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ABSTRACT

Dislocation arrangements developed in large grained polycrystalline copper after small deformations in compression or bending were studied by the etch pit technique. The results indicated that the only role of grain boundaries in the preyield deformation of copper was to act as barriers to moving dislocations and not as sources of dislocations. Some special boundaries did however permit the passage of dislocations across them even in the microstrain region. It is suggested that the presence of such "transparent" boundaries is partly responsible for the lowering of the elastic limit of a polycrystal with increasing grain size.

Multiple slip always occurred in the preyield region and significantly affected the motion of primary dislocations both during loading and unloading by forming attractive junctions. It was found that dislocations in a pileup never collapsed completely back to the source on stress-reversal.

* This paper is partly based upon a thesis accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Engineering by the University of California, Berkeley, California in September 1968.

I. INTRODUCTION

It is well known that high-sensitivity strain measurements or dislocation etch-pit techniques can detect small plastic strains well below the macroscopic yield stress in many metals and alloys.[1] In particular Thomas and Averbach[2] observed plastic strains in the range of $1\text{-}500\times10^{-6}$ in high purity polycrystalline copper specimens at stresses above about 700 g/mm^2. With a still higher strain resolution of the order of 10^{-8} Tinder and Washburn[3] detected measurable plastic strains in tubular polycrystalline specimens of OFHC copper at stresses as low as 2 g/mm^2. However the author is not aware of any previous attempts to observe directly the dislocation arrangements developed within the grains of a copper polycrystal during these early stages of plastic deformation. In the theory of the microstrain region as originally developed by Thomas and Averbach and later modified by Brown and Lukens,[4] it is assumed that Frank-Read sources are present within the grains and that they begin to be activated on the application of a sufficiently high stress. The resulting dislocations then pile up at the grain boundaries and their back stresses eventually cause the source to cease operating. The principal role assigned to grain boundaries in the microstrain region is to act as barriers to moving dislocations and to impose a definite limitation on the number of dislocations that can be generated by a source at a given stress. The macroyield point is supposed to be reached when the piled up dislocations eventually produce the required stress concentration needed to activate new sources in the adjoining grain.

The etch-pit technique is obviously one method for directly observing the early dislocation arrangements in the individual grains of a deformed polycrystal and thereby testing the validity of this microstrain model. Etch-pit studies have so far been made only in a few materials with body centered cubic structure, the most extensive work having been done on silicon-iron.[5-8] The experimental results are partially in disagreement with the previously mentioned microstrain theory. The grain boundaries act as sources for slip nucleation in the early deformation stages rather than only as barriers to slip. No comparable observations have been made in any face centered cubic material, probably because dislocations in them can be revealed by etching only when a low index plane is closely parallel to the surface of observation. In a fairly large grained polycrystalline specimen the chances of being able to observe the dislocations in any surface grain become extremely small. In the present investigation large grained copper polycrystals with one or more etchable grains on the surface were prepared by special techniques and the dislocation arrangements in these grains were studied by the etch pit technique after small deformations in compression or bending. It was particularly hoped that information could be obtained on the nature of the dislocation sources and on the role of grain boundaries in the beginning stages of plastic deformation.

2. EXPERIMENTAL PROCEDURE

2.1 Specimen Preparation.

Since reliable etchants have been developed only for the {111} planes in copper,[9] experiments were aimed at obtaining a specimen in which at least one grain would have a {111} plane parallel to the surface. It was found that recrystallization of extruded copper rods resulted in the preferential formation of grains with a {111} type plane approximately normal to the extruding direction. Specimens for compressive loading were obtained by machining from extruded 3/4 inch square OFHC copper rods, (analysis given in Table I) chemically polishing[10] a surface that was normal to the extruding direction and annealing at about 1060°C for 48-72 hours in a vacuum of less than 10^{-5} mm of Hg. This treatment usually developed at least one etchable grain on the polished surface and an average grain size of about 5 mm. Specimens for bending experiments were similarly prepared from extruded 1-1/4 inch square OFHC copper rods. The dimensions of the specimens and their relation to the extruding direction of the starting material are indicated in Fig. 1.

2.2 Compression Experiments.

Specimens were compressed at room temperature using either an Instron machine or a micro-compression device schematically illustrated in Fig. 2. The device is made of stainless steel and is essentially like a C clamp in which, by turning the micrometer head, an increasing load could be applied to the specimen placed between the bottom end plate and the modified spindle of the micrometer as shown in the figure. The ball and socket joint at the head of the spindle ensured proper alignment and a uniform distribution of the load. The stainless steel plate P, kept flush against the vertical arm, minimized any torque that might have been transmitted to the specimen during loading. The two Teflon plates served to electrically insulate the specimen during electropolishing. Insulating lacquer was applied on all the surfaces of the specimen except the one on which observations were to be made and the one in contact with the stainless steel plate at the bottom through which electrical connection was made to the specimen. A calibration of the loads applied by the device was obtained by finding the number of turns required on the micrometer to cause the same deflection of the end plates as that caused by hanging a known weight from one of the end plates. The particular advantage of the microcompression device was that specimens could be etched and observed under the microscope while under stress. Also it enabled investigation of the dislocation distribution down to a limited depth below the surface of a specimen in the stressed condition by immersing the whole device in the polishing solution and reetching.

2.3 Bending Experiments.

A cantilever beam-type bending was employed by clamping the specimen (Fig. 1) securely in a vise at the left end and carefully attaching the necessary weights on a flexible rubber band running through the shallow V-grooves at the right end of the specimen. The V-groove only served to facilitate loading. Stress reversal was accomplished by simply turning the specimen upside down and applying the same load as before. Observations were always made on grains sufficiently distant from the clamping end. However, perceptible damage was generally found only in grains directly in contact with the vise.

2.4 Etching Procedure.

The etchant used to reveal dislocations was the one developed by Livingston[9] and consisted of 1 part bromine, 15 parts acetic acid, 25 parts hydrochloric acid, and 90 parts water. The specimens were always lightly electropolished prior to etching using a solution of 60 parts phosphoric acid and 40 parts water at a cell voltage of 1.5 volts and a current density of 0.1 amps/cm^2. A double etching technique was used whenever the new positions of dislocations were to be related to the old ones. Unless otherwise mentioned the amount of material removed from the surface between successive etchings was always of the order of 5-10 microns.

3. RESULTS AND DISCUSSION

The recrystallized grains had a relatively high perfection with dislocation densities always less than $10^4/cm^2$ and frequently of the order of $10^3/cm^2$. Dislocations were quite uniformly distributed and there were usually no subboundaries. Conditions were thus ideal to study the early dislocation phenomena, particularly the role of grain boundaries because there were no other grown-in internal barriers to significantly affect the motion of dislocations.

As the applied stress was gradually increased the first conspicuous indication of the occurrence of plastic deformation was frequently the appearance of groups of dislocations piled up against the grain boundaries. Figures 3(a) to (d) show a few typical examples of such dislocation pile-ups in specimens etched after application of a compressive stress of 40, 50, 50 and 55 g/mm^2 respectively. (All stressed reported are applied stresses unless otherwise mentioned. Also the direction of the applied stress is indicated in all micrographs by a black line of 0.5 mm length unless otherwise noted.) When dislocation pile ups were present at one grain boundary there was usually a corresponding pile-up at the opposite boundary as may be seen in Figs. (3a) and (b). In similar etch pit experiments on silicon-iron, grain boundaries were concluded to be the sources for slip nucleation primarily from the observation that isolated grains typically yielded by slip bands coming from only one side of grain, there being no matching slip bands on the opposite side of the grain (for example see Fig. 1 in Reference 7). The definite matching, in the present experiments, of the pile ups on opposite sides of the grain suggest that in copper the early dislocation sources are located within the grain and not in the grain boundaries. The possibility that the corresponding sets of etch pits might represent the points of intersection with the surface of dislocation loops sent out from a grain boundary source located at some place away from the surface, was eliminated by a direct examination of the three-dimensional configuration of the dislocations responsible for the formation of such a typical double ended pile up. Figure 4 (a) shows such a pile up in a specimen subjected to a stress of 50 g/mm^2 and etched after unloading. The fact that one group of dislocations appears as dark pits and the other as light pits suggests that they mark the points of emergence of opposite segments of the same dislocation loops.[11] Figures 4(b) and (c) show the same grain etched after removal of 50 and 100 microns, respectively, of material from the surface. In general no new etch pits were revealed as the surface was polished down; the spacing between the etch pits in the pile ups increased and finally both sets of pits disappeared. These results indicate that the double-ended pileup originally consisted of dislocations of approximately semicircular shape generated from a single-ended surface source. Details of the generation and operation of such sources have been discussed elsewhere.[12] The fact that the dislocations in the piled up groups were traveling towards the grain-boundaries during the initial application of the stress is also indicated by the back motion of these dislocations on unloading. Figure 5 shows a grain in a specimen subjected to a compressive stress of approximately 50 g/mm^2 and consecutively etched in the stressed and unstressed conditions. The many small (black) etch pits along the direction of the arrow represent the stress relaxed positions of some dislocations of the pile up A whose original positions are indicated by the larger flat bottomed (white) etch pits near the grain boundary.

The difference between the preyield deformation behavior of b.c.c. and f.c.c. materials may be due to the fact that grown-in dislocations in b.c.c. materials are all strongly pinned. Multiplication can then occur only where fresh dislocations have been introduced or in regions of high stress concentration. Grain boundaries may provide regions of stress concentration in the form of precipitates or inclusions. Moon and Vreeland[8] observed that slip bands in polycrystalline silicon-iron formed preferentially from fresh scratches rather than grain boundaries indicating that the stress required to activate grain boundary sources was greater than that required to multiply fresh dislocations. When some grown-in dislocations are highly mobile as in f.c.c. materials they are probably able to multiply and propagate much before the operation of any potential grain boundary sources.

Additional information on the nature of the early dislocation pile ups was obtained from a study of their behavior under stress reversal. In particular it was hoped to discover whether dislocations in a pile up could run completely back into their original source or whether they would be annihilated by dislocations of opposite sign generated by the same source under the reversed stress. A typical sequence is shown in Fig. 6. Figure 6 (a) shows a grain on the tensile surface of a specimen deformed in bending to a stress of about 20 g/mm^2 and etched in the stress relaxed condition. The dislocation groupings A and B represent the opposite halves of the same disloaction pile up. On application of an equal load in the opposite direction the dislocations in group A undergo considerable back motion as seen from Figs. 6 (b) and (c) which show the same grain after etching in the unloaded condition and after removal of about 20 microns of material from the surface, respectively. The new positions of the dislocations are at C in Figs. 6 (b) and (c). The fact that they have moved away from the grain boundary on the right is indicated by the flat pits in Fig. 6 (b) and the disappearance of these pits on surface polishing as seen from Fig. 6 (c). The relative immobility of the dislocation segments at B in Fig. 6 (a) is probably due to interaction of some of these dislocations with dislocations of another active slip system evident at the bottom left region of the picture. Figure 6 (d) shows the same grain after increasing the reverse applied stress to 29 g/mm^2 and etched after unloading with an intermediate polishing off of about 20 microns of material. There is still no indication that dislocations of opposite sign have been generated from the original source. It was not possible to make all the original dislocations run back into their source even at a reverse stress of about 40 g/mm^2 as seen from Fig. 6 (e). When the same stress of 40 g/mm^2 was then applied in the forward direction the dislocations again moved back to the boundary in the right as shown by the pile ups A in Fig. 6 (f).

The results may be of some significance in the interpretation of the Bauschinger effect. It is usually observed (see, for example, Edwards and Washburn(13)), that when the stress is reversed plastic flow begins at a very low stress, much below the original yield, but thereafter hardening is rapid and after a small additional strain the original hardening curve is continued, shifted to the right along the strain axis. The present results suggest that during the initial loading a few sources begin to operate and the resulting dislocations pile up against the grain boundaries or some other internal barriers. On stress relaxation a few of these dislocations which have not been trapped by interactions move part of the way back towards the sources. The reason why they cannot travel all the way back to the sources is probably as follows. During the initial loading sources on both primary and secondary systems operate but not simultaneously. Many dislocation loops reach grain boundaries after traversing the entire grain without many interactions with dislocations on other systems. However, on unloading all these loops start to contract simultaneously resulting in formation of a great many more attractive junctions. These interactions eventually stop the back motion. On applying a stress in the reverse direction some dislocations will be able to unpin from nodes and continue their back motion towards the source. This can still occur at a lower stress than that which started the operation of the source during the first loading. The result is a lower yield stress on reverse loading. The etch pit pictures in Fig. 6 show no evidence for any new sources beginning to operate in the initial stages of the reverse loading.

The apparent stability of some pileups even in the stress-relaxed condition, as in Figs. 3 (a) and (b) for example, is probably due to the interaction of their dislocations with the grown in dislocation network. Direct observations of dislocations by x-ray topography in lightly deformed copper single crystals[14] have shown that edge dislocations move at a faster rate than screw dislocations. When the active Burgers vector is parallel to the surface of observation as in the present experiments and when the sources are located near the surface the fast moving edge segments of freshly generated dislocation loops travel all the way across the grain towards the opposite boundaries before the screw parts of the loops penetrate very far into the grain. During their inward motion from the surface region the screw segments become incorporated into the grown in dislocation network by forming attractive junctions along their length. On removal of the applied stress the screw parts of the loop may not be able to break away from these junctions under the action of the back stress and only very limited reverse motion near the grain boundaries would occur. The result is the retention after unloading of many stable dislocation loops elongated in the direction of the Burgers vector whose edge segments are revealed by etching in the pile-ups.

Although the etching restrictions in copper normally precluded the possibility of simultaneously observing dislocations in adjoining grains there were a few instances where adjoining grains both happened to have an etchable {111} type plane nearly parallel to the surface of observation. Etch pit observations on such specimens after light deformation usually showed no correspondence of pile-ups or slip bands across the grain boundaries. However, there were a few special instances where slip continuity across the grain boundaries was apparent even from the very early stages of plastic deformation. The most frequently observed case was that of a coherent twin boundary when the operative Burgers vector was parallel to the boundary. The reason why in spite of the etching restrictions in copper direct observation of such slip propagation across twin boundaries could be made was that the surface of observation was never exactly parallel to any {111} plane and that dislocations could be revealed by etching on planes close to {111} within a few degrees. If the surface of observation is inclined at a small angle to a {111} twin boundary plane as schematically illustrated in Fig. 7 successive etchings at levels corresponding to A, B and C should reveal dislocations in grain 1, in parts of grain 1 and 2, and finally in grain 2 only. When the active Burgers vector is parallel to the twinning plane (as was usually the case in the present experiments) tions along their length. On removal of the applied stress the screw parts of the loop may not be able to break away from these junctions under the action of the back stress and only very limited reverse motion near the grain boundaries would occur. The result is the retention after unloading of many stable dislocation loops elongated in the direction of the Burgers vector whose edge segments are revealed by etching in the pile-ups.

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Direct passage of dislocations across a boundary was also found for other special boundaries. A good example is seen in Fig. 9 which shows many continuing slip bands across a small angle boundary in a specimen etched under a stress of 60 g/mm^2. Back reflection Laue pictures of the two grains showed that their relative orientations corresponded to a 6° rotation about a ⟨111⟩ axis normal to the surface of observation. The above slip continuity examples are consistent with the more general slip continuity requirements expressed by Ogilvie.[15] He observed that in aluminum and brass slip was continuous across straight boundaries when the lines of intersection of the slip plane with the boundary were within about two degrees of one of the directions ⟨110⟩, ⟨112⟩ or ⟨123⟩ for each grain, not necessarily the same direction in adjacent grains. Slip line continuity across less ideal grain boundaries has been observed more recently in aluminum bicrystals by Davis, Teghtsoonian, and Lu[16] but only at comparatively much higher strains than in the present experiments. These cases would probably have involved the activation of slip sources by dislocations piled up against the boundary. The ability of dislocations to pass through grain boundaries of special misorientations may have an important effect on the grain size dependence of the elastic limit of polycrystalline aggregates. Specimens with different grain sizes for experimental determination of elastic limits are obtained by different heat treatments. Large grain sizes are obtained by employing either higher annealing temperatures or longer annealing periods. Under these conditions special low energy grain boundaries such as twin boundaries and small angle boundaries will be retained while the higher energy boundaries will be preferentially eliminated. Dislocation propagation across the remaining boundaries in a coarse grained polycrystal would on the average be more frequent than in a fine grained specimen. The experimentally observed lowering of the elastic limit of a polycrystal with increasing grain size should then be at least partially due to the presence of an increasing proportion of "transparent" boundaries rather than entirely due to the change in grain size itself.

4. CONCLUSIONS

1. The only role of grain boundaries in the preyield deformation of copper seems to be as barriers to moving dislocations. Unlike in many b.c.c. materials grain boundaries do not act as sources of dislocations. This difference is probably due to the high mobility of some grown-in dislocation segments in pure f.c.c. metals which act as Frank Read sources at low stresses.

2. Multiple slip always occurs in the preyield region and significantly affects the motion of primary dislocations both during loading and unloading by forming attractive junctions. Interactions are most frequent during unloading because of the simultaneous back movement of dislocations on intersecting slip planes.

3. Dislocation loops that have spread out to the grain boundaries on initial loading contract to some extent on inloading and contract further on application of a reverse stress. However even for reverse stresses greater than the original forward stress they do not generally collapse completely back to the source and no loops of opposite sign are sent out from the source.

4. Propagation of slip across grain boundaries does not usually take place in copper in the microstrain region except in those special cases where dislocations from one grain can actually pass through into the next grain.

ACKNOWLEDGEMENTS

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Table I. Analysis of the OFHC Copper

Element	Copper	Iron	Lead	Nickel	Sulphur	Silver
%	>99.98	0.002	0.003	0.003	0.002	0.005

FIGURE CAPTIONS

Fig. 1 Dimensions and orientations of specimens used for bending experiments.

Fig. 2 Schematic drawing of the microcompression device (a) as viewed from the front (b) as viewed from the side.

Fig. 3 Typical dislocation pileups in specimens etched after application of a compressive stress of (a) 40 g/mm^2, (b) 50 g/mm^2, (c) 50 g/mm^2 and (d) 55 g/mm^2.

Fig. 4 (a) Dislocation pileups of opposite sign held up at opposite boundaries in a specimen subjected to a compressive stress of 50 g/mm^2; (b) and (c) Same region as in (a) etched after successive removals of about 50 microns of material each time.

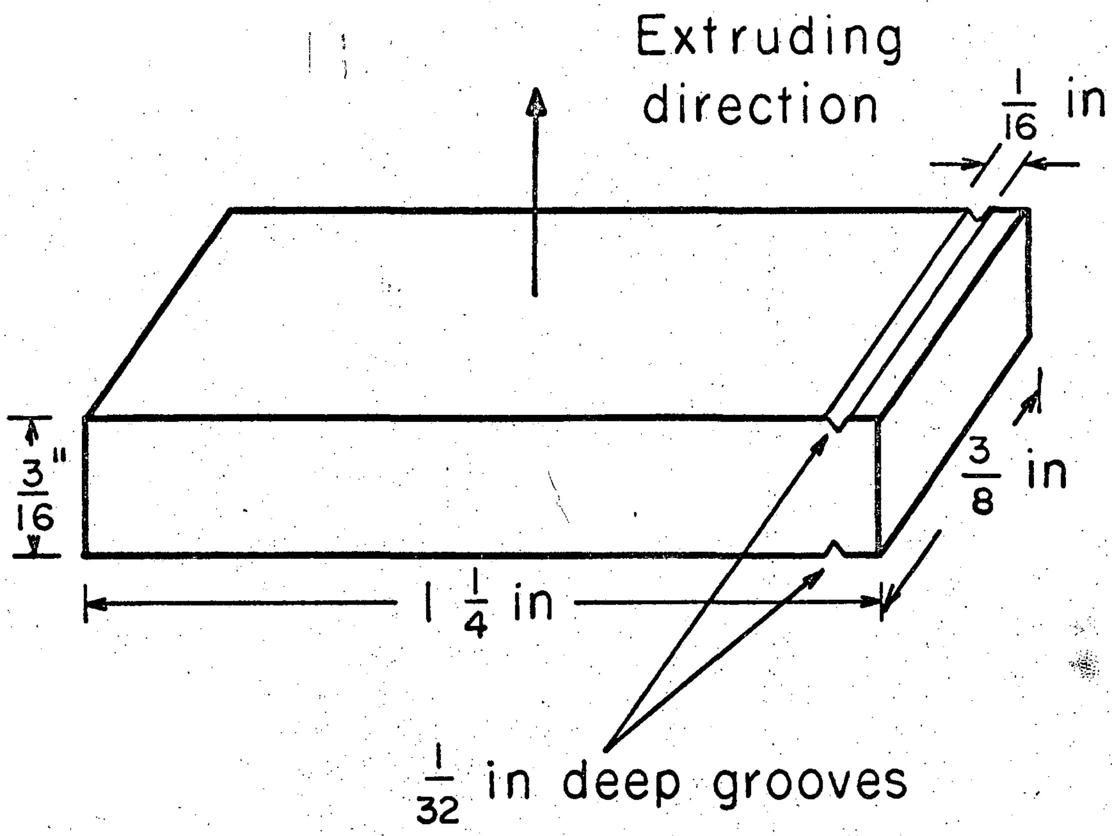
Fig. 5 Stress relaxation of a pileup A in a specimen subjected to a compressive stress of 50 g/mm^2 and consecutively etched in the stressed and unstressed conditions.

Fig. 6 A sequence showing the behavior of dislocation pileups on stress reversal (a). Etched in the unloaded condition after a stress of about 20 g/mm^2. (b) Double etched after application of a stress of 20 g/mm^2 in the reverse direction. (c) Etched after removing 20 microns of material from the surface after stage (b). (d) Etched after increasing the reverse applied to 20 g/mm^2. (e) Etched after applying a stress of 40 g/mm^2 in the reverse direction. (f) Etched after applying a stress of 40 g/mm^2 in the forward direction.

Fig. 7 Schematic illustration showing the relation between the surface of observation and the twin boundary.

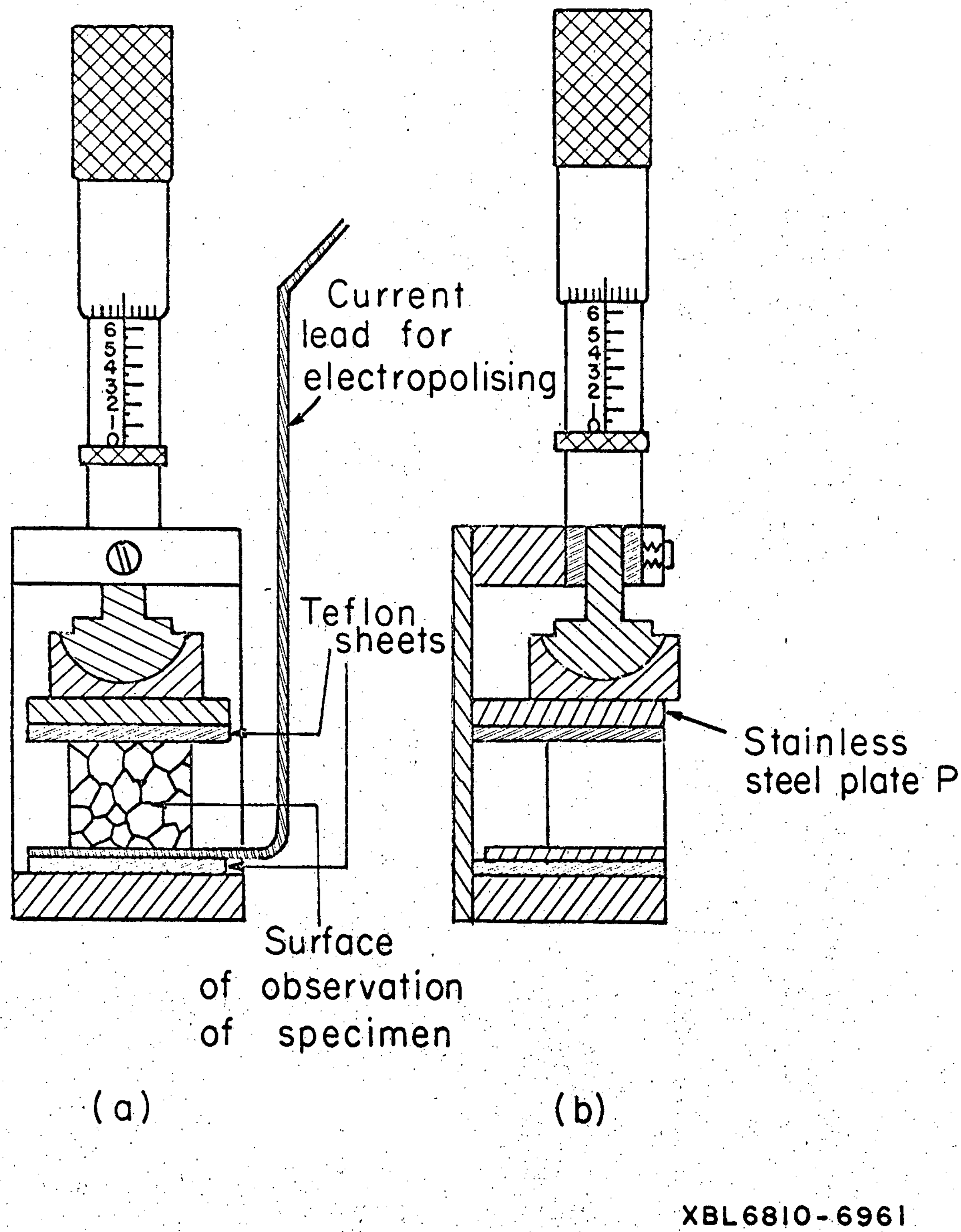
Fig. 8 Sequence showing twin boundary continuity of slip in a specimen etched (a) under a stress of about 50 g/mm^2, (b) after stress relaxation and removal of about 50 microns of material form the surface, and (c) after removal of an additional 50 microns of material from the surface. See text for details.

Fig. 9 Continuing slip bands across a low angle boundary in a specimen etched under a compressive stress of 60 g/mm^2.



XBL687- 3327

Fig. 1



Current lead for electropolising
6 5 4 3 2 1 0
6 5 4 3 2 1 0
Teflon sheets
Stainless steel plate P
Surface of observation of specimen
(a)
(b)
XBL6810-6961

Fig. 2

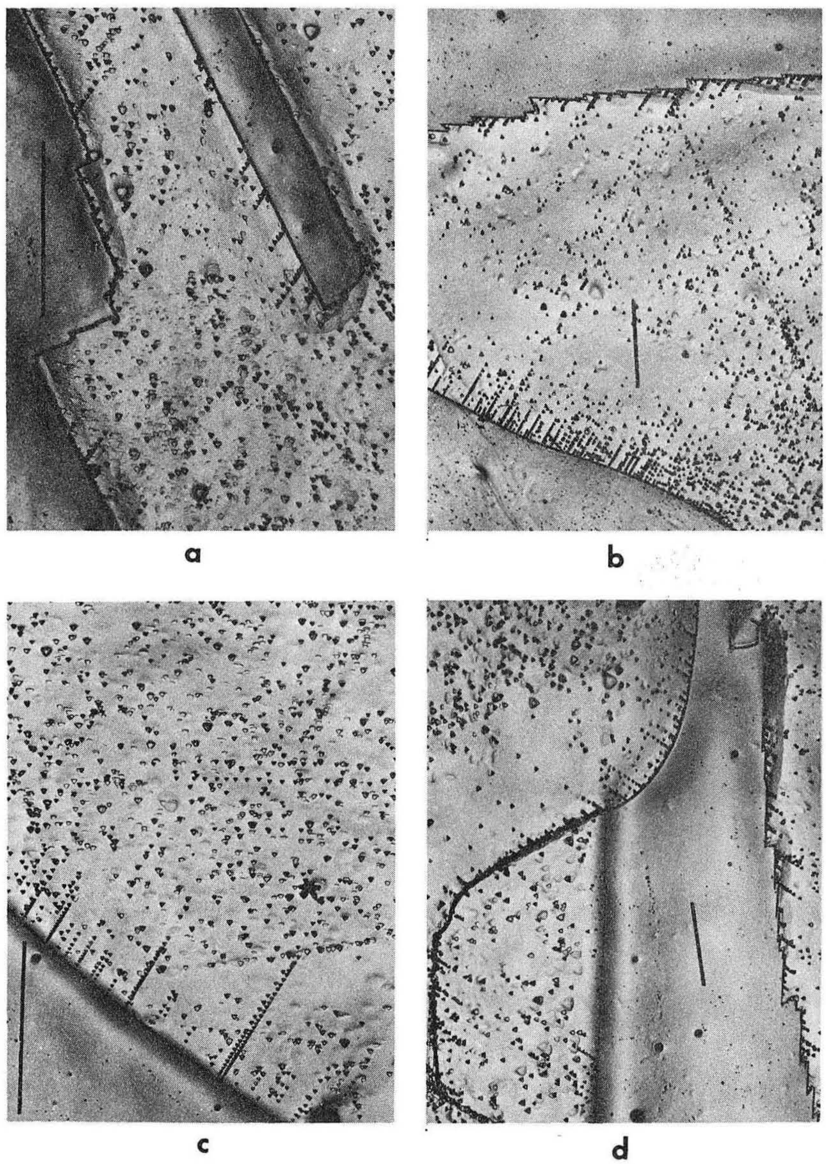


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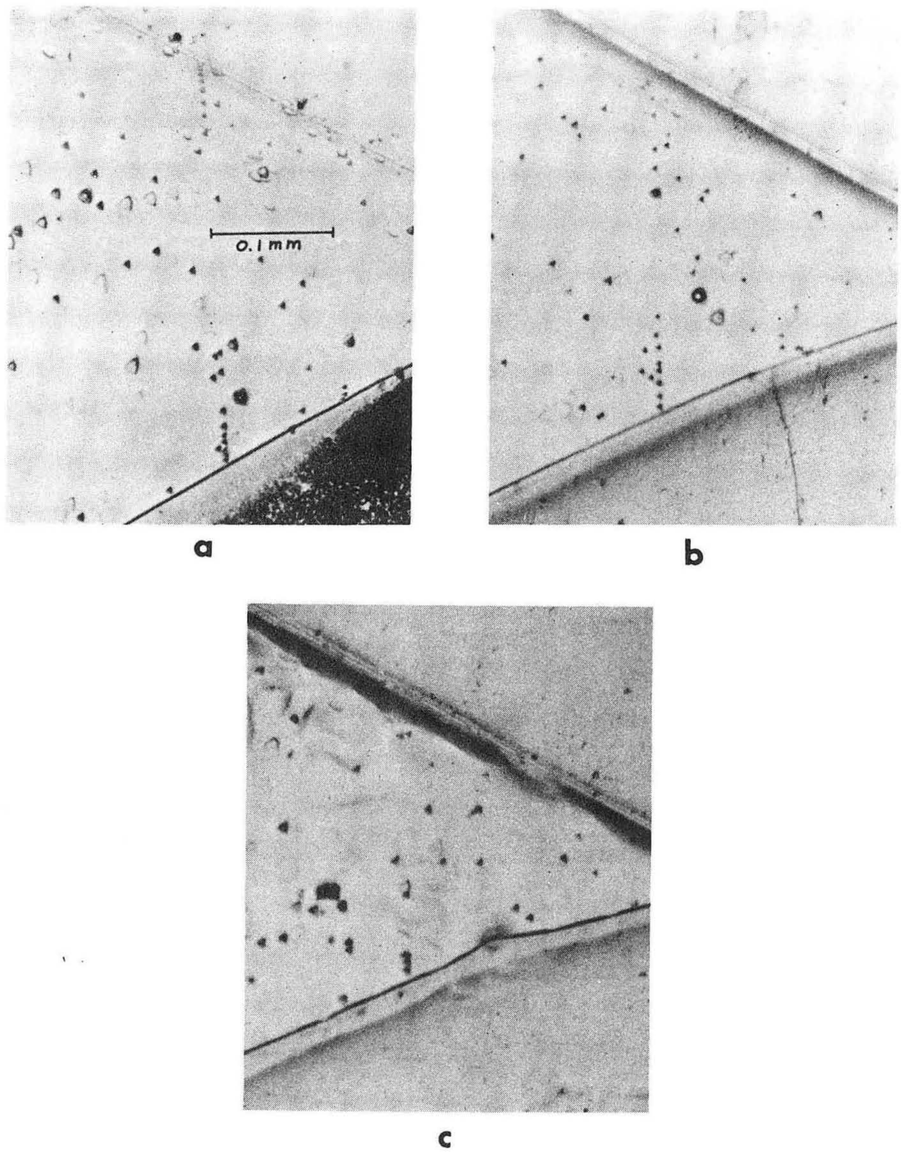
Figure 3



XBB 6812-7676

Figure 4

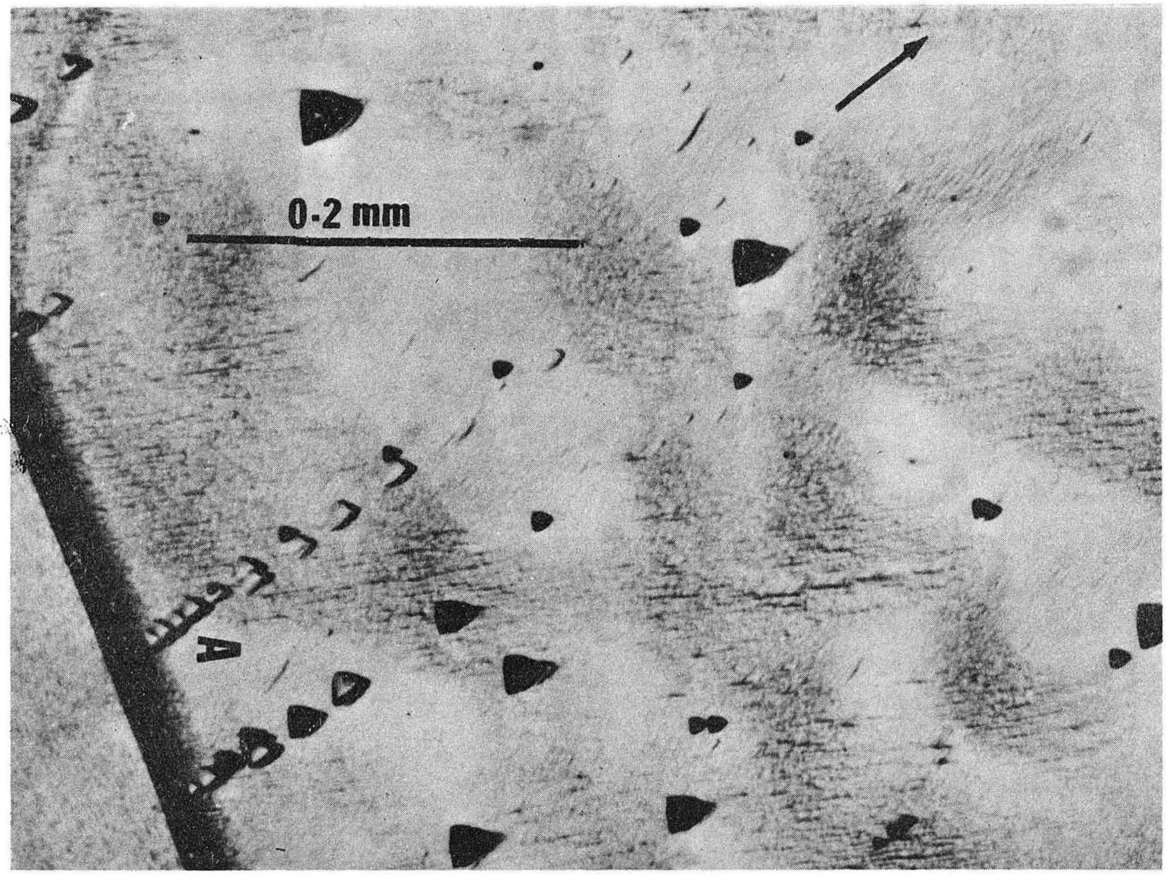


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Figure 5

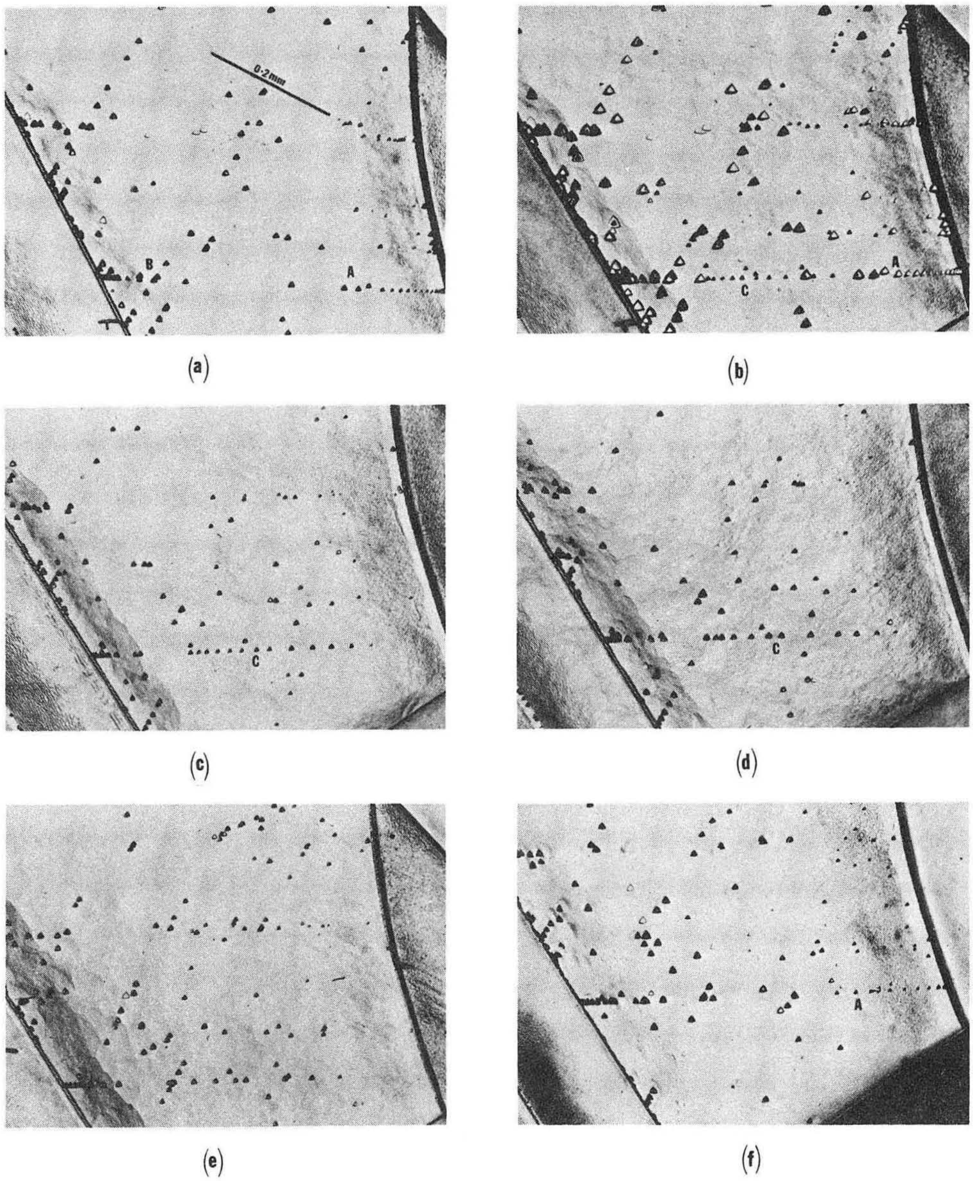

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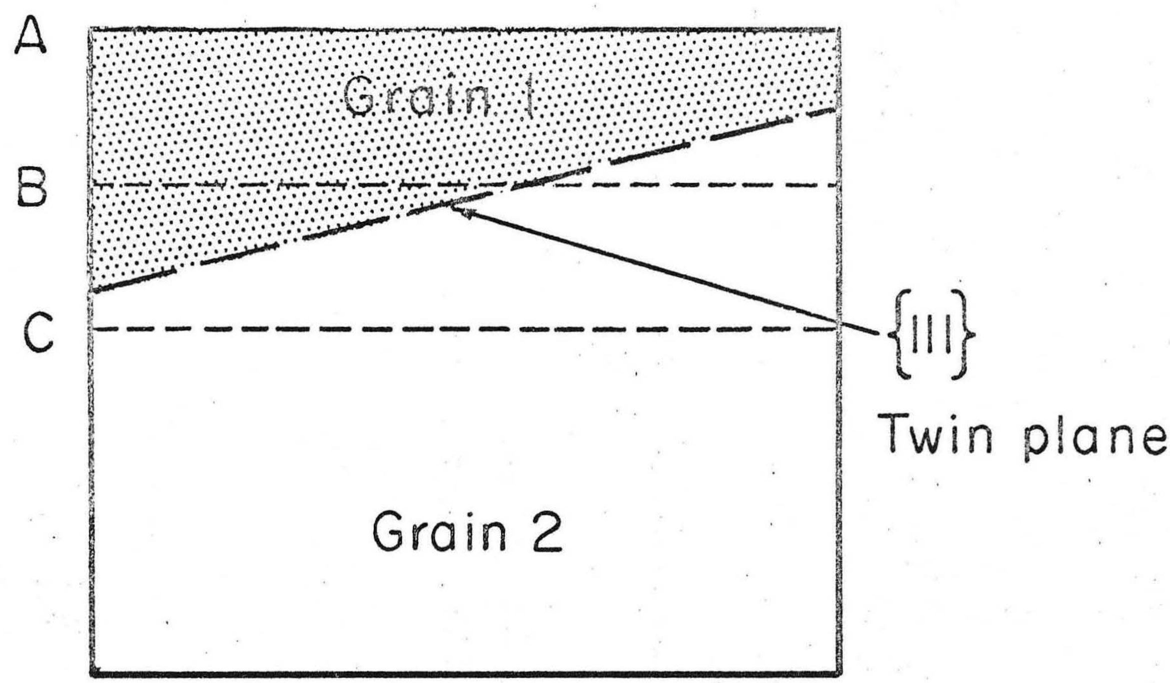
Figure 6



XBL687- 3326

Fig. 7

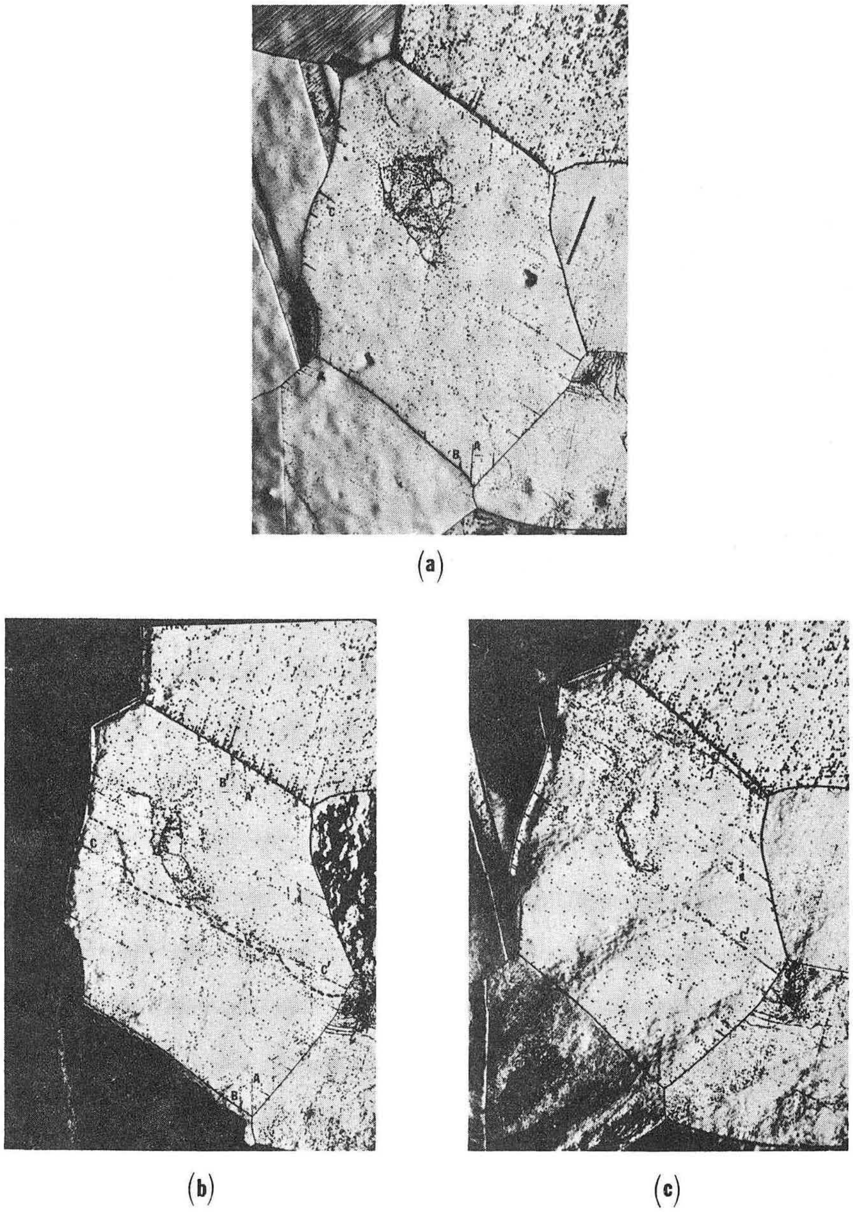


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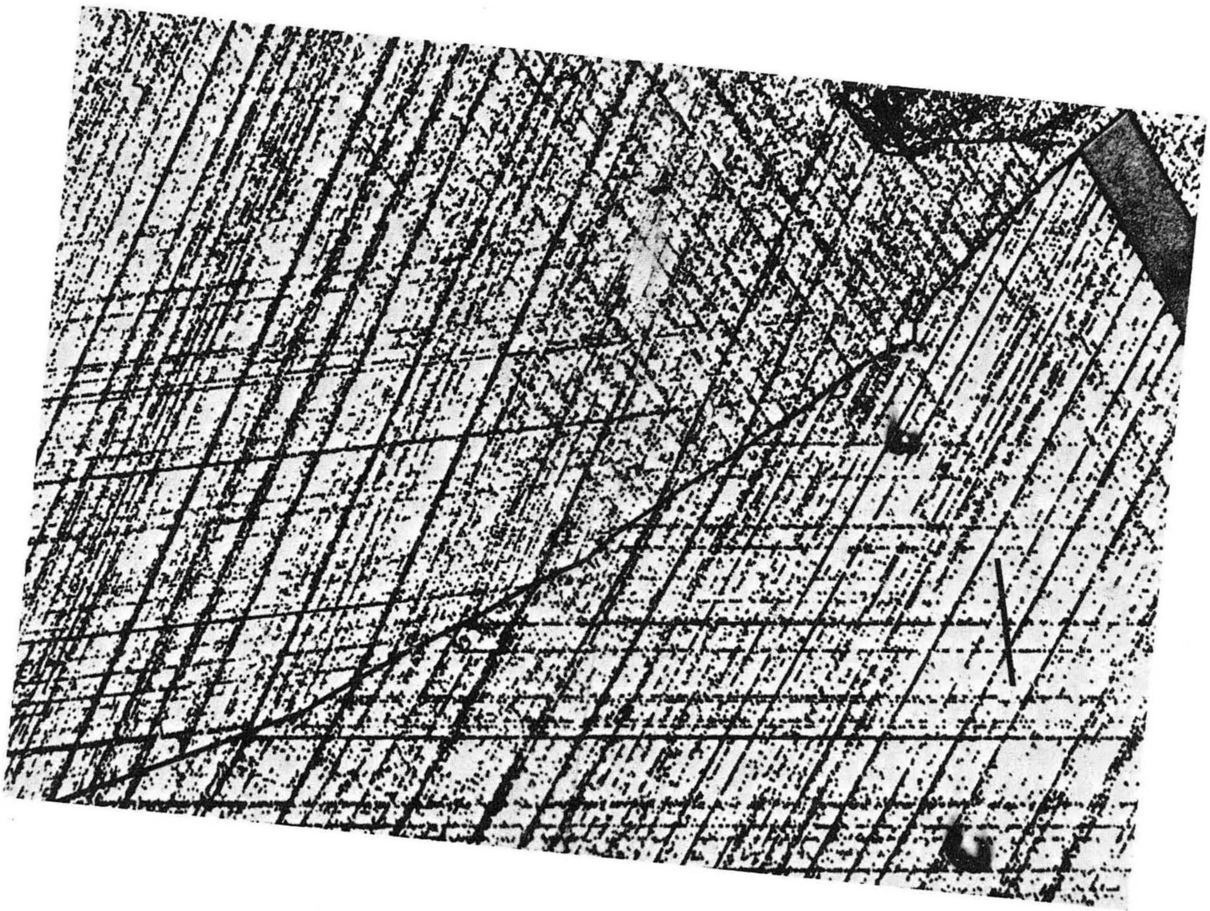
Figure 8



XBB 688-4983

Figure 9

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