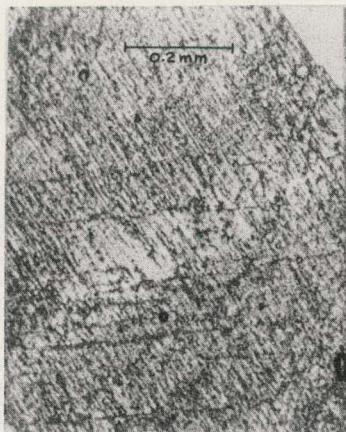


Fig. 6

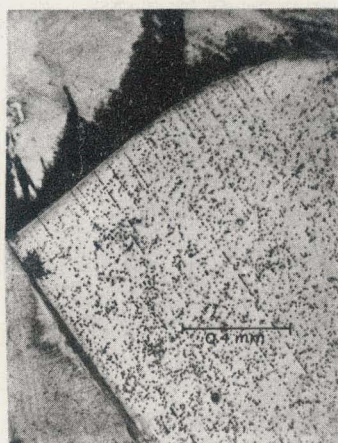


Fig. 7a

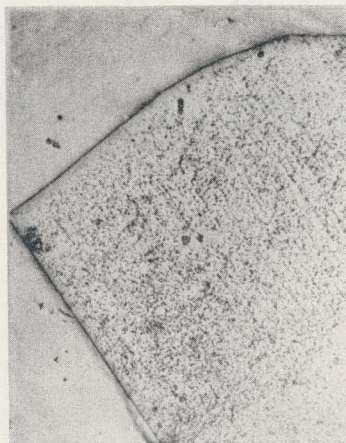


Fig. 7b

ZN-4701

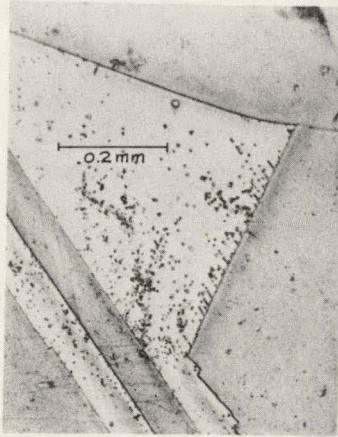


Fig. 8

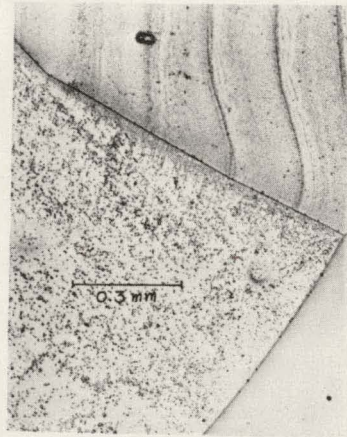


Fig. 9

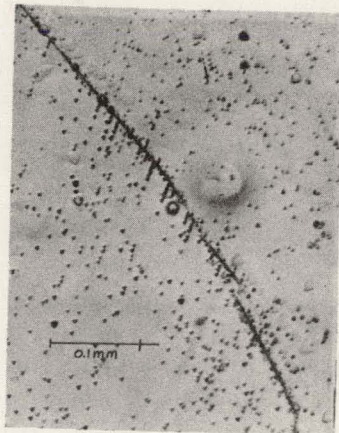


Fig. 10

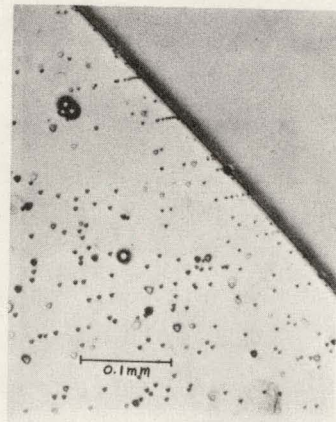
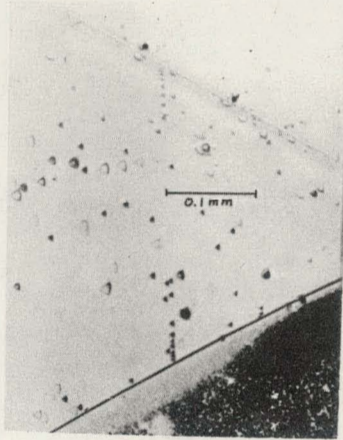


Fig. 11

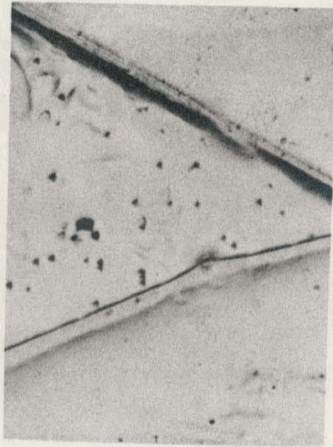
ZN-4702



(a)



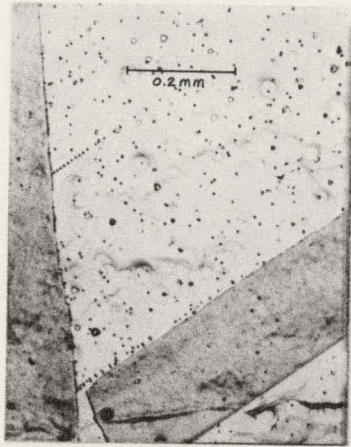
(b)



(c)

Fig. 12

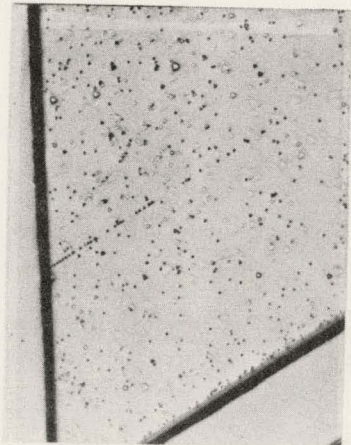
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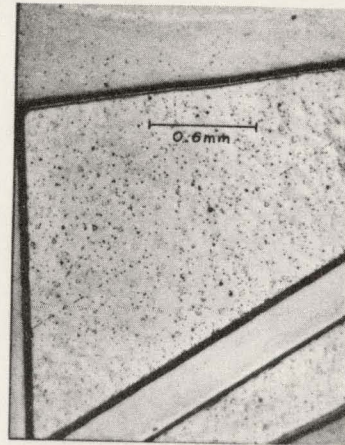
(a)



(b)



(c)



(d)

Fig. 13

ZN-4704

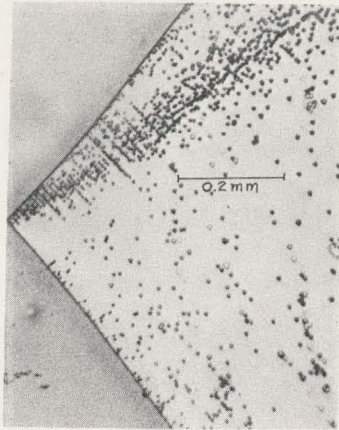


Fig. 14

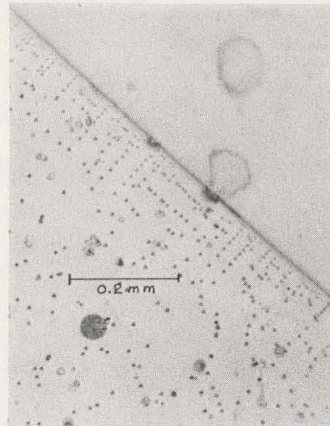


Fig. 15

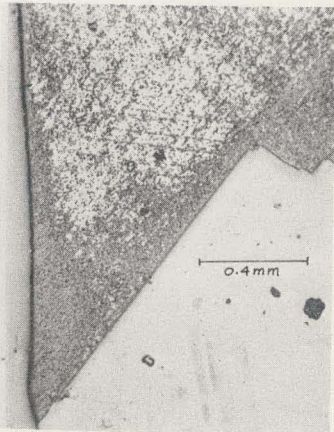


Fig. 16

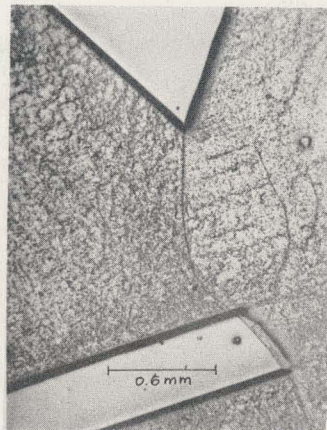


Fig. 17

ZN-4705

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