University of California

Ernest O. Lawrence Radiation Laboratory

DISLOCATION MOTION, MULTIPLICATION, AND INTERACTIONS IN THE PREYIELD REGION OF COPPER POLYCRYSTALS

Gopinathan Vellaikal

(Ph. D. Thesis)

September 1968

Berkeley, California

DISCLAIMER

UCRL-18435

UNIVERSITY OF CALIFORNIA
Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

DISLOCATION MOTION, MULTIPICATION, AND INTERACTIONS IN THE PREYIELD REGION OF COPPER POLYCRYSTALS

Gopinathan Vellaikal

(Ph.D. Thesis)

September, 1968

TABLE OF CONTENTS

DISLOCATION MOTION, MULTIPICATION, AND INTERACTIONS IN THE PREYIELD REGION OF COPPER POLYCRYSTALS

Gopinathan Vellaikal

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering, of University of California, Berkeley, California

ABSTRACT

Direct observations of dislocation motion, multiplication and interactions were made, using the etch-pit technique, in the preyield region of large grained copper polycrystals. The specimens were specially prepared so as to overcome the usual etching restrictions in copper and deformed in compression or bending. The use of a microcompression device enabled the observation of dislocation arrangements in the stressed condition both at and below the surface.

The results are consistent with previously reported results regarding the influence of the degree of jogging of dislocation segments on their mobility.. Heavily jogged dislocations did not seem to take part in the early multiplication process. The generation of many dislocations by the multiplication of even comparatively less jogged dislocation segments in an annealed crystal appeared possible only under special situations where the source could operate in an unsymmetric way. It is suggested that such special situations are more easily developed near a free surface than in the interior of a grain thereby causing the generally observed higher dislocation activity near the surface regions of a crystal.

The primary role of grain boundaries in the preyield region of copper was shown to be as barriers to moving dislocations and not sources of dislocations. However, direct evidence was obtained for the passage

of dislocations through certain special boundaries; the possible importance of such boundaries in the usual grain size dependence of yield stress of polycrystals is discussed.

Freshly generated dislocations in the early deformation stages have been found to move very large distances both during loading and unloading. Internal barriers responsible for holding up these dislocations were probably Lomer-Cottrell dislocations formed because of the unavoidable traces of secondary slip. A tentative mechanism based on the collapse of Lomer-Cottrell dislocation locks is proposed to qualitatively explain the phenomenon of macroscopic yielding in single crystals.

I. INTRODUCTION

It is well known that high-sensitivity strain measurements or dislocation etch-pit observations can detect small plastic strains far below the macroscopic yield stress in many metals and alloys. With a strain resolution of the order of 10^{-9} Tinder and Washburn[1] detected measurable plastic strains in tubular polycrystalline OFHC copper at stresses as low as $2 g/mm^2$. Using etch-pit techniques on high purity copper single crystals many investigators[2-6] have observed appreciable dislocation motion and multiplication much before yielding. Apart from its importance in understanding the phenomenon of yielding itself, direct observation of dislocation behavior in the pre-yield region provides the best method for a detailed study of dislocation motion, multiplication and interactions. A significant amount of information on these various aspects has been obtained from the many etch-pit studies referred to earlier and also more recently by x-ray topography studies[7-9] on copper single crystals. There have, however, been very few attempts to directly observe, by the etch-pit technique, the behavior of dislocations within the grains of a polycrystal during plastic deformation. This is primarily due to the fact that dislocations in copper can be revealed by etching only when a low-index plane is parallel to the surface of observation. In general this restriction implies that in a fairly large-grained polycrystalline specimen the chances of being able to observe the dislocations in any surface grain are extremely rare. Special techniques are thus needed to prepare specimens that will have one or more surface grains in an etchable orientation. In spite of the obvious experimental problems, direct observation of the dislocation behavior in polycrystals should be of interest for the following reasons:

1. Such observation provides a natural extension of the many previous single crystal studies and should serve to confirm, contradict and/or clarify the various conclusions drawn from these experiments.

2. The initial dislocation structure in the recrystallized grains of a polycrystal could be different from that found in single crystals that are grown from the melt and annealed. This might lead to significant differences in dislocation behavior in polycrystals from that hitherto observed in single crystals.

3. The possibility that grain boundaries act as the sources of dislocations in the early stages of slip can be directly checked.

4. Grain boundaries might greatly facilitate the detection of the beginning of plastic deformation by serving as effective barriers to the moving dislocations. Also the resulting dislocation pile-ups should serve to identify the early regions of slip and thus help detect and analyze the nature of any damage produced in the slipped areas in the wake of these early moving dislocations.

5. Experiments using polycrystals should provide unique opportunities for detailed study of dislocation pile-ups such as their behavior under stress relaxation, stress reversal, etc.

6. Also polycrystals provide the chance to identify, separate or avoid possible complications in the observed dislocation distribution due to the presence of a free surface by studying both surface grains and grains that were totally enclosed during the deformation.

7. Finally if two or more neighboring surface grains in a polycrystal happen to be in etchable orientations it might be possible to observe instances of slip continuity across the grain boundaries and to study the conditions under which they occur.

In the present experiments many of the above aspects have been studied in some detail using compressive and bending deformations on fairly large grained polycrystalline OFHC copper specimens that were prepared by special techniques designed to overcome the previously mentioned etching restrictions.

II. EXPERIMENTAL PROCEDURES

A. Preparation of Specimens

As indicated in the introduction the primary experimental problem was to prepare specimens having at least one surface grain with a low index plane parallel to the surface of observation. Since most reliable etchants have been developed only for planes of the {111} type in copper[10] experiments were aimed at obtaining one of these planes in a grain parallel to the surface. Two different techniques were employed for this purpose.

In the first technique the starting material was an extruded OFHC copper rod with a square cross-section of 2cm × 2cm. The main impurities and their level are given in Table I.

Table I. Analysis of OFHC copper

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

Specimens 3 cm long were cut from this rod and annealed at about 1060°C for 48 hours in a vacuum of less than 10^{-5} mm of Hg. This treatment produced large grains with an average size of 5 mm. The next step was to find a grain on one of the 3cm × 2cm surfaces that had a {111} plane approximately (within about 10 degrees) parallel to that surface. The search for such a grain was considerably narrowed down by the presence of a large number of twins in the annealed specimens. Since the twinning plane in copper is of the {111} type the straight twin traces visible on the surfaces of the specimen 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 often reduced to finding a grain having three (or sometimes only 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 cut out using an acid saw.[11] A goniometer was used to hold the specimen and tilt it to the exact orientation. The resulting slice was further suitably cut on the acid saw to get specimens approximately 2cm × 1cm × 1cm with a central grain on one of the 2cm × 1cm faces having a {111} plane parallel to the surface. The orientation of the grain was again checked by x-rays and then polished to within less than a degree off {111} on a chemical polishing wheel.[11] using a solution of the following composition:

50 parts nitric acid

25 parts acetic acid and

25 parts phosphoric acid

The two 1cm × 1cm faces were also planed on the polishing wheel and used as the compression faces.

The second technique was based on the observation that high temperature recrystallization of extruded OFHC copper rods often resulted in the preferential formation of a large number of grains with one of their {111} planes approximately normal to the extruding direction. The method of preparation of specimens thus simply consisted of machining specimens of the required size and shape (see later) from the extruded rods, chemically polishing the surface that was parallel to the original 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. Very often there were more than one grain in an etchable orientation and when they happened to be neighbors, as they frequently did, phenomena like slip continuity across grain boundaries could be studied. The great advantage of this method was the saving of a considerable amount of time that would normally be involved in the acid-sawing and wheel-polishing operations necessary in the first technique and also the avoidance of any surface damage that might be incurred during these operations, however minor they might be. Experimental results to be reported later did indicate significant differences in dislocation behavior in crystals produced by the two techniques. In general results from the second type of crystals were more characteristic of an unperturbed three-dimensional network of dislocations. In view of the simplicity of the technique and the consistently higher perfection and freedom from surface damage in the resulting specimens, most of the compression and bending experiments to be reported in this work were done using specimens prepared by the second technique unless mentioned otherwise.

B. Compression Experiments

Specimens for compression prepared by the first technique had dimensions approximately 2cm × 1cm × 1cm with the 1cm × 1cm faces being the compression faces as already described. Compression specimens prepared by the second technique were usually cube shaped with sides equal to 3/4 inches and were machined from 3/4 inch square extruded rods. The surface of observation was always the one normal to the extruding direction.

Compression experiments were performed at room temperature using either an Instron machine (with a strain rate of approximately 0.0005/min) or a specially constructed micro-compression device schematically illustrated in Fig. 1. The device is made of stainless steel and is essentially like a C clamp in which, by turning the micrometer head, an increasing stress can 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-type joint at the head of the spindle ensures proper alignment and a uniform distribution of the load. The stainless steel plate (marked P in Fig. 1b) kept flush against the vertical arm prevents any torque being transmitted to the specimen during loading. The two teflon plates serve to electrically insulate the specimen during electropolishing. Insulating lacquer is 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 rough estimate 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 caused by hanging known weights from one of the end plates. Although it was difficult to know exactly the beginning of application of stress, incremental increases in stress could be applied reasonably precisely.

The particular advantage of the microcompression device was that specimens could be etched and observed under the microscope while under stress thus permitting the study of dislocation behavior under increasing stresses or stress relaxation. Also, it enabled the observation of the dislocation distribution below the surface of a specimen in the stressed condition by immersing the whole device in the polishing solution (chemical or electropolishing) and reetching. Furthermore specimens could be deformed while immersed in the electropolishing solution and etched before and after drying so as to indicate possible effects of any surface films formed on the surfaces of specimens during the normal drying operation after electropolishing.

Electropolishing was always carried out at room temperature using a solution of 60 parts phosphoric acid and 40 parts water at a cell voltage of 1.5 volts and at a current density of approximately 0.1 amps/cm^2. The etchant used to reveal the dislocations was the one developed by Livingston[10] and consisted of

1 part bromine

15 parts acetic acid

25 parts hydrochloric acid and

90 parts water.

Unless otherwise mentioned the amount of material removed from the surface between successive etchings was always of the order of 5-10 microns.

Specimens compressed in the Instron machine were etched only in the stress relaxed condition. Those compressed in the micro-device were etched in the stressed or stress-relaxed condition depending on the experiment. A double etching technique* was used whenever the new dislocations were to be related to the old ones. The observations of etch-pits were made with a Carl-Zeiss optical bench metallograph.

C. Bending Experiments

The dimensions of the specimens used in the bending experiments and their relation to the extruding direction of the starting material (1-1/4 inch square rod) are indicated in Fig. 2. The bending specimens were prepared by the simpler recrystallization technique described earlier.

A cantilever beam-type bending was employed by clamping the specimen securely in a vise at the left end and carefully attaching the necessary

---

* In a double-etching experiment the crystal is etched before and after applying a small stress. The second etch usually reveals three kinds of pits. There are large sharp bottomed pits, large flat bottomed pits and small sharp bottomed pits. The large sharp bottomed pits represent the sites of dislocations unmoved by the stress that was applied between the two etches. The large flat bottomed pits show the original position of dislocations that were moved by the stress and the small sharp bottomed pits show their final position.

weights on a flexible rubber band running through the shallow V-grooves at the right end of the specimen . The V-grooves 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 made on grains sufficiently distant from the clamping end whenever possible. However it was generally found that there was perceptible damage only in the grains directly in contact with the vise. The specimens were etched before, during or after the loading depending on the nature of the particular experiment.

D. Observation of the Dislocation Distribution Below the Surface

Material removal up to a depth of about 1 mm below the surface was normally accomplished by using the same chemical polishing solution as used for the wheel-polishing. The rate of removal of material was about 8-10 microns per minute. For observations at depths of more than 1 mm it was more convenient to use a modified polishing apparatus as shown in Fig. 3. The principle is basically the same as in the polishing wheel except that the specimen is held in a vertical position and the thin film of the polishing solution on the wheel cloth is replaced by a pool of the same solution in a large dish. There are provisions for rotation as well as vertical motion of the specimen holder at variable speeds. Material removal is usually accomplished by maintaining the specimen surface just below the liquid level. The rate of material removal was again about 8-10 microns per minute.

When still larger amounts of material had to be removed from the surface (as was usually necessary in the study of completely enclosed grains) considerable saving in time could be achieved by initially using 1:1 nitric acid to remove most of the necessary material followed by a final polishing in the chemical polishing solution.*

---

* The alternate method of sectioning the specimens using the acid saw and wheel polishing the resulting slices was not preferred since many observations on specimens so prepared suggested the possibility that the dislocation structure was somewhat modified through minor damages incurred during the above operations.

III. RESULTS

A. Nature of the Recrystallized Grains

Apart from their general preference to have a {111} plane nearly normal to the extruding direction the most characteristic feature of the recrystallized grains was their relatively high perfection. The dislocation densities were always less than $10^4/cm^2$ and frequently of the order of $10^3/cm^2$. The dislocations were quite uniformly distributed and there was usually little evidence of subboundaries. These observations are consistent with the generally observed high perfection of copper crystals grown by secondary recrystallization as revealed by the Schulz x-ray technique.[12] This structure is, however, to be contrasted with that generally found in single crystals that have been grown from the melt and annealed. In the latter the dislocation structure usually consists of regions of low dislocation density surrounded by well developed subboundaries. Although no direct observation (by an x-ray topography technique, for instance) of the internal dislocation structure of the grains in recrystallized copper has yet been made the above observations seem to indicate that their dislocation structure should approach more closely an ideal three-dimensional Frank network. There was no significant variation in dislocation density as a function of depth from the free surface. Even inner grains which had no free surfaces during the annealing exhibited extremely low dislocation densities.

B. Dislocation Motion and Multiplication

Double etching experiments on specimens deformed by compression or bending generally showed no instances of simple dislocation motion until the resolved shear stress was raised to about 15-20 g/mm^2. Figures 4 and 5 show grains in the double etched condition in specimens subjected to a compressive stress of about 25 and 40 g/mm^2 respectively.* The second etching in both cases was done after removal of the applied stress. No dislocation motion is evident in Fig. 4. However many cases of simple dislocation motion can be observed in Fig. 5. A few of them are indicated by the small black circles.** It can be seen that the distances of such individual dislocation motion are generally small and fairly uniform.

The beginning of dislocation multiplication was usually inferred from the appearance of small groups of dislocations piled up at the

---

* All stresses reported are applied stresses unless mentioned otherwise. Also the direction of applied stress is indicated in all micrographs by a black line of length 0.5 mm, unless shown otherwise.

** At the comparatively low magnifications employed in the present experiments large flat-bottomed pits formed after a double-etch sometimes appeared as white pits and large sharp bottomed ones as black pits. The fact that the large white pits did indeed represent the original position of moved dislocations was confirmed by the absence of any etch pits at those places on subsequent electropolishing and etching.

grain boundaries.* Many dislocation pileups can be seen in Fig. 5 and also in Fig. 6 the latter showing only a few representative examples of some very commonly observed structures after dislocation multiplication had started. In general a resolved shear stress of about 15-20 g/mm^2 was enough to produce such pileups so that it was hard to observe in a double etching experiment simple dislocation motion without dislocation multiplication taking place at the same time. One significant aspect of dislocation multiplication evident from many double etched micrographs was the frequent inability to detect the source dislocation responsible for the early dislocation pileups. The source was usually not a dislocation originally intersecting the surface as can be seen clearly from Fig. 7 which shows a double etched grain in a specimen subjected to a compressive stress of approximately 50 g/mm^2. The group of dislocations marked A and B indicate that dislocation multiplication has occurred. However a careful examination of the two slip plane traces corresponding to pileups A and B along the entire length of the grain fails to show any large flat bottomed pit which might represent the original position of a surface-intersecting dislocation that might have moved and multiplied.

Also when dislocation pileups could be detected at the opposite boundaries of a grain there was usually an almost one-to-one corre-

* For the present purposes a pileup is defined as a group of dislocations which apparently lie on a single slip plane.

spondence between them as seen in Fig. 5 and also in examples (a) and (b) of Fig. 6.

C. Dislocation Structure of the Pileups

Although the dislocations in some of the pileups appeared to lie in the same slip plane as far as could be judged at the low magnifications employed there was, in a great number of cases, considerable deviations from a strictly linear array. Figures 8(a) to 8(d) show a few particularly good examples of such deviations as seen by etching specimens in the stressed condition after deformation in the micro-compression device to stresses of approximately 40, 60, 50, and 50 g/mm^2 respectively. Very often it appeared as though the dislocations in a pileup belonged to a few distinct slip planes. The high frequency of such observations suggests that even in an apparently linear pileup the dislocations might actually belong to separate slip planes too close to be distinguished at the relatively low magnifications employed in the present experiments. The significance of this observation with regard to the dislocation multiplication mechanism will be discussed later.

D. Stress-Relaxation of Dislocation Pileups

There was considerable stress relaxation in the case of all dislocation pileups formed in the very early stages of plastic deformation. The extent of stress relaxation and the nature of the resulting dislocation configurations strongly depended on the detailed structure of the individual dislocation pileups, their proximity to each other and also on whether there was dislocation activity on any intersecting slip system or not. When there was no significant overlapping of dislocations in a pileup they moved back very large distances on stress relaxation as illustrated by the pileup A in Figs. 9(a) and 9(b) which show a grain in a specimen subjected to a compressive stress of approximately 50 g/mm^2 and etched in the stressed and stress relaxed conditions respectively. The many small etch pits along the direction of the arrow in Fig. 9(b) represent the stress relaxed positions of the dislocations belonging to the "upper half" of the pileup A in Fig. 9(a). The dislocations belonging to the "lower half" of the pileup have apparently moved far back beyond the field of view of Fig. 9(b). When the pileups contained overlapping dislocations belonging to fairly distinct slip planes, stress relaxation often led to arrangements where many dislocations in one slip plane took up positions vertically above those in the other as seen at A in Fig. 10 which shows two separate grains in a specimen etched in the stress-relaxed condition after a compressive stress of approximately 55 g/mm^2. When there were a large number of distinct but close pileups formed against a grain boundary nearly at right angles to the slip plane trace stress relaxation often resulted in appreciable "glide polygonization"* as shown in the sequence of Fig. 11 which shows a grain in a specimen etched under a stress of approximately 52 g/mm^2 and later etched after stress relaxation. The large amount of stress relaxation that has occurred can be particularly well seen by comparing the pileup A in Figs. 11(a) and (b), and also from the large

* By the term "glide polygonization" is meant the short range alignment of many (edge) dislocations in a direction perpendicular to the slip plane trace that takes place at temperatures too low for climb to have occurred.

depletion of dislocations in the area B of Fig. 11 (b). When the grain boundary was at a much smaller angle to the slip plane trace there was usually no tendency for glidepolygonization to occur. This is clearly seen from Fig. 12 which shows the dislocation configurations formed on stress-relaxation at the two different kinds of boundaries after a compressive stress of approximately 55 g/mm^2. A strong one to one correspondence of the dislocation pileups is also obvious across the top and bottom boundaries of the grain.

Whenever there was dislocation activity on more than one plane it was generally observed that when the applied stress was removed the relaxing dislocation loops on the different planes interacted locally to form stable dislocation groupings thus preventing the complete relaxation of the pileups. Examples of such local interaction and stabilization of the relaxing pileups can be seen at A in Figs. 12 and 13, the latter showing a grain in a specimen etched in the stress relaxed condition after a compressive stress of approximately 55 g/mm^2.

E. Behavior of Dislocation Pileups on Stress Reversal

An experiment was conducted to study the behavior of dislocation pileups on reversal of the direction of the applied stress. In particular it was hoped to find out whether dislocations in a pileup ran back into their original source or whether they were annihilated by dislocations of opposite sign generated by the source under the reversed stress. As already mentioned in the experimental procedures a stress-reversal is accomplished by simply turning the specimen upside down and applying the same load as before.

Figure 14 (a) shows a grain on the top surface of a specimen deformed in bending to a stress of about 20 g/mm^2 and etched in the stress-relaxed condition. This specimen had been chemically polished for about 20 minutes before the deformation so that inside dislocation segments were probably exposed. The dislocation groupings A and B represent the opposite halves of the same dislocation pileup. The reason why the pileups have not completely relaxed may be partly due to interaction with dislocations of another slip system evident at the left side of the grain. However, on application of an equal load in the opposite direction, the dislocations in group A undergo considerable reverse motion as seen froms Figs. 14 (b) and (c) which show the same grain after etching in the stress-relaxed condition and after removal of about 20 microns of material from the surface by chemical polishing, respectively. The new positions of the dislocations are at C in Figs. 14 (b) and (c). The fact that they have indeed moved away from the grain boundary on the right is indicated by the flat bottomed pits A in Fig. 14 (b) and the disappearance of these pits on surface polishing as seen from Fig. 14 (c). The group of dislocations B in Fig. 14 (a) does not however seem to have moved presumably because of strong interaction with some secondary dislocations. Figure 14 (d) shows the same grain after increasing the applied stress to about 29 g/mm^2 and etched after stress relaxation with an intermediate polishing off of about 20 microns of material before etching. There is still no indication of dislocations of opposite sign being generated from the original source leading to partial annihilation of dislocations of the original pileups. It is also not possible to make all the original dislocations run back into their source even at an applied stress of about

40 g/mm^2 (twice the original stress) as seen from Fig. 14 (e). When the same stress of 40 g/mm^2 was then applied in the opposite direction the dislocations again moved back to the boundary on the right as shown by the pileup A in Fig. 14 (f). There is however a small reduction in the number of dislocations in the pileup A in Fig. 14 (f) from that in Fig. 14 (a). A decrease in the number of pits may also be seen while comparing the dislocation group C in Figs. 14 (c) and (d). Such loss of dislocations are actually due to a polishing off of some semicircular loops (as will be discussed later).

F. Slip Continuity Across Grain-Boundaries

The results of the present studies indicate that grain boundary propagation of slip does not usually take place in copper in the microstrain region except in some special cases where dislocations in one grain can actually pass through the boundary into the next grain. So far the only definite instances of such grain boundary propagation have been found only across coherent twin boundaries. The reason why in spite of the etching restrictions in copper direct observation of such dislocation propagation 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. As schematically illustrated in Fig. 15, the surface of observation A is inclined at a small angle to the {111} twin plane. Etching of plane A reveals the dislocations in the top grain only. However when the surface is polished down to level B and etched dislocations in parts of both the top and bottom grains would be simultaneously revealed. Finally when the surface is polished down to C, only dislocations in the bottom grain can be seen. The changes in the position of the etching planes will also be reflected in the relative orientation of the etch pits as indicated in Fig. 16 where (a), (b) and (c) correspond to levels A, B and C of Fig. 15. If the plane of the paper represents the twinning plane then the relative orientations of the other {111} planes in the two grains will be as shown in Fig. 17 with points D and D' both lying below the plane of the paper. When the active Burgers vector is parallel to the twinning plane i.e. is either AB, BC or CA, then it will have, along the twin boundary, a common {111} plane in each grain. When a dislocation loop P with Burgers vector AB expands on the plane ABD of grain 1 those segments of the loop pressed against the twin boundary will acquire a screw character as at MN. Those segments should then be able to transfer themselves onto the plane A'B'D' of grain 2 and continue expanding basically as in cross-slip. Under these conditions twin boundary continuity of slip will be indicated by the persistance of an almost collinear pileup across the grain through the stages represented by A, B and C of Fig. 15. This is clearly illustrated by the pileups A, B and C in the sequence of Fig. 18 which shows the same grain 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 from the surface; and (c) after removal of an additional 50 microns of material from the surface.

Figures 18 (a), (b) and (c) correspond respectively to levels A, B and C of Fig. 15. For clarity the position of the twin boundary in Fig. 18 (b) is marked by a dashed line. Although the region above the twin boundary in Fig. 18 (b) now repsents a different grain from that in Fig. 18 (a), one can still see in it pileups A', B', and C' to match the original pileups A, B and C in the region below the twin boundary. The slight deviation from linearity of the dislocation arrays AA' and BB' across the boundary in Fig. 18 (b) is simply due to the definite deviation of the surface of observation from a {111} plane of the grain. When still more material is removed from the surface so as to completely expose the originally underlying grain (Fig. 18 (c)) one is still able to observe a pileup C" essentially at the same place as C in Figs. 18 (a) and (b) and matching the pileup C' in Fig. 18 (b). The change in the orientation of the etch pits, as expected from Fig. 16, can be seen through the sequence of Fig. 18.

It might be mentioned that the observed twin-boundary continuity of slip is only a special case of the more general slip continuity observed by Ogilvie[13] in aluminum and brass across straight boundaries when the lines of intersection of the slip plane with the boundary are within about two degrees of one of the directions ⟨110⟩, ⟨112⟩ or ⟨123⟩ for each grain, not necessarily the same direction in adjacent grains. The above discussion does not imply however that twin boundaries never act as strong barriers to slip. They certainly do so whenever the operating Burgers vector does not lie in the twinning plane.

There were also a few isolated instances where some slip continuity was evident across a low angle boundary. A typical example is seen in Fig. 19, which shows a low angle grain boundary in a specimen etched under a compressive stress of about 60 g/mm^2. Many matching slip bands can be seen across the boundary. An analysis of the orientations of the two grains by the back reflection Laue method indicated that they differed by as much as 7 degrees. However the lines of intersection of the active slip planes in the two grains with the almost straight sections of the boundary were nearly always coincident. Unlike in the twin boundaries, however, the line of intersection in this particular example was not exactly $\langle 110 \rangle$, but almost 4 to 5 degrees off.

IV. DISCUSSION

A. Mechanism of Recrystallization

It has not been possible to get a definite idea as to the mechanism of formation, on annealing, of large number of grains with a {111} type plane normal to the extruding direction of the starting OFHC copper rods. Diffractometer and transmission Laue experiments showed that the original rods had a very strong ⟨111⟩ fiber texture similar to drawn f.c.c. wires. Drawn wires are known to retain their deformation texture even after high temperature annealing.[14] There is no reported data on the annealing texture of extruded rods. In view of the similarity of the deformation textures of drawn wires and extruded rods it is conceivable that the rods also retain their deformation texture on high-temperature annealing. Recrystallization and grain growth under these conditions should necessarily lead to the formation of many large grains with a {111} type plane normal to the extruding direction.

B. Jog Density and Dislocation Mobility

One of the significant observations in the present experiments was the considerably higher stress (10-15 g/mm^2) needed for the motion of dislocation segments in OFHC polycrystalline copper as compared to a stress of only 2-4 g/mm^2 in 99.999% copper single crystals.[5,6,8] Though part of this difference may be due to an impurity pinning effect many aspects of dislocation motion and multiplication in the present experiments seem to suggest that the basic factor controlling early dislocation motion is the degree of jogging of the dislocation segments.

At high temperatures the dislocation network in a crystal would tend to approach a configuration of metastable equilibrium by both conservative and non conservative motion. Assuming that the energy associated with a jog is small elastic strain energy is minimized when dislocation segments approach linearity and nodes become symmetrical. In a metal of medium stacking fault energy like copper, few dislocation segments in the annealed or recrystallized specimens will lie exactly on low index glide planes; in other words most of the dislocation segments will be jogged. When dislocation segments intersect a free surface there is usually an additional surface image force which tends to further shorten their lengths and hence increase their jog density by causing them to become normal to the surface. Direct observations of dislocations in annealed copper crystals by x-ray topography do support this picture. Consequently in those experiments in the present investigation where only about 5 microns of material were removed from the surface after the recrystallization treatment and prior to the initial deformation most of the dislocations intersecting the surface should have been heavily jogged and hence require the comparatively higher motion stresses observed. Under these conditions dislocation segments inside the crystal with both their ends fixed should be less jogged than the surface segments and so should move and multiply at lower stresses. This indeed appeared to be the case because, as mentioned earlier, whenever dislocation pileups were first detected in a double etching experiment the source dislocations were never those originally intersecting the surface as indicated by the absence of any flat bottomed pits along the slip plane traces of the pileups. The pileups should thus have resulted from the operation of dislocation segments with both ends fixed inside the grain and hence not revealed on the first etching. It is interesting to note in this connection that in Young's and Marukawa's experiments also dislocation multiplication was inferred only from the appearance of "groups" of dislocations and/or a general increase in dislocation density but that no attempt was made to correlate the new dislocations with any dislocations that originally intersected the surface. Also the stress needed to cause multiplication in their experiments corresponded more closely to that calculated for disloactions with both ends fixed within the crystal. Such preferential multiplication of inside dislocationsin spite of an approximate factor of two reduction in the theoretical stress needed to cause multiplication of a single ended surface source strongly indicates that the degree of jogging of dislocation segments basically controls their motion and multiplication in the early stages of deformation.

The importance of jog density is also shown by the recent experimental observations by Petroff and Washburn[5] that heavily jogged dislocations even in 99.999% copper do not move at an appreciable rate at stresses of 50 g/mm^2.

The present results suggest that even in many previous pre-yield experiments in copper single crystals only accidentally introduced dislocations or dislocations that had already been moved by handling of the specimens could have moved and multiplied at the reported low stresses of $2g/mm^2$ and $7g/mm^2$ respectively. A closely related problem is the possible influence of a free surface on the characteristics of plastic deformation as will be discussed next.

C. Surface Effects

It is well known that even in specimens with conventionally clean surfaces there exist appreciable differences in plastic behavior between the surface and the inside regions. Such differences have usually been explained on the basis that the free surface may act either as an impediment to the motion of dislocations or as a ready source or sink of dislocations.[15] Of particular interest to the present investigation was the question whether, at the very low stresses employed, the surface was indeed the site of the early dislocation multiplication and if so, why?

Although some recent etch-pit experiments (S. Kitajima, private communication) and x-ray topography experiments[9] in the preyield region of copper single crystals have shown direct evidence for the operation of surface sources, no satisfactory explanation has been given as to their origin. Young[9] however noted that all dislocations that moved and multiplied from the surface could have been accidentally introduced during handling.

In general there are many reasons why a surface might act as a source of dislocations. Irregularities such as scratches and etch-pits can provide stress concentrations at which plastic flow is initiated. So also might any films left behind in cases where specimens are electropolished and dried before the deformation. Even when an atomically smooth surface is present there can be an enhanced dislocation activity near the surface because dislocations intersecting a free surface can theoretically start multiplying by a Frank-Read mechanism at stresses half that needed to operate an interior source with both ends fixed.[16] In the present experiments the chances of accidentally introduced dislocations acting as surface sources had been practically eliminated since there was no acid-sawing or wheel-polishing involved after the recrystallization treatment. The only necessary electropolishing was usually carried out with the specimen already mounted in the micro-compression device. The commonly observed non-correlation (in the double etch experiments) of the dislocation pileups with any of the pre-existing etch pits indicates that etch-pits themselves are not the sources for early dislocation multiplication. The possibility of electropolishing films leading to random nucleation sites on the surface has also to be discarded because when specimens were repeatedly electropolished and etched under constant stress there were no instances of new dislocation pileups apart from those originally present. If random nucleation sites were created by any film one should expect new dislocation pileups after each electropolishing and etching. The same conclusion was also reached from experiments where specimens were deformed while immersed in the electropolishing solution and etched before and after drying. Figure 20 shows a grain etched in the two conditions under a stress of approximately $40 g/mm^2$. Apart from an increase in the size of the pits there was no dislocation motion or multiplication induced on drying. Also since about 10 microns of material were usually removed from the surface of the specimen by electropolishing before any deformation was carried out the surface should have been smooth on a microscale and so any gross surface-roughness could not have been directly responsible for the early dislocation multiplication. The above results indicate that in copper specimens carefully prepared to avoid any accidentally introduced damage the surface, by itself, is not a source of dislocations.

The early dislocation pileups observed in the present work should thus have resulted from the multiplication of segments of the grown-in dislocation network.

It is not clear exactly what is the nature of the damage introduced on the surfaces of specimens or how exactly they are introduced. In specimens prepared by the first technique, it is conceivable that the unavoidable surface contact during the acid-sawing or wheel-polishing operations introduced damage in the form of dislocation half-loops too small to be visible with an otpical microscope. The loops may be similar to those discussed by Johnston and Gilman[17] in connection with the plastic flow in LiF crystals and more recently by Worzala and Robinson[18] in connection with the distribution of dislocations during stage I deformation of silver single crystals. The expansion of such half loops might provide a natural explanation for the very shallow elongated loops observed by Young[9] near the surface of lightly deformed copper single crystals using x-ray topography. Similar loops near the surface were also indicated from successive polishing and etching experiments during the present investigation on lightly deformed specimens prepared by the first technique. Such loops never seemed to be present in lightly deformed specimens prepared by the second technique. Nor were they observed in those x-ray topography experiments of Young using crystals handled with special care. It thus appears that the surface sources of dislocations are really only accidentally introduced dislocations on the surface in the form of half loops or other configurations. When surface contact is minimized after growth, as in the present experiments, surface sources can be completely avoided and the observed dislocation distribution should then be the result of the operation of only grown-in dislocation segments.

The possibility remains, however, that the details of the operation of even a grown-in dislocation segment may be considerably modified by the presence of a free surface leading to significant differences in the nature of plastic deformation in the bulk of the specimen and at a damage-free surface. As already discussed, the presence of a free surface during high temperature annealing leads to considerable immobilization of the many surface intersecting dislocation segments by causing them to acquire a comparatively higher jog density. However, even the multiplication of inner dislocation segments was able to produce a considerable amount of plastic deformation at the surface as indicated by the many large pile-ups observed in the early stages of deformation. On the other hand, examination of many grains that were totally enclosed during deformation showed no evidence of such dislocation pileups even at stresses two or three times higher than those at which pileups were observed at the surfaces.* These observations indicate that the surface is indeed a region of enhanced slip as compared with the inside of a crystal. As will be shown later this is primarily due to a modification of the slip propagation mechanism itself at the surface rather than due to a lowering of the theoretical critical multiplication stress of a single ended surface source.

*Examination of the inner grains was made after polishing off the necessarily large amount of material from the surface using 1:1 nitric acid in the modified polishing apparatus shown in Fig. 3.

D. Grain Boundary Sources

The results of the present investigation gave no evidence for grain boundaries in copper acting as sources of dislocations in the early stages of deformation. Their primary role seemed to be to act as barriers to dislocations generated from segments of the grown-in dislocation network. The almost one-to-one correspondence of the pileups found against the opposite boudaries (as seen in Figs. 6 and 12) could hardly result from a random nucleation of dislocations from these 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 by a single grain boundary source that was 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 end pileup. Figure 21(a) shows such a pileup in a specimen subjected to a stress of approximately $50 g/mm^2$ and etched in the stress relaxed condition. The fact that one group of dislocations appears as dark pits and the other as light pits,[19] and also the collinearity of the two groups suggest that they mark the points of emergence of opposite segments of the same dislocation loops. Figure 21(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 pileups increased and finally both sets of pits disappeared after removal of about 100 microns of material. These results indicate a three dimensional configuration of the dislocations that may be schematically represented as in Fig. 22. It can be seen that the dislocations do originate from within the grain and not from the grain boundaries. The fact that the dislocations in a pileup were indeed moving towards the grain boundaries during the initial application of the stress is also shown by the motion of these dislocations in a direction away from the boundaries during stress relaxation (see Fig. 9) or stress reversal (see Fig. 14). Furthermore the detailed structure of the dislocation pileups and its variation with increasing applied stress are consistent only with a mechanism involving the motion and mulitplication of in-grain dislocation segments as will be discussed later.

E. Slip Propagation Across Grain-Boundaries

Although no systematic study of slip continuity as a function of the orientation relationships across a grain boundary has been made in the present experiments the few cases of slip continuity that were actually observed seem to indicate that in the microstrain region slip propagation involves the transfer of dislocations from one grain to the other. The one common feature of the cases of slip continuity so far encountered was that the lines of intersection of the active slip planes in the two grains with the grain boundary were nearly coincident. In the case of a coherent twin boundary this coincidence was exact and along a $\langle 110 \rangle$ direction in each grain. The transfer of dislocations from one grain to the other under these conditions might then easily occur by the mechanism already discussed. It is not quite clear how dislocations pass from one grain to another under less ideal conditions as in Fig. 19 where the line of intersection of the active slip planes with the grain boundary does not exactly lie along a $\langle 110 \rangle$ type direction. However the observation by Ogilvie[13] that even after 10% strain slip continuity in aluminum was observed predominantly only along those straight sections of the grain boundaries where the intersection of the active slip planes lies at an angle of less than two degrees from one of the ⟨110⟩, ⟨112⟩ or ⟨123⟩ directions in both the slip planes indicates again that some special orientations are particularly favorable for slip propagation, most probably because of the ability of dislocations to pass through such boundaries.

These observations might suggest an explanation for the gran-size dependence of the elastic limit of polycrystalline aggregates different from the many that have hitherto been proposed. In general specimens with different grain sizes for such experimental determination of elastic limits are obtained by different heat treatments. In particular, 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, tilt boundaries and others with low misorientations will be preferentially retained while the higher energy boundaries will be preferentially eliminated. Slip propogation across such low energy boundaries would be comparatively easier.* 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 such "weak" boundaries developed during the necessary heat treatment.

---

*Ogilvie indeed observed that when the duration of annealing, before the final deformation, was increased, there was, within a given surface area, an increase in the number of grain boundary sections where slip lines crossed.

F. Dislocation Multiplication Mechanism

The observation that the dislocations in a pileup often belong to discernibly distinct slip planes in the beginning stages of plastic deformation definitely rules out the operation of a classical Frank-Read multiplication mechanism[20] according to which all the freshly generated dislocations should lie on the same or nearly the same slip plane. It is unlikely that cross-slip of some of the piled up dislocations could have caused the generally observed deviation of a pileup from a linear array because for the normally operative burgers vector in the present experiments the cross slip plane was the one that is almost parallel to the surface of observation and consequently had a negligible shear stress acting on it. Also such dislocation configurations as in Figs. 8(a) to 8(d) are difficult to visualize as resulting from cross slip of the leading dislocations in a pileup.

It is thus to be assumed that the dislocations in many early pileups have originated from different sources. The fact that such dislocation configurations are usually observed even in the first few pileups indicate that some kind of a cooperative mechanism rather than a coincidental operation of a few sources on nearby glide planes is involved in their formation.

The basic reason for the non-operation of a conventional Frank-Read source mechanism is the inability of the opposite dislocation segments to annihilate when they arrive at the same point by two different paths. There are many reasons why these opposite segments of the same dislocation will generally be on different glide planes.[21] In a well annealed specimen as employed in the present experiments the most obviuos reason seems to be the jogged nature of the dislocation segments. As discussed earlier only those dislocations with both ends fixed within the crystal seemed to take part in the early dislocation multiplication under the experimental conditions employed in the present experiments. Though less jogged than dislocations already intersecting the surface even the first dislocation segment to expand beyond its critical radius for multiplication would not, in general, have its ends lying on the same glide plane. If the initial orientation of the dislocation is near screw this would lead to the formation of a dipole P when the two edge segments approach each other as shown in Fig. 23(a). The formation of a second dislocation loop from this source would require the dragging of the dipole to the point D before the source dislocation can expand to its critical radius as shown in Fig. 23(b). To attain the configuration shown in (b), however, the dislocation segments Ax and By in (a) have to bow through a critical radius equal to half their lengths and hence require a stress twice as high as that which started the operation of the original segment AB. This usually causes the source to cease operating after it has emitted only one dislocation loop.

In a real crystal, however, the bowing of a dislocation segment would be more complex.[22] If the bowing segment is initially longer than the average for the network it will usually come up against other dislocation segments that pierce its glide plane before it reaches equilibrium curvature. By forming new attractive junctions with some of these intersecting dislocations its length will be reduced to something close to the average length for the network. Continued expansion of such a segment can then occur only when its effective length is increased by glide of the connecting forest dislocations along their own glide surfaces as indicated in Fig. 24. This should frequently occur at a stress smaller than that necessary to complete the intersection. The longest bowing segments may grow slowly by this process to the length $\mu b/\tau$ where τ is the resolved shear stress on the primary system. In this way the primary dislocation can move forward successively pusing aside the most easily displaced intersecting dislocations. The moving dislocation loop would follow a tortuous path partially clearing away the forest of intersecting dislocations in the neighborhood of its glide plane as shown in Fig. 25(a). According to this process also progressive strain multiplication will become increasingly difficult because when the partially cleared trails that would be created turn back on themselves as in Fig. 25(b) a dipole will be formed since the sum of the burgers vectors of the intersecting dislocations will not generally be zero.*

It is proposed that the presence of dislocations belonging to distinctly different glide planes in the very early pileups is due to a cooperative dislocation multiplication mechanism in which an initially expanding loop is able to induce the operation of other shorter sources on nearby glide planes under the combined influence of the applied stress and its own stress field. Each of these new sources should normally send out only one loop for the same reason as discussed earlier; but they can in turn trigger the operation of additional dislocation segments lying within less than a critical distance of their path. Assuming that

*Special situations under which large amounts of slip can result from the multiplication of a single dislocation segment will be discussed later.

the length of the induced segment would be smaller than that of the initially operating segment a rough estimate can be made of this critical distance.

Let the average dislocation density ρ be $10^4/cm^2$. Then the critical stress for the multiplication of a segment of average length $\ell (= \rho^{-1/2})$ is given by

$$\tau_{Crit} = \frac{\mu b}{\ell}$$

where μ is the shear modulus and b is the magnitude of the burger's vector of the dislocation

Taking $\mu = 4.5 \times 10^6$ g/mm^2
$b = 2.56 \times 10^{-7}$ mm and
$\ell = (10^4)^{-1/2} = 0.1$ mm

τ_{Crit} turns out to be approximately 12g/mm^2. The critical distance h between two slip planes above which an edge dislocation on one plane can pass over another identical edge dislocation on the second plane is approximately given by

$$h = \frac{\mu b}{8\pi(1-\nu)\tau}$$

where

ν = Poisson ratio, and
τ = Resolved shear stress

Taking $\nu = 0.34$, the value of h at τ_{Crit} turns out to be approximately 6 microns. It was experimentally observed that 6 microns was about the maximum distance measured between the operative slip planes in a pileup in the beginning stages of dislocation multiplication.

Although the cooperative multiplication of dislocations discussed above can account for the consistent presence, in the eary pileups of dislocations belonging to fairly separate glide planes the presence of many dislocations on the same or nearly the same glide plane as seen in Figs. 5 and 6, for example, requires a mechanism which makes possible the repeated generation of dislocation loops from a single segment. The fact that such dislocation pileups were seldom seen in the interior grains suggests that the presence of a free surface might itself enable a dislocation segment to undertake repeated slip. A possible mechanism is indicated in Fig. 26. When a mobile dislocation segment intersects a free surface its expansion should lead successively to the configurations shown in the sequence (a) to (d). It can be seen that no dipole is formed in this case and that the recreated source dislocation in Fig. 26(d) can repeatedly follow an already cleared path. Unlike in the case of Fig. 23, a surface source is thus able to generate many dislocations once it starts operating. Such potential surface sources could either be dislocations already intersecting the surface or those double ended dislocations with one of their fixed ends close to the surface so that in the process of expansion they intersect the surface to provide a long mobile segment and a relatively immobile short segment. The fact that the many dislocations already intersecting the surface in specimens prepared by the second technique did not undergo such multiplication should only be due to their being heavily jogged as a result of the high temperature anneal. Most of the pileups that are observed at the surface should thus have resulted from multiplication of the comparatively less jogged double ended dislocations lying in regions close to the surface. The three dimensional configuration of the piled up dislocations suggested from repeated polishing and etching experiments and schematically represented in Fig. 22 is consistent with the multiplication mechanism depicted in Fig. 26.

It should be emphasized that this enhanced slip activity at the surface is not directly related to a possible factor of two reduction in the theoretical stress needed to expand a single ended surface dislocation beyond its critical radius for multiplication. The latter effect is only of secondary importance. A double ended dislocation segment twice as long as a surface intersecting one might not, in general, start to multiply at the same stress at which the latter does.

The fact that dislocation pileups like those on the surface grains were seldom observed in grains that were totally enclosed during deformation does not imply however that plastic deformation is not occurring in the latter at all. Even in the absence of a free surface situations can develop under which the cooperative glide mechanism of even a jogged dislocation can result in fairly large amounts of localized slip. As already discussed earlier when the partially cleared areas turn back on themselves as in Fig. 25(b) a dipole would generally be formed. However the cleared areas might occasionally reach the back side of the original starting point as in Fig. 25(c) when the expansion of one portion of the original dislocation is somehow strongly hindered, as for example, by a region of high dislocation density. Alternately a cleared area may turn back on itself as in Fig. 25(b) where a segment of the expanding loop has been annihilated by recombination with a forest dislocation of antiparrallel burgers vector.[23] In either case a slip band will have been nucleated within the grain because the dislocation loop can then repeatedly follow an already cleared path. The activation of such an internal source in the early stages of deformation is probably indicated from the sequence of micrographs in Fig. 27. Figure 27(a) shows an isolated group of dislocations piled up at a twin boundary in a specimen etched after a compressive stress of about 60g/mm^2. Figure 27(b) shows a similar group of dislocations in the interior of the grain and apparently along the same slip plane as those dislocations near the boundary in Fig. 27(a). There was no pronounced alignment of etch pits visible along the same slip plane trace in the region between these two groups. However, on removal of about 50 microns of material from the surface and reetching many more dislocations were revealed along this trace (Fig. 27(c)). A low magnification picture (Fig. 27(d)) clearly showed that the two isolated groups of dislocations in Figs. 27(a) and (b) were really part of the same slip band. These observations can be best explained on the basis that the dislocation loops originally had the configuration schematically represented in Fig. 28. As material was progressively removed from the surface more and more dislocation loops should have intersected the surface (as at points marked x in Fig. 28) causing an increase in the number of etch pits observed.

A yet another mechanism that can produce a limited number of dislocation loops from a jogged single dislocation segment is the unsymmetrical operation of a source as schematically indicated in Fig. 29. If the expansion of the dislocation AB is hindered at only some portions of the loop then its continued expansion will lead initially to a configuration shown in Fig. 29(a). Such local hindering could result from unequal densities of forest dislocations in the vicinity of the expanding loop. Continued expansion of the loop should result in a configuration as in (b) where an additional loop (2) is created which can follows an already cleared path. This loop will however get stuck behind the first one at the locally hard region near A. Figure (c) shows a later stage in the expansion process where a third dislocation loop is beginning to be formed and also the second loop is beginning to turn back on itself at C. Figure (d) shows a still later stage where the third loop is more fully formed. The operation of the source will eventually cease when the back stress due to the piled up dislocations near A reduces the stress at the source to a value below the critical multiplication stress.

The infrequent occurence of such examples as in Fig. 28 as well as the general inability to directly observe dislocation pileups in the interior grains imply, however, that the special situations discussed are either less frequent or less effective than the surface modified mechanism in nucleating a slip source or that these situations are more often met near the surface than in the interior of the grain. Another reason for the general absence of any sort of piled-up dislocation arrangements in the inside grains could be that, using the present experimental methods, their dislocation configuration could be observed only in the stress relaxed condition. The present experimental results have definitely indicated that dislocations undergo appreciable reverse motion on removal of the applied stress. Usually the ability to detect pileups on the surface even in the stress relexed condition is mostly due to Lomer-Cottrell locks (formed through traces of secondary slip) preventing the complete relaxation of the piled up dislocations. As may be seen from inspecting a Thompson tetrahedron Lomer -Cottrell locks can form over an appreciable length only when the interacting dislocations are in nearly edge orientations. When both the interacting burgers vectors are parallel to the surface, as they frequently happened in the present experiments, almost all dislocations that intersected the surface and that can potentially form Lomer Cottrell locks would have developed an orientation close to edge as may be seen from Fig. 22. Under these conditions the nearly parallel interacting dislocations can form long and stable Lomer-Cottrell locks. In a completely enclosed grain dislocation loops on intersecting planes can meet only over very short lengths and the resulting Lomer -Cottrell dislocations may be too weak to prevent the running back of piled up dislocations into their sources on stress relaxation.

It should be mentioned that whenever a slip source is nucleated by the cooperative mechanism each successive trip of the dislocation loop should generally be on a glide layer displaced above or below that of the immediately preceeding passage. In the case of edge dislocations, they should form a short dislocation wall rather than a planar pileup. The apparently planar nature of the pilups that are observed at the surface might indicate that the few jogs remaning on the source segment after its first circuit (see Fig. 26) are able to glide off to the surface very early during its subsequent expansion thereby confining the slip activity to almost a single glide plane.

It is probable that a group of primary dislocations represents a sufficient stress concentration so that multiplication of dislocations of the other two co-planar burgers vectors might always be associated with its motion. The two different kinds of pits seen in the pileups A and B of Fig. 30(a) might indicate dislocations of different burgers vectors generated by such a process. Fig. 30(a) shows a grain etched in the stress-relaxed condition after a compressive stress of $50g/mm^2$. Dark and light pits can be seen in pileup A and pits of two different sizes are evident in pileup B. These differences in the nature of the pits were apparently not due to differences in the sign of the dislocations because they were found to move in the same reverse direction ie away from the grain boundaries, when the specimen was subsequently compressed in a direction at right angles to the original direction and reetched. The double etched grain is shown in Fig. 30(b). The second compression should have reversed the direction of the shear stress as schematically indicated in Fig. 31.

G. Multiple Slip and Dislocation Interactions

In spite of the relatively low stresses and large grain sizes employed in the present experiments slip was seldom confined to only one system. Large amounts of secondary slip on planes other than the primary plane, whenever present, could be detected by secondary pileups at the grain boundaries or other internal barriers. Typical examples of pileups formed under such multiple slip conditions can be seen in many previous Figs. such as 8b, 13 and 18. The presence of small traces of secondary slip can, however, be only indirectly detected from the appearance of primary dislocation pileups in regions away from the grain boundaries. Fig. 32 shows a typical example of such an internal pileup at B in a specimen etched under a stress of approximately $40g/mm^2$. The barriers responsible for the formation of such pileups are most likely to be Lomer-Cottrell dislocation locks formed by the interaction of a primary and a secondary dislocation for the following reasons. The type of barrier had to be one that existed along a line rather than at a point because these pileups could be detected at the same place even after removal of more than 200 microns of material from the surface. Also it is inconceivable for any point type obstacle (including forest intersection) to be able to support the magnified stress due to a pile-up of about 10-20 dislocations. Furthermore in the usual modes of testing crystals such as tension, compression or bending the system most highly stressed next to the primary system is alwasy the conjugate system and these two systems are the ones that can react to form Lomer-Cottrell dislocations.

In the case of internal dislocation pileups such as at B in Fig. 32 there is always the question whether the secondary dislocation responsible for the formation of the barrier was a stationary segment of the original network on one that had been moving. However there were many evidences to indicate that such interactions predominantly occur among moving dislocations. The fact that dislocations in the pileup A and B of Fig. 32 apparently lie along the same glide plane and are held up while moving in the same direction implies that the basic mechanism leading to the formation of these pileups is that indicated in Fig. 33. A dislocation source S_1 starts to send out dislocation loops as in (a) at some stress level during the application of the load. The first few loops travel the entire cross section of the grain without encountering any barriers. However, one of the subsequent dislocations might form a sessile lock by interacting with another dislocation moving on an intersecting slip plane due to unavoidable traces of secondary slip activity. This would lead to the formation of a grain boundary pileup A and an internal pileup B as shown in (b)*. Progressive formation of many such internal pileups

* Figures 33(c) and (d) represent possible mechanism for the formation of dislocation pileups like A in Fig. 9.

provides a natural explanation for the frequently observed alignment of dislocations over long distances along a few slip plane traces in the early deformation stages.

The fact that dislocation interactions as exemplified in Fig. 13 are predominantly observed only after stress relaxation also indicates that during the application of the load different sources should have operated at different times. Only during stress relaxation will there be a simultaneous movement of dislocations on all slip planes and hence the opportunity for them to meet and interact. Such interactions provide a natural expalnation for the ability to detect pileups like in Figs. 14(a) (stress reversal experiments) and 21 even in the stress relaxed condition. In particular the source dislocation S itself may be immobilized at the position shown in Fig. 22 by interaction with a moving secondary dislocation. The pileups A and B cannot relax completely because of the presence of the immobilized source segment S. Even when the direction of the applied stress is reversed the source cannot operate in the reverse direction. Under the situation depicted in Fig. 22, however, dislocations in the pileup A have some room to undertake some motion as the direction of the stress is changed. The experimentally observed behavior of dislocation pileups upon stress reversal (as explained in Section E of Results) fits into such a picture. Furthermore, the reduction in the number of dislocations in the pileup A in Fig. 14(f), from that in Fig. 14(a) is a direct consequence of the loss of some semicircular loops that should have occurred during the necessary removal of about 100 microns of material between the two stages.

The frequent observation of dislocation pileups in the present experiments is contrary to the general idea that pileups occur only very rarely in pure face centered cubic metals. It appears that in most of the previous experiments using single crystals where the observations were usually made in the stress relaxed condition dislocation pileups would have been hard to detect in the early deformation stages because many dislocations could have either completely escaped through the surface or undergone apprecialbe stress relaxation. The fact that isolated dislocation pileups at the grain boundaries were frequently the very first indications of the occurence of plastic deformation in polycrystals imply that the dislocation loops emanating from the first few sources to become active are indeed able to glide over substantially long distances. A similar conclusion was also reached by Tinder and Washburn[1] from their high sensitivity strain measurements on tubular polycyrstalline OFHC copper specimens.

The present experiments also showed, contrary to usual reports, that dislocations of even secondary systems can sometimes travel very long distances in the early stages of plastic deformation. Fig. 34 shows a grain etched after application of a compressive stress of about $60 g/mm^2$. The primary and secondary slip directions are indicated by the letters 'p' and 's' respectively. The group of dislocations marked x represent the first few dislocations sent out by a secondary source. These dislocations were able to travel all the way to the grain boundaries for the same reason as for the first few primary dislocations viz that they did not encounter and interact with any other (primary) dislocation. However, the generally higher dislocation activity on a very large number of primary planes might provide enough primary dislocations to interact with most of the subsequent secondary dislocations. This results in the formation of a large number of dislocation locks along the trace of the original secondary slip plane. Subsequent blockage of primary dislocations at this extended array of dislocation locks leads to the "decoration" of these secondary slip plane traces as seen at a few places in Fig. 34. As the total strain on the primary system increases more and more dislocations get caught along these traces leading to the development of heavy dislocation tangles. Figure 35 shows a particularly good example of such dense bands of dislocations along a few secondary slip plane traces in a specimen etched after a compressive stress of about 200g/mm^2. The implication of the above discussed mechanism is that the reportedly low distances of motion of secondary dislocations in the early deformation stages is only due to their immobilization by interaction with a steady stream of primary dislocations on a vast number of primary planes.

The reason for the operation of secondary sources even in the pre-yield region is not quite clear. The necessary scatter in the network size might provide a few long enough secondary dislocation segments particularly because the symmetry of the f.c.c. structure provides a large resolved shear stress on many of the secondary systems also. The stress field due to a group of primary dislocations can somethimes aid the applied stress in operating nearby sources on a secondary slip plane.[24] The glide of some connecting forest dislocation segments caused by the bowing of a highly mobile segment on the primary glide system as discussed in connection with the cooperative glide mechanism may be yet another way of inducing secondary slip. In any case the incidence of such slip activity should depend on the resolved shear stress acting on the secondary planes and hence on the orientation of the applied stress.

H. Macroscopic Yielding

It is interesting to consider what information, if any, the present experimental results provide regarding the general concept of macroscopic yield stress. As already mentioned in the introduction and indicated from the present results substantial dislocation motion and multiplication do take place at stresses considerably below the yield stress. Macroscopic yielding characterized by the appearance of slip markings at the surface and by a sharp change in the slope of the load-elongation curve (in single crystal) thus takes place only after pre-yield plastic strain has produced a population of dislocations that is very different from the network that existed after recrystallization or after growth from the melt. One of the most characterisitc observations in the present experiments was the consistent occurence of traces of secondary slip which caused the primary dislocations from a source to pileup into discrete groups at various points along their glide plane conceivably through the formation of Lomer Cottrell locks. Similar dislocation arrangements should also occur in single crystals in the pre-yield region. It is proposed that macroscopic yielding takes place when the magnified stress due to the dislocations at such internal pileups becomes large enough to break down the Lomer Cottrell barriers, thereby permitting a very large number of dislocations to glide long distances and eventually escape through the surfaces. This would explain the sudden appearance of prominent slip markings on the surfaces at yielding as well as the usual absence of any appreciable residual dislocation damage in the slipped regions of metal crystals. If a definite critical stress is needed to break down the barriers then the experimentally observed increase in the yield stress of copper single crystals for orientations favoring multiple slip (S. Kitajima, private communication) can be qualitatively explained as follows. The incidence of secondary slip and hence the number of Lomer Cottrell locks formed within a certain length of an active primary slip plane would, in general, be greater in the case of those orientations that result in a higher resolved shear stress on the secondary systems. Figure 36 shows the very large number of such locks formed in a grain with an almost duplex slip orientation in a specimen etched under a stress of approximately $55 g/mm^2$*. The increase in the number of such locks, however results in a decrease in the number of dislocations piled up at each of these locks. The applied stress has consequently to be increased before the piled up dislocations can raise the local stress to the critical amount necessary to break the barriers and cause yielding. Even though the above mechanism can explain the yielding of individual grains the mechanism for a cooperative yielding of many grains necessary for any appreciable over-all plastic flow in a polycrystal is not clear from the present results.

---

* The arrow-headed appearance of dislocation groups as at A in Fig. 36 with the dislocation pileups almost always pointing in the same direction is a necessary consequence of the mechanism of formation of such pileups discussed in connection with Figs. 32 and 33.

V. CONCLUSIONS

1. The present results are consistent with previously reported results regarding the influence of the degree of jogging of dislocation segments on their mobility. Heavily jogged dislocation segments do not seem to take part in the early multiplication process.

2. A clean surface by itself is not a source of dislocations in the early deformation stages of copper. Most of the previously reported surface sources in copper appear to be only accidentally introduced dislocations resulting from surface contact.

3. Grain boundaries also do not act as sources of dislocations in the pre-yield region of copper. Their main role is to act as barriers to moving dislocations.

4. In a well annealed crystal dislocation segments that can potentially act as Frank Read sources will always be jogged. The generation of many dislocations from such a segment is only possible when the source can operate in an unsymmetric way. Frequently such multiplication is accompanied by the cooperative motion of dislocation segments lying on nearby parallel glide planes.

5. The presence of a free surface can cause an increase in the extent of strain at the surface when a mobile dislocation segment intersects the surface. This effect is basically due to a modification of the strain multiplication mechanism cuased by the surface but has nothing to do with a possible factor of two reduction in the theoretical stress needed to expand a single ended surface dislocation beyond its critical radius for multiplication.

6. The first few dislocation loops generated by the early operating sources are able to travel the entire cross section of the grain until stopped by the boundaries. This result suggests that in single crystals some of the first sources to operate may not be detected at all by etch pit observations because the dislocations may have moved entirely out of the crystal.

7. Traces of secondary slip always occur even in the pre-yield region and are responsible for the development of internal barriers (presumably Lomer Cottrell locks), to the motion of primary dislocations.

8. The phenomenon of macroscopic yielding in single crystals and its variation with the orientation of the applied stress can be qualitatively explained in terms of the collapse of internal Lomer Cottrell locks at a critical stress.

9. Grain boundary propagation of slip does not usually take place in copper in the microstrain region except in some special cases such as twin boundaries where dislocations from one grain can actually pass through into the next grain.

ACKNOWLEDGEMENTS

The author wishes to express his gratitude to Professor Jack Washburn for his encouragement, advice and continued interest in the present investigation. This work was carried out under the auspices of the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Radiation Laboratory, University of California, Berkeley, California.

REFERENCES

1. R. F. Tinder and J. Washburn, Acta Met. 12, 129 (1964).

2. F. W. Young, Jr., J. Appl. Phys. 32 1815 (1961).

3. F. W. Young, Jr., J. Appl. Phys. 33 963 (1962).

4. F. W. Young, Jr., J. Appl. Phys. 33 3553 (1962).

5. P. Petroff and J. Washburn, J. Appl. Phys. 37 4987 (1966)

6. K. Marukawa, J. Phys. Soc. Japan, 22 499 (1967).

7. F. W. Young, Jr. F. A. Sherrill and M. C. Wittels, J. Appl. Phys. 36 2225 (1965).

8. F. W. Young, Jr. and F. A. Sherrill, Can J. Phys. 45 757 (1967).

9. F. W. Young, Jr. To be Published.

10. J. D. Livingston, J. Appl. Phys. 31 1071 (1960).

11. F. W. Young, Jr, and T. R. Wilson, Rev. Sci. Inst. 32 559 (1961).

12. K. T. Aust, The Art and Science of Growing Crystals, (John Wiley an Sons, 1965) p. 494.

13. G. J. Ogilive, J. Inst. Met. 81 491 (1952).

14. C. S. Barrett and T. B. Massalski, Structure of Metals (McGraw-Hill Series, 1966), p. 569.

15. F. R. N. Nabarro, Z. S. Basinski and D. B. Holt, Adv. Physics 13 , 193 (1964).

16. J. C. Fisher, Trans. AIME, 194 531 (1952).

17. W. G. Johnston and J. J. Gilman, J. Appli. Phys. 30, 129 (1959).

18. F. J. Worzala and W. H. Robinson, Phil Mag. 15 939 (1967).

19. J. D. Livingston, Direct Observation of Imperfections in Crystals (Interscience Publishers, New York 1962). p. 115.

20. F. C. Frank and W. T. Read, Phys. Rev. 79 722 (1950).

21. J. Washburn "Dislocation Multiplication" UCRL Report 10200, June 1962.

22. J. Washburn and G. Murty, Can J. Phys. 45 523, (1967).

23. J. Washburn and G. Murty, J. Appl. Phys. 37 2511 (1966).

24. D. H. Avery and W. A. Backofen, Trans. AIME, 227 835 (1963).

FIGURE CAPTIONS

Fig. 1 Schematic drawing of the microcompression device.

Fig. 2 Dimensions and orientation of specimens used in the bending experiments.

Fig. 3 Modified polishing apparatus for removal of large amounts of material from the surface without mechanical damage.

Fig. 4 A double etched grain in a specimen subjected to a compressive stress of approximately 25 g/mm^2. No dislocation motion is evident.

Fig. 5 A double etched grain in a specimen subjected to a compressive stress of approximately 40 g/mm^2. The black circles indicate some typical instances of dislocation motion.

Fig. 6 Typical dislocation pileups in specimens prepared by the first technique and subjected to a compressive stress of approximately: (a) 40 g/mm^2, (b) 50 g/mm^2, (c) 50 g/mm^2, (d) 55 g/mm^2.

Fig. 7 Double etched grain in a specimen subjected to a compressive stress of about 50 g/mm^2. Note the absence of any large flat bottomed pits along the slip plane traces of pileups A and B.

Fig. 8 Examples showing the deviation of the dislocations in a pileup from a strictly linear array as seen by etching specimens under a stress of approximately: (a) 40 g/mm^2, (b) 60 g/mm^2, (c) 50 g/mm^2, (d) 50 g/mm^2. Examples (b), (c), and (d) are double etched.

Fig. 9 Sequence showing stress relaxation of a pileup A in a specimen subjected to a compressive stress of approximately 50 g/mm^2 and etched in: (a) the stressed condition, (b) the stress-relaxed condition.

Fig. 10 Two separate grains in a specimen showing the configuration (A) formed from dislocation pileups on close planes on stress relaxation after a compressive stress of 55 g/mm^2.

Fig. 11 Sequence showing the development of "glide polygonization" near a grain boundary on stress relaxation: (a) specimen etched under a compressive stress of approximately 52 g/mm^2, (b) etched after stress-relaxation and removal of about 10 microns of material from the surface.

Fig. 12 Preferential "glide polygonization" at a grain boundary nearly at right angles to the active slip plane trace in a specimen etched in the stress-relaxed condition after a compressive stress of about 55 g/mm^2. Note also the one to one correspondence of the pileups across the opposite boundaries.

Fig. 13 Interaction of relaxing dislocation loops after a compressive stress of approximately 55 g/mm^2. Note the dislocation arrangement at A.

Fig. 14 A sequence showing the behavior of dislocation pileups on stress reversal. (a) Etched in the stress relaxed condition after an applied 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 applying a stress of 29 g/mm^2 in the same direction as in (b), (e) Etched after applying a stress of 40 g/mm^2 in the same direction as in (b), (f) Etched after applying a stress of 40 g/mm^2 in the original direction as in (a).

Fig. 15 Schematic illustration showing the relation between the surface of observation and the twin boundary.

Fig. 16 Schematic illustration of the change in the orientation of the etch pits on a nearly {111} surface of observation as a function of its position relative to a {111} coherent twin boundary: (a) above the twin boundary, (b) intersecting the twin boundary, (c) below the twin boundary.

Fig. 17 Relative orientations of the {111} planes in the two regions of a twinned crystal as viewed perpendicular to the twinning plane.

Fig. 18 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 from the surface, and (c) after removal of an additional 50 microns of material from the surface. See text for details.

Fig. 19 Matching slip bands across a low angle boundary in a specimen etched under a compressive stress of about 60 g/mm^2.

Fig. 20 Photomicrograph showing the surface of a specimen subjected to a compressive stress of about 40 g/mm^2 while immersed in the electropolishing solution and etched (a) before drying, (b) after drying. No dislocation multiplication is evident in (b) as a result of the drying operation.

Fig. 21 (a) Dislocation pileups of opposite sign held up at opposite boundaries in a specimen subjected to a compressive stress of about 50 g/mm^2. (b) and (c) Same region as in Fig. 30(a) after successive removals of about 50 microns of material each time and reetching.

Fig. 22 Schematic representation of dislocation loops generated by a surface source.

Fig. 23 Successive stages in the operation of a Frank Read source with its ends not lying in the same glide plane.

Fig. 24. Increase in the distance between pinning points by glide of forest dislocations.

Fig. 25 Formation of a source by cooperative glide of forest dislocations.

Fig. 26 Operation of a surface source by the cooperative glide mechanism.

Fig. 27 Sequence showing the operation of an internal source in a specimen subjected to a stress of approximately 60 g/mm^2. (a) and (b) Dislocation pileups at a twin boundary and at an internal barrier respectively and apparently lying along the same glide plane. (c) and (d) Same area as in (a) at two different magnifications after removal of about

50 microns of material and re-etching. Note the increase in the etch pit density along the glide plane trace between the two original dislocation groups.

Fig. 28 Schematic representation of dislocation loops generated by an inner source.

Fig. 29 Sequence showing the unsymmetric operation of a jogged double ended source to create a limited number of dislocation loops.

Fig. 30 (a) Pileups (A and B) containing more than one kind of dislocations in a specimen subjected to a compressive stress of approximately 50 g/mm^2 and etched in the stress-relaxed condition. (b) Same region as in (a) after double etching under a stress of 15 g/mm^2 in a direction at right angles to the original one. Note the motion of all the piled up dislocations in the direction away from the boundary.

Fig. 31 Reversal of the direction of shear stress on changing the direction of compression through 90 degrees.

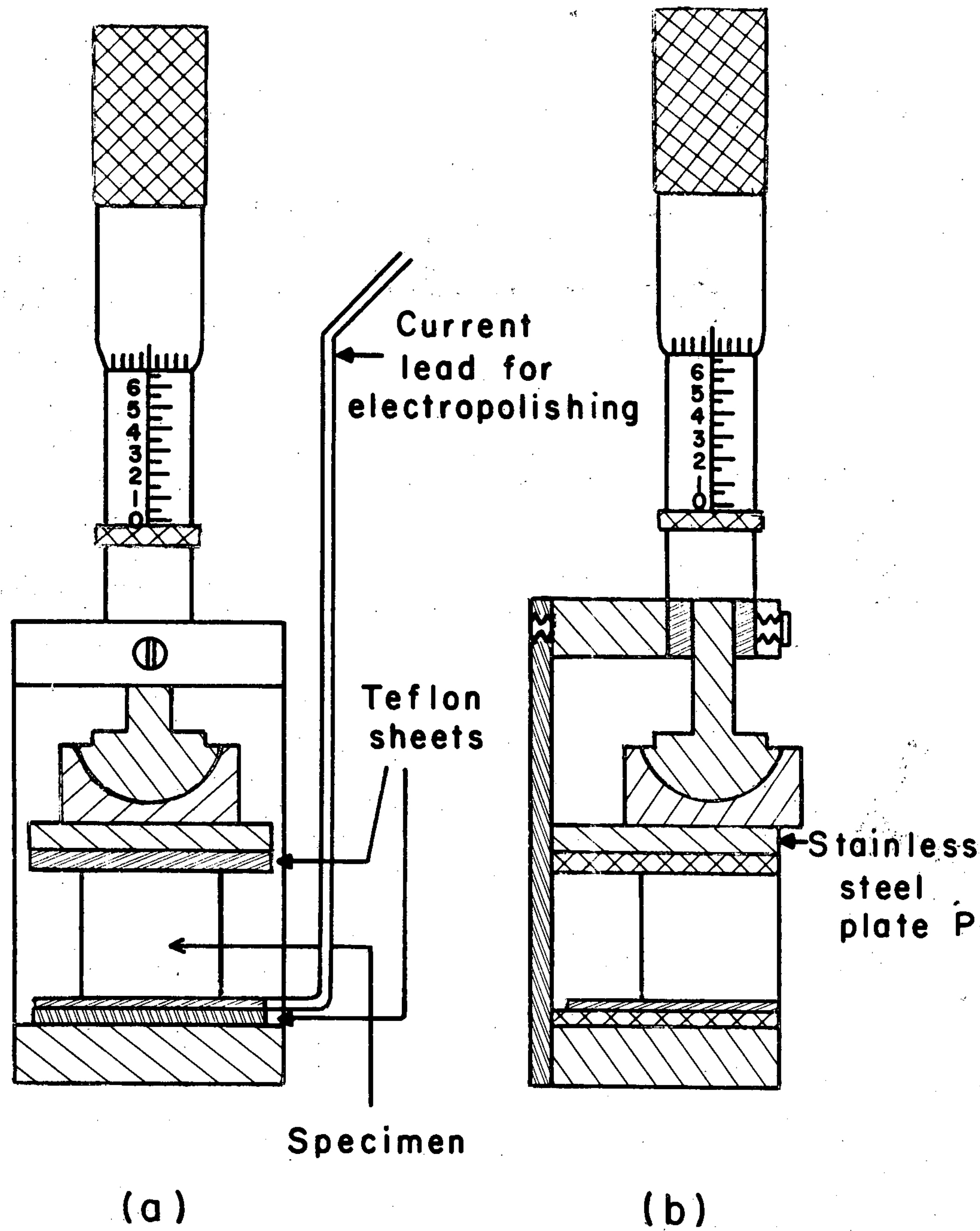
Fig. 32 Piling up of dislocations against an internal barrier at B in a specimen etched under a stress of approximately 40 g/mm^2.

Fig. 33 (a) and (b). Mechanism of formation of internal dislocation barriers along primary glide planes. (c) and (d) indicate a possible mechanism for the formation of dislocation loops like A in Fig. 9.

Fig. 34 Decoration of secondary slip traces in a specimen etched after a compressive stress of approximately 60 g/mm^2.

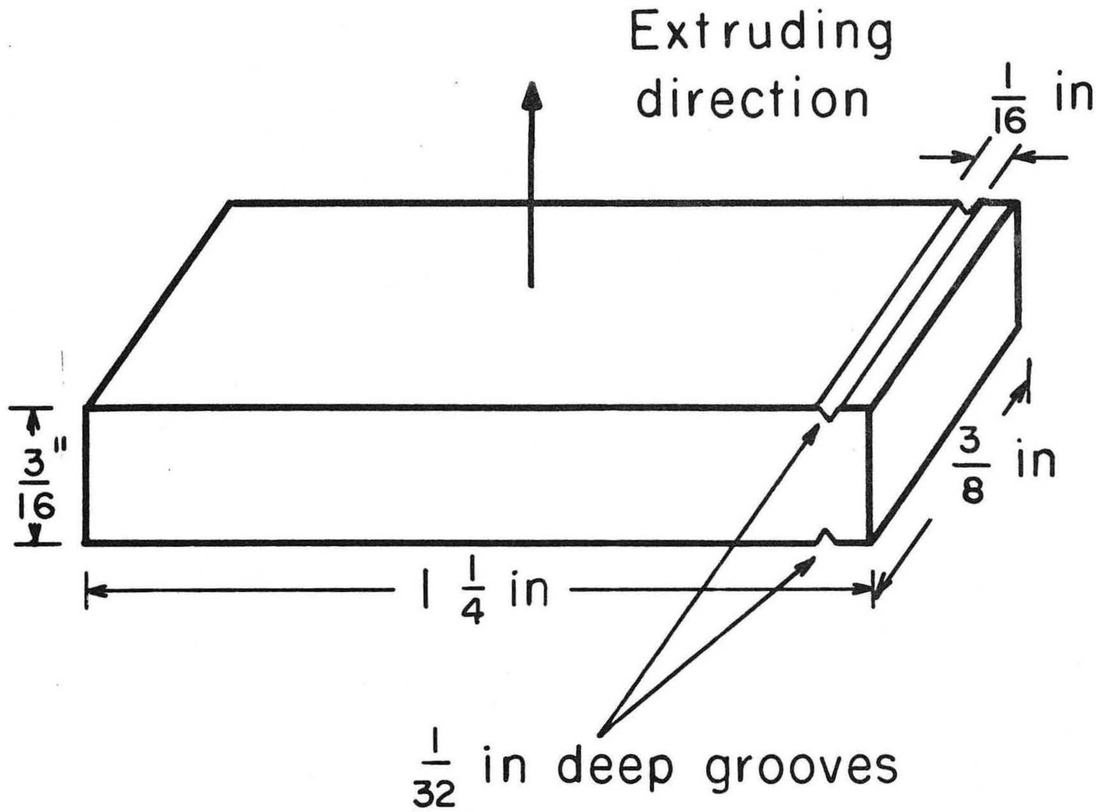
Fig. 35 Dense dislocation bands along traces of a secondary slip system in a specimen after a compressive stress of about 200 g/mm^2.

Fig. 36 Dislocation pileups at internal barriers in a nearly duplex slip oriented grain under a stress of approximately 55 g/mm^2.



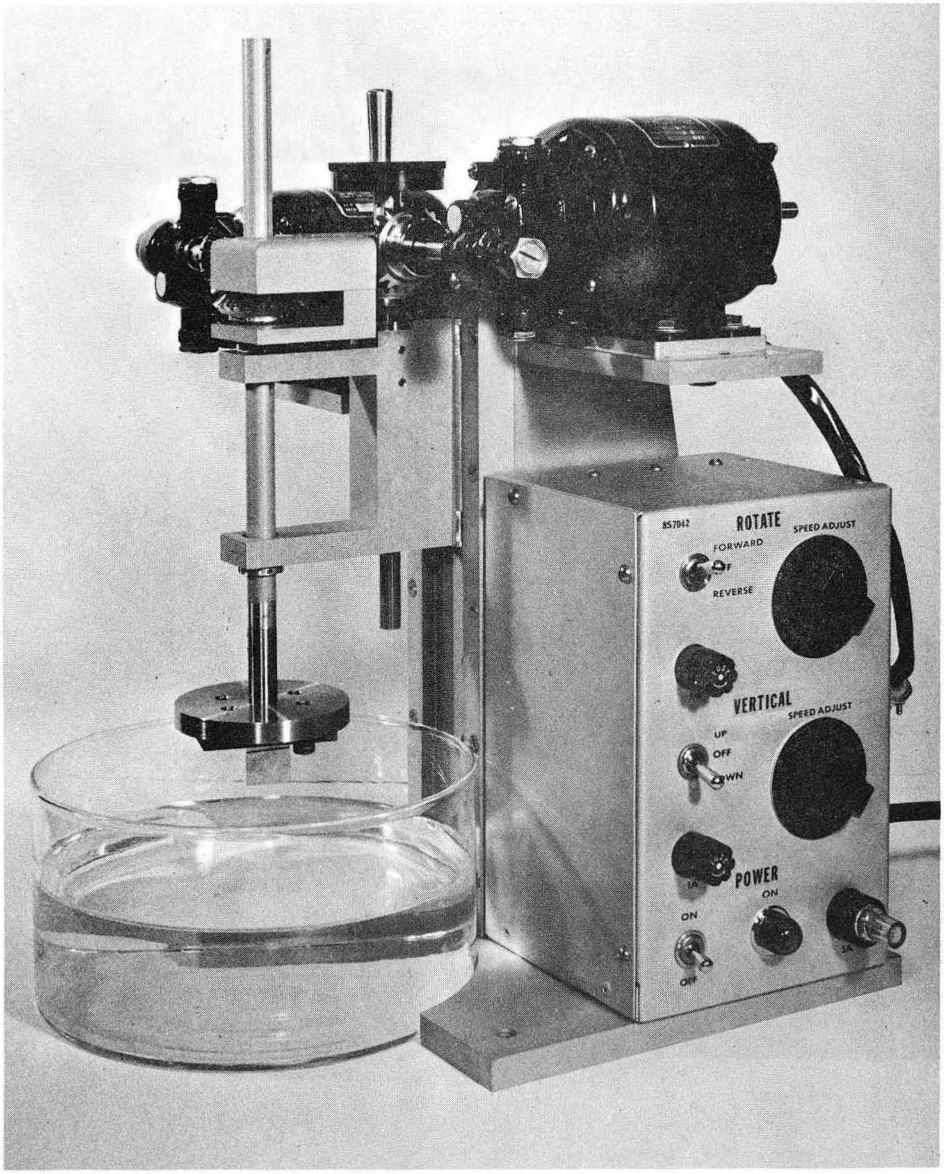
XBL687-3321

Figure 1



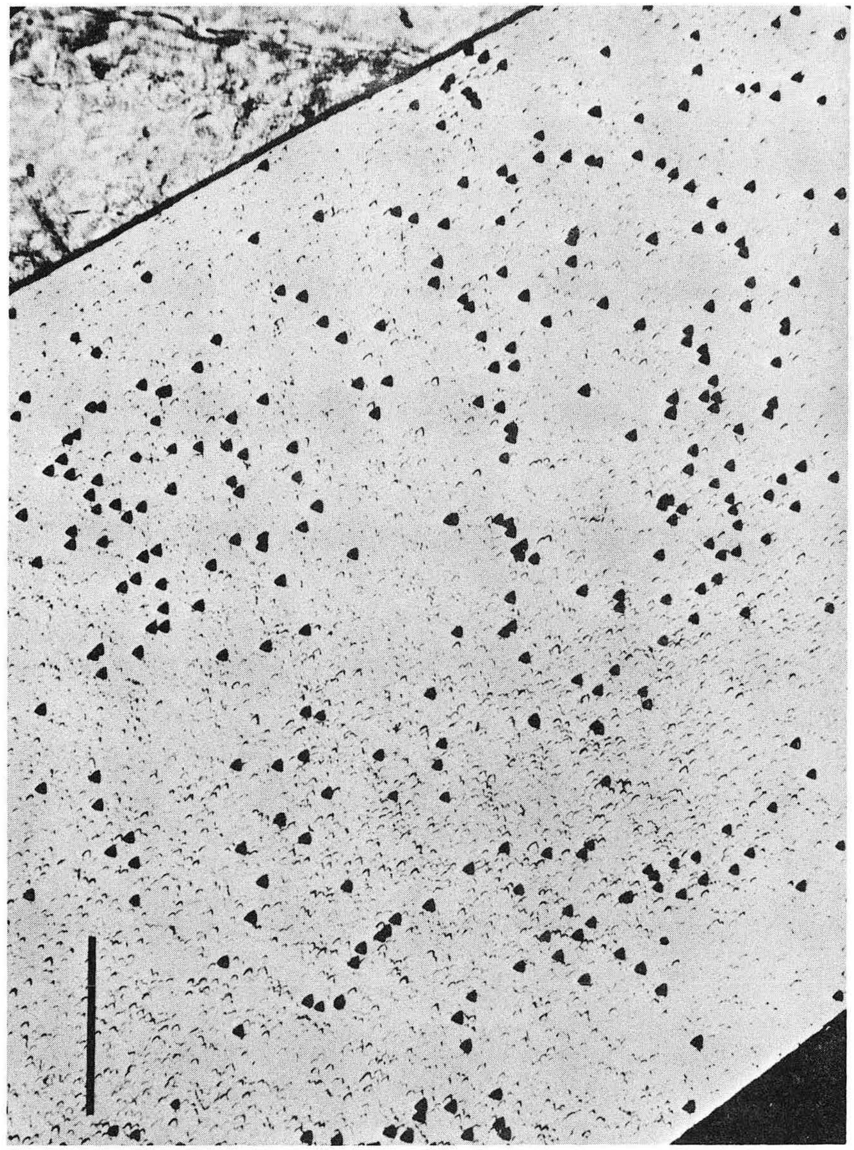
XBL687- 3327

Figure 2



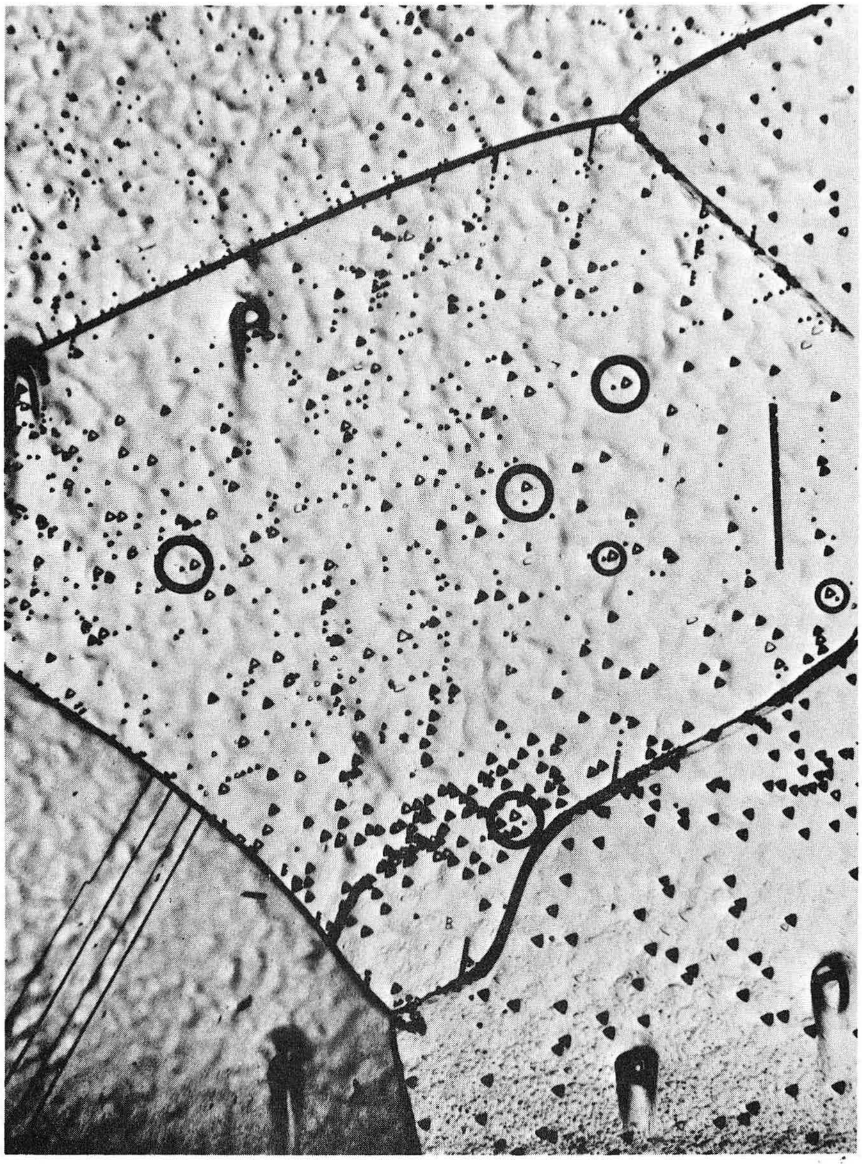
XBB 684-1669

Figure 3



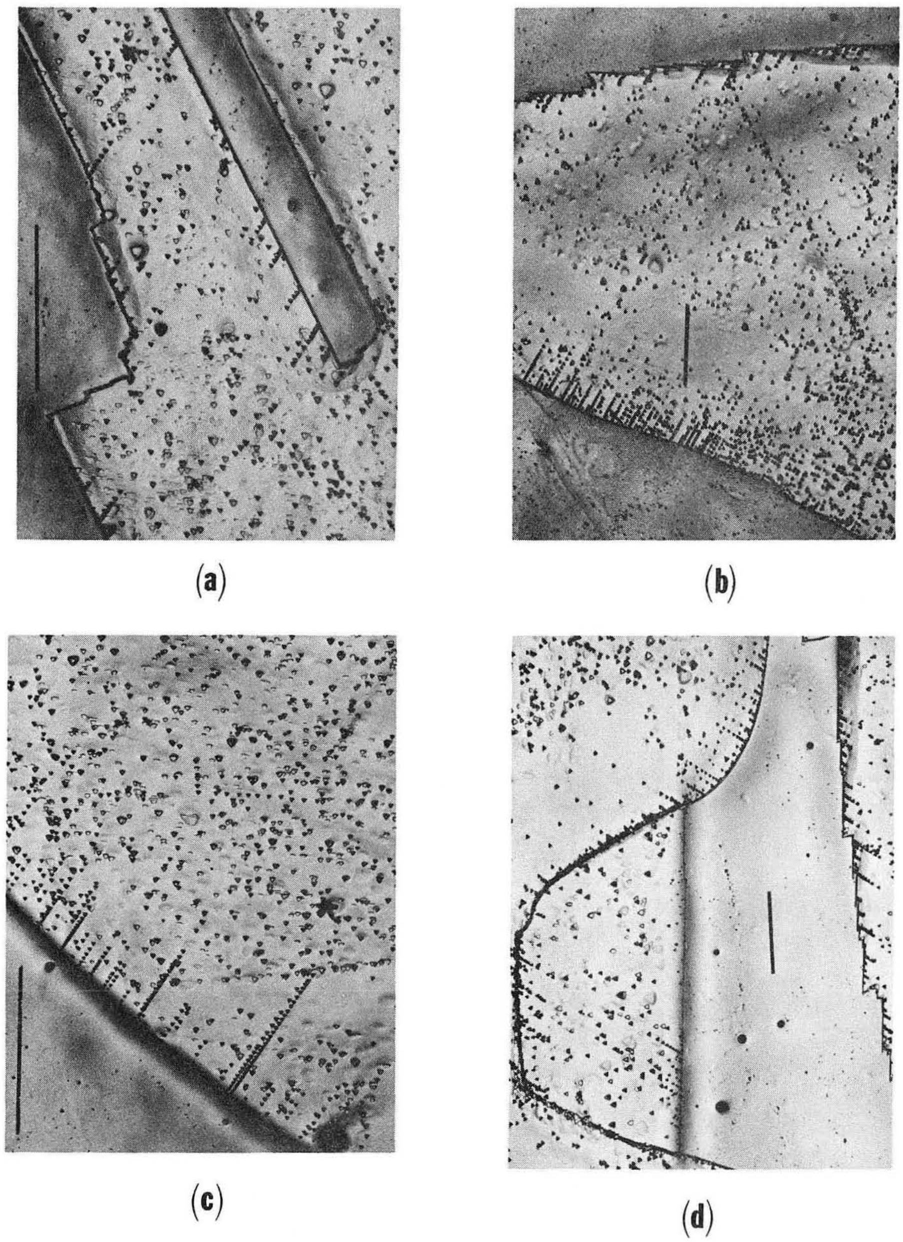
XBB 688-4979

Figure 4



XBB 688-4980

Figure 5

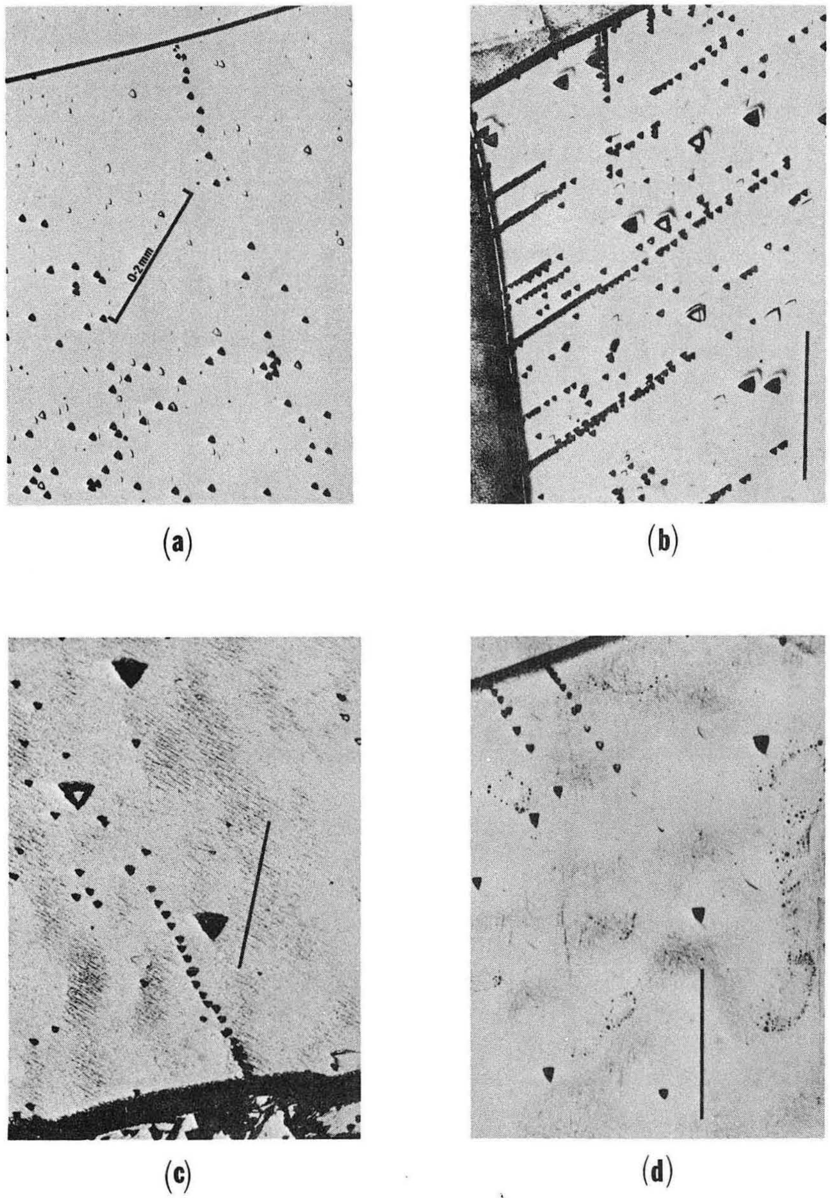


XBB 688-4991

Figure 6

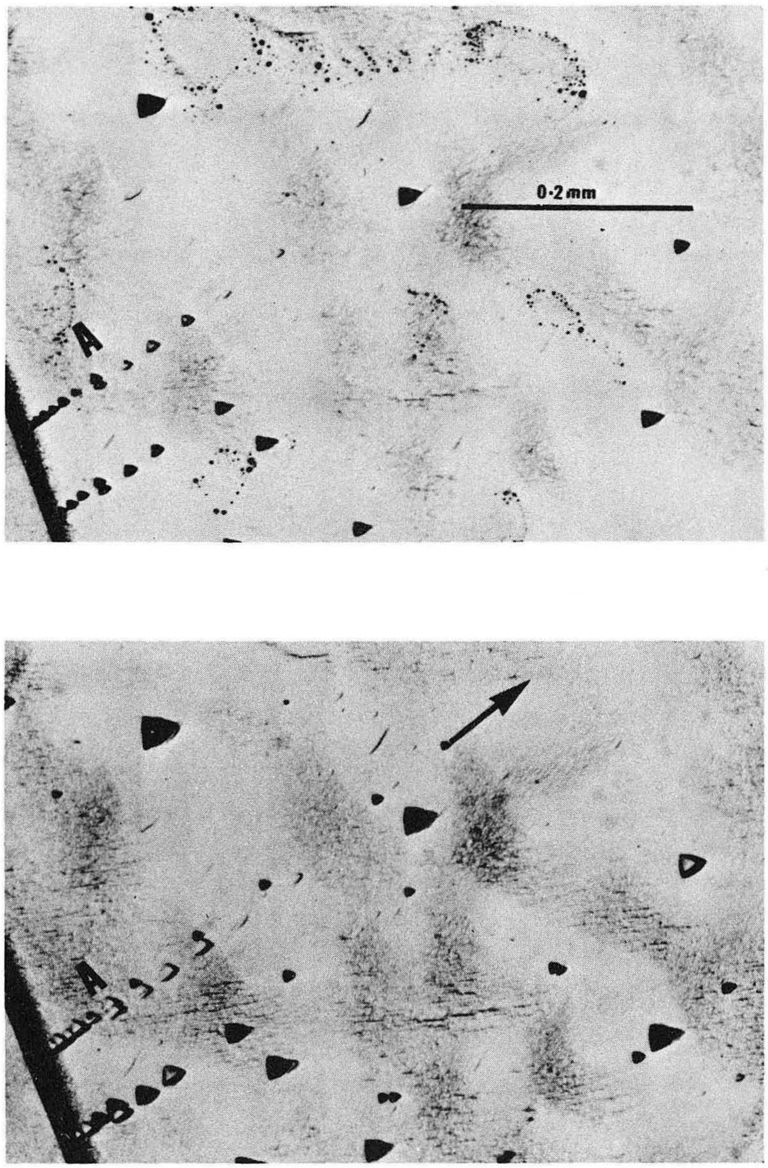
XBB 688-4977

Figure 7



XBB 688-4994

Figure 8



(a)

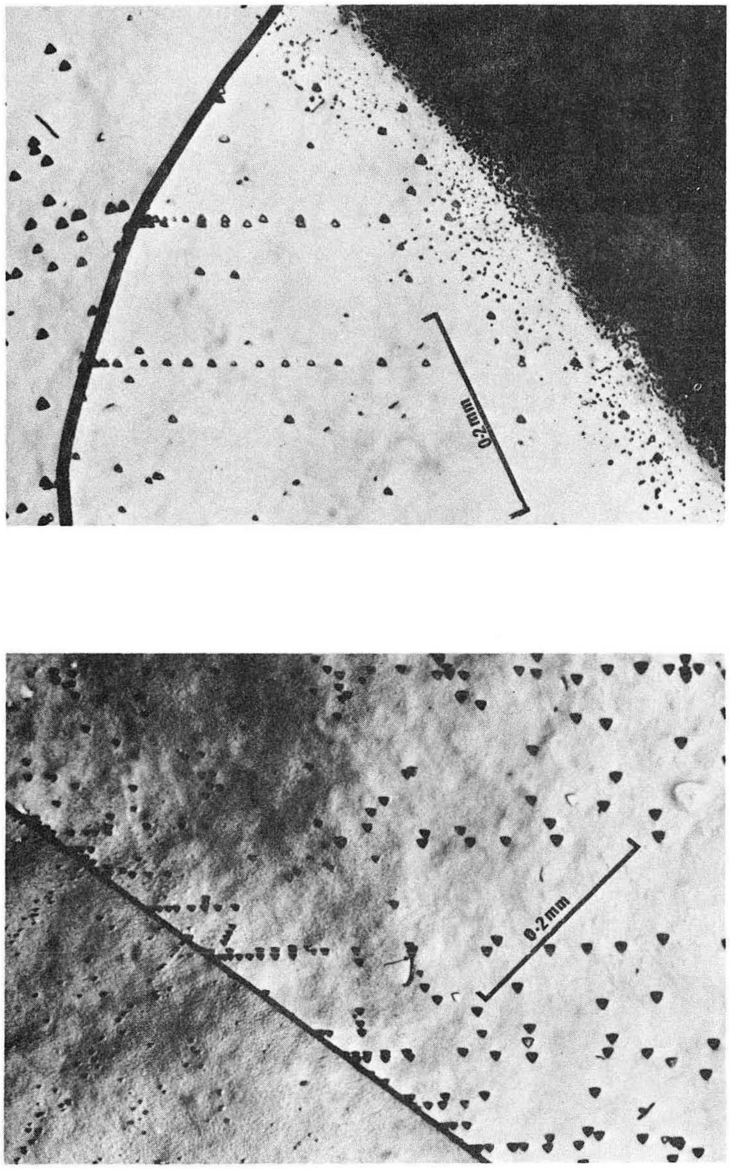
(b)

XBB 688-4985

Figure 9

(a)

(b)

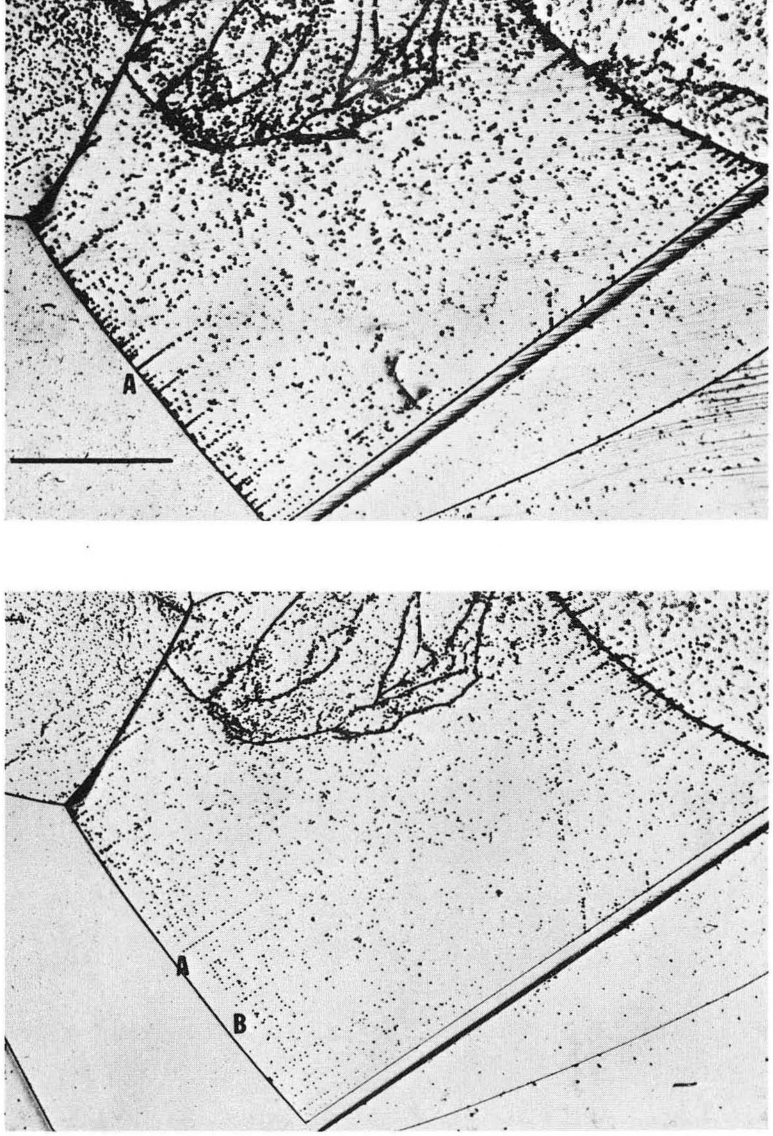


XBB 688-4986

Figure 10

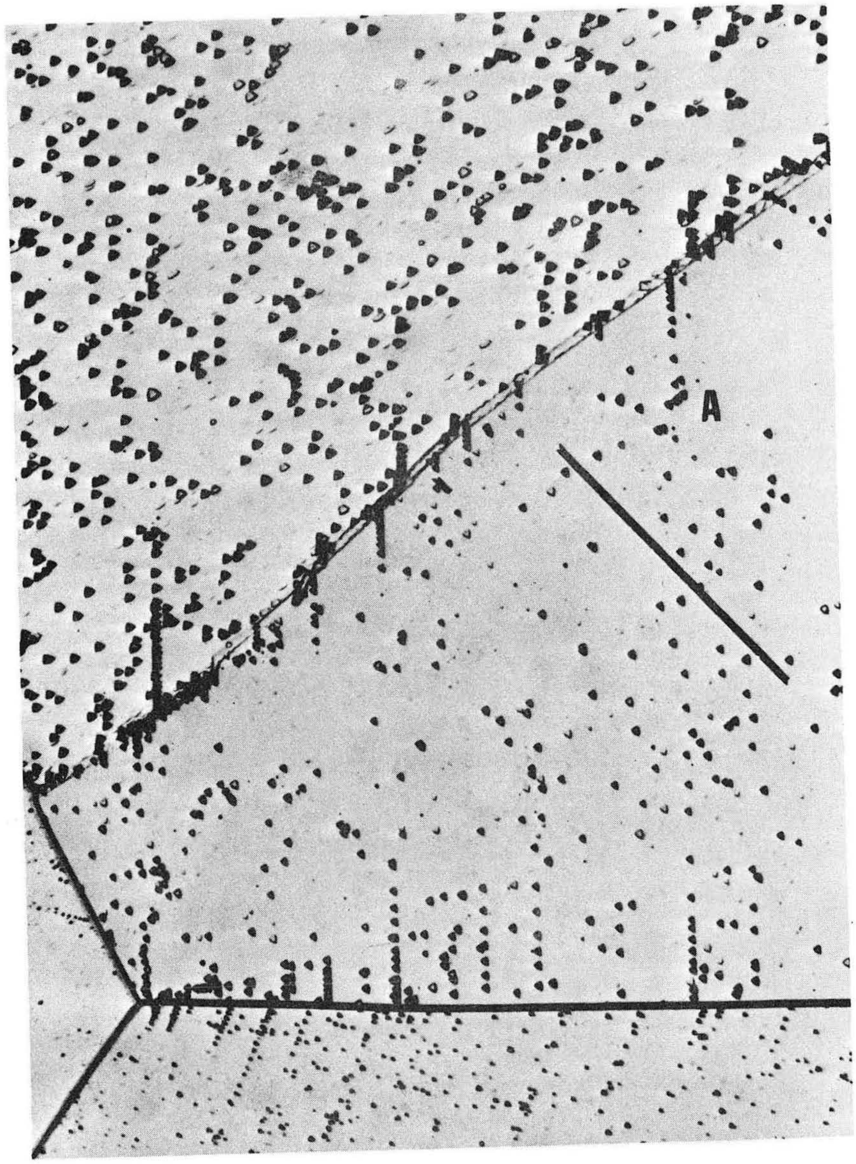
(a)

(b)



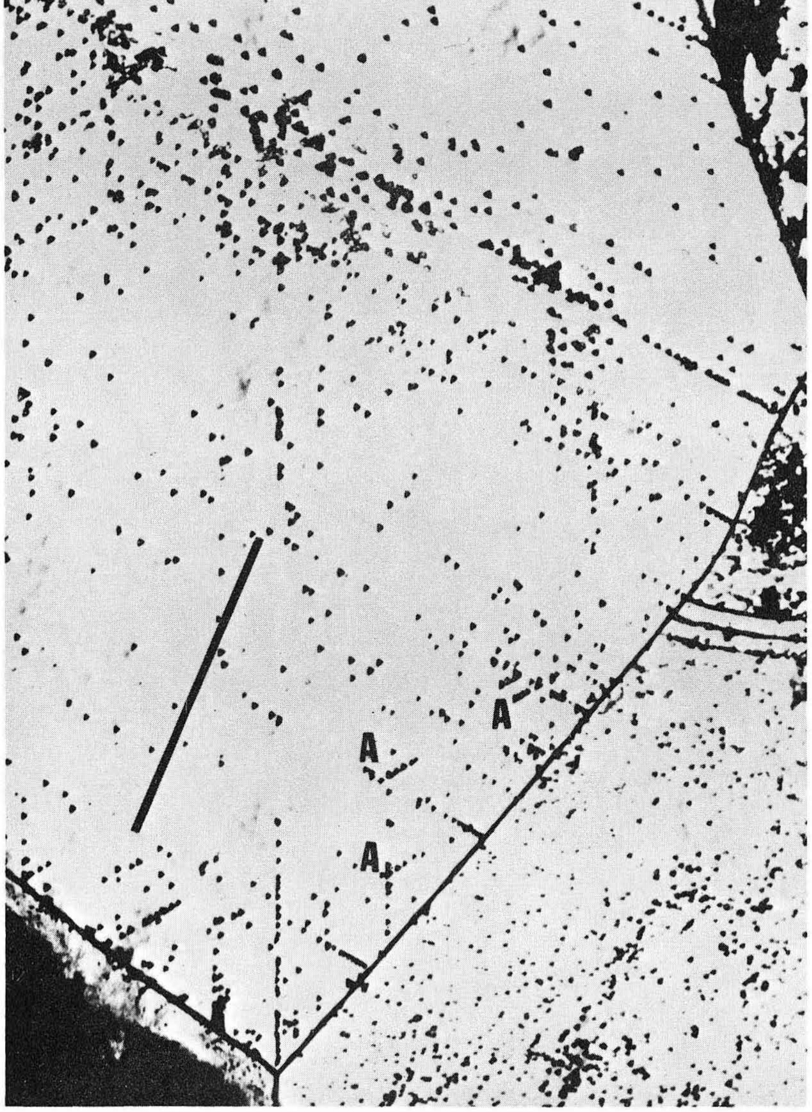
XBB 688-4984

Figure 11



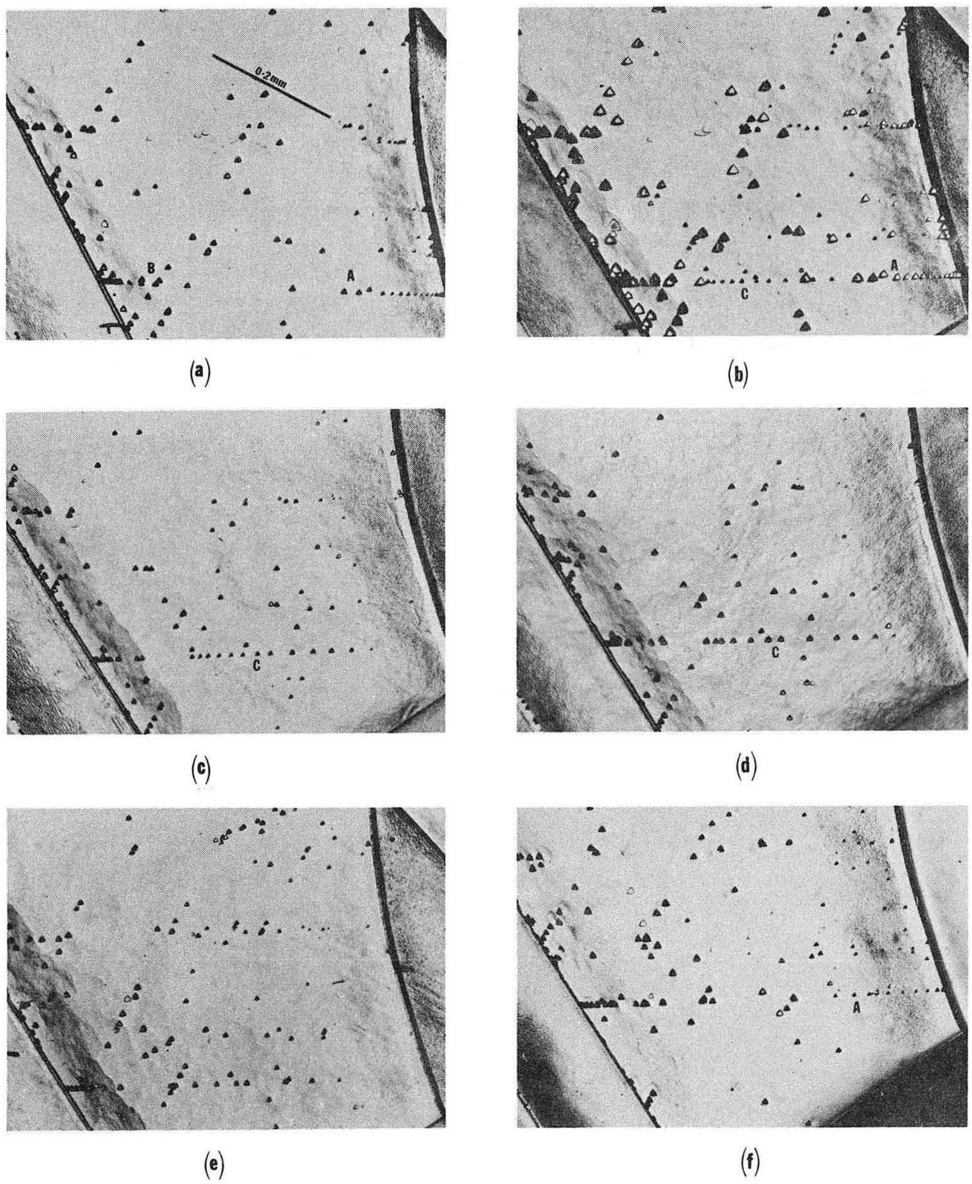
XBB 688-4982

Figure 12



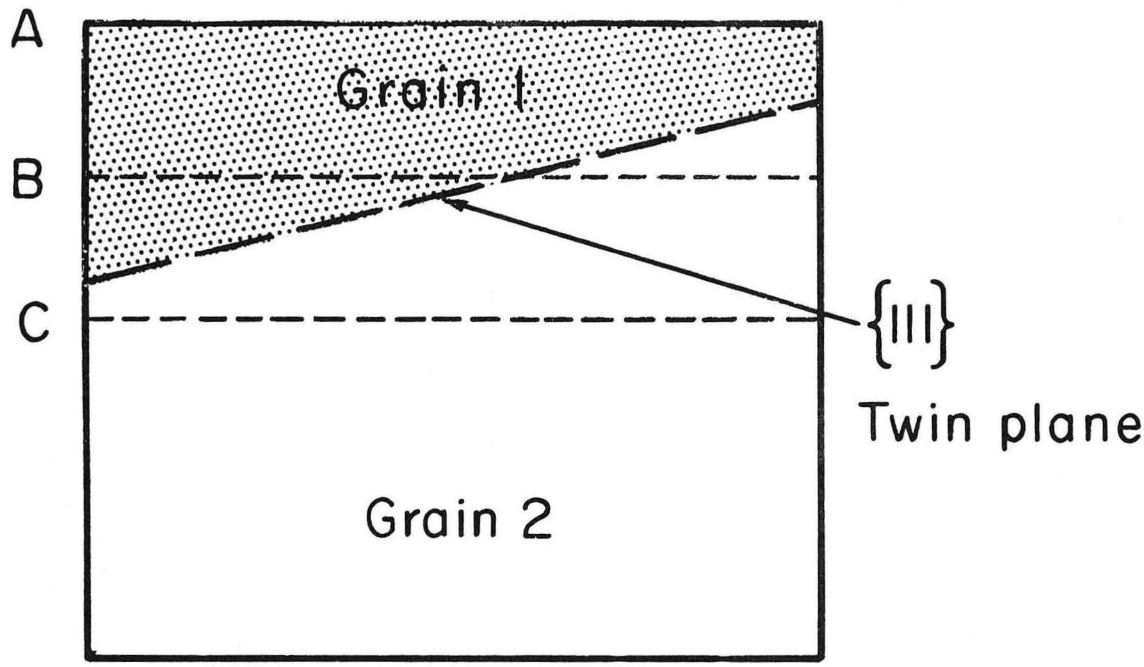
XBB 688-4981

Figure 13



XBB 688-4993

Figure 14



XBL687- 3326

Figure 15

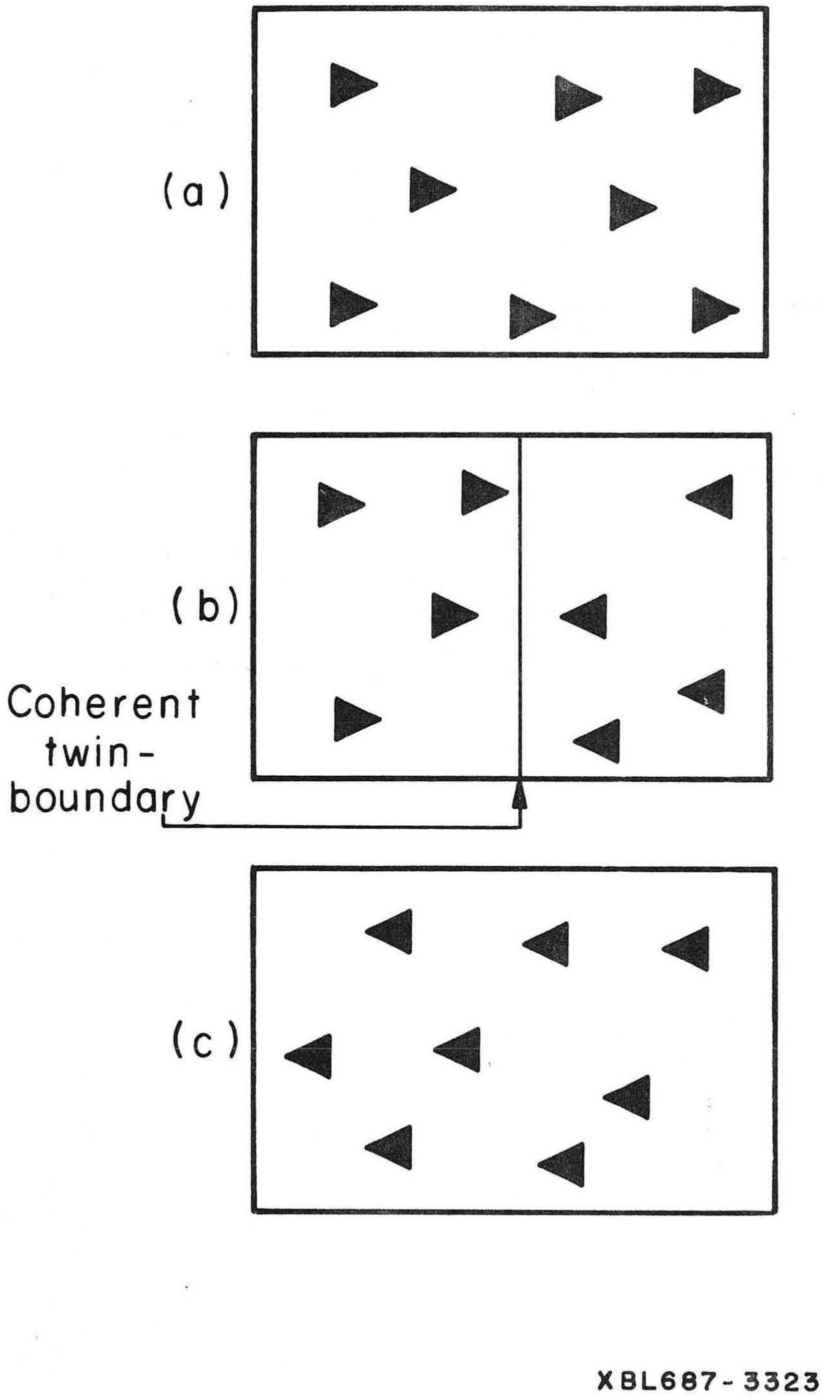
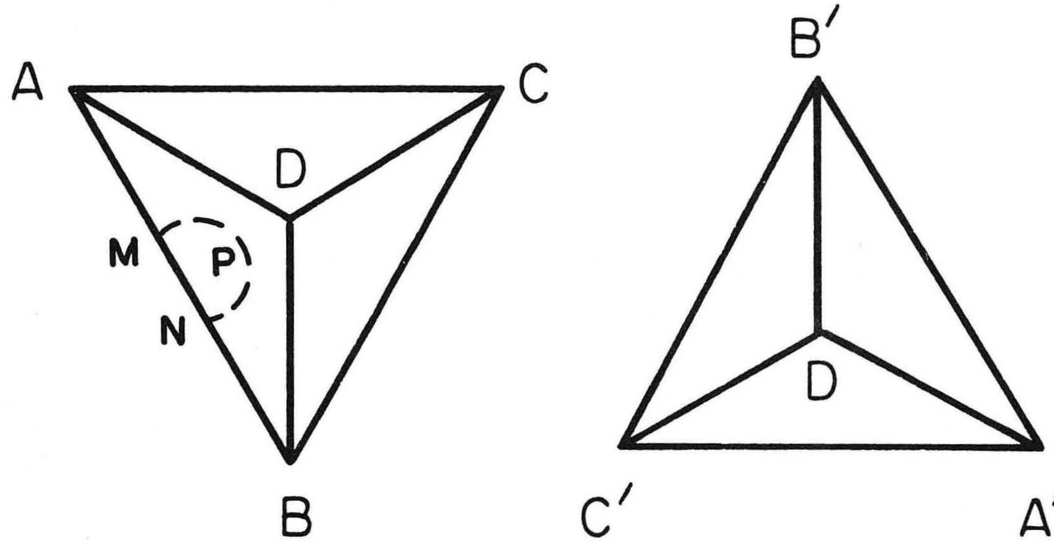
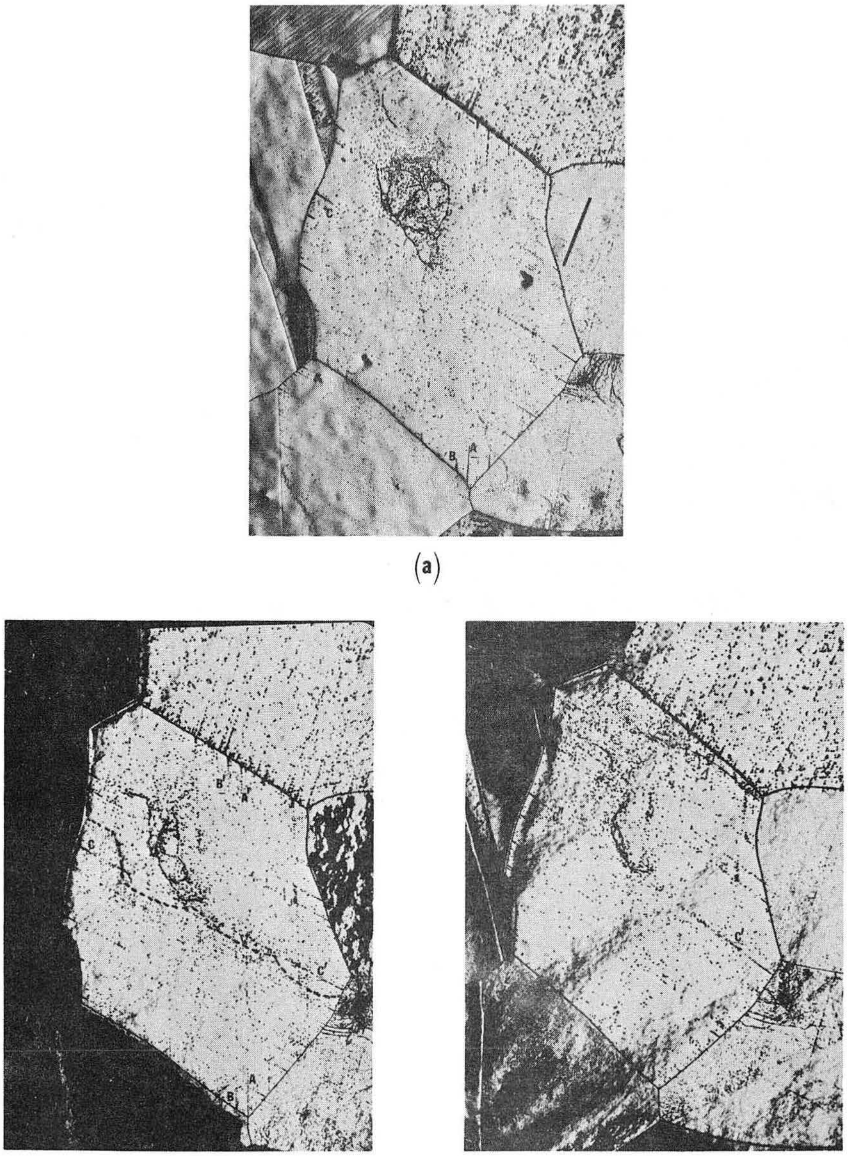


Figure 16



XBL687-3324

Figure 17

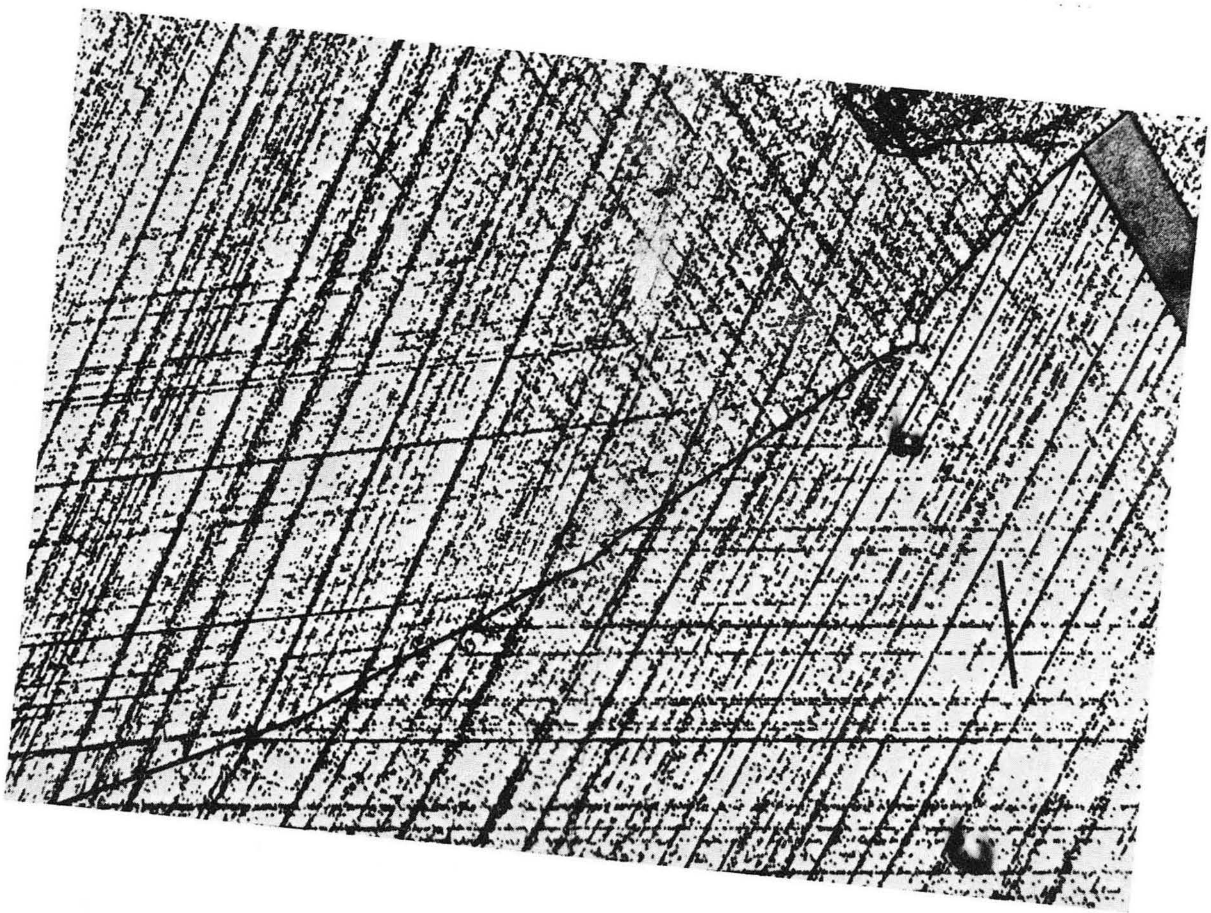


(a)

(b) (c)

XBB 688-4992

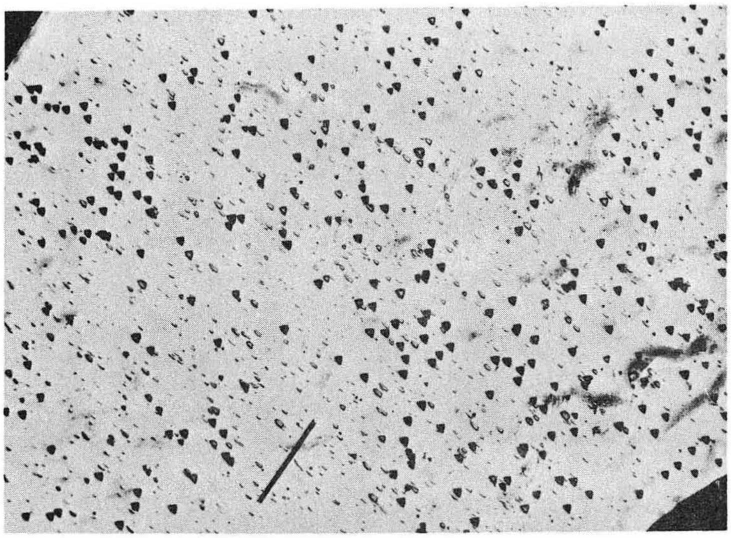
Figure 18



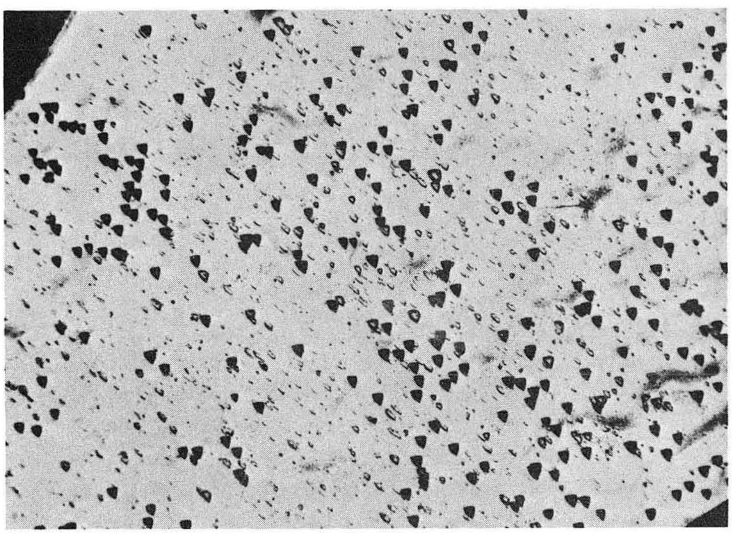
XBB 688-4983

Figure 19

(a)

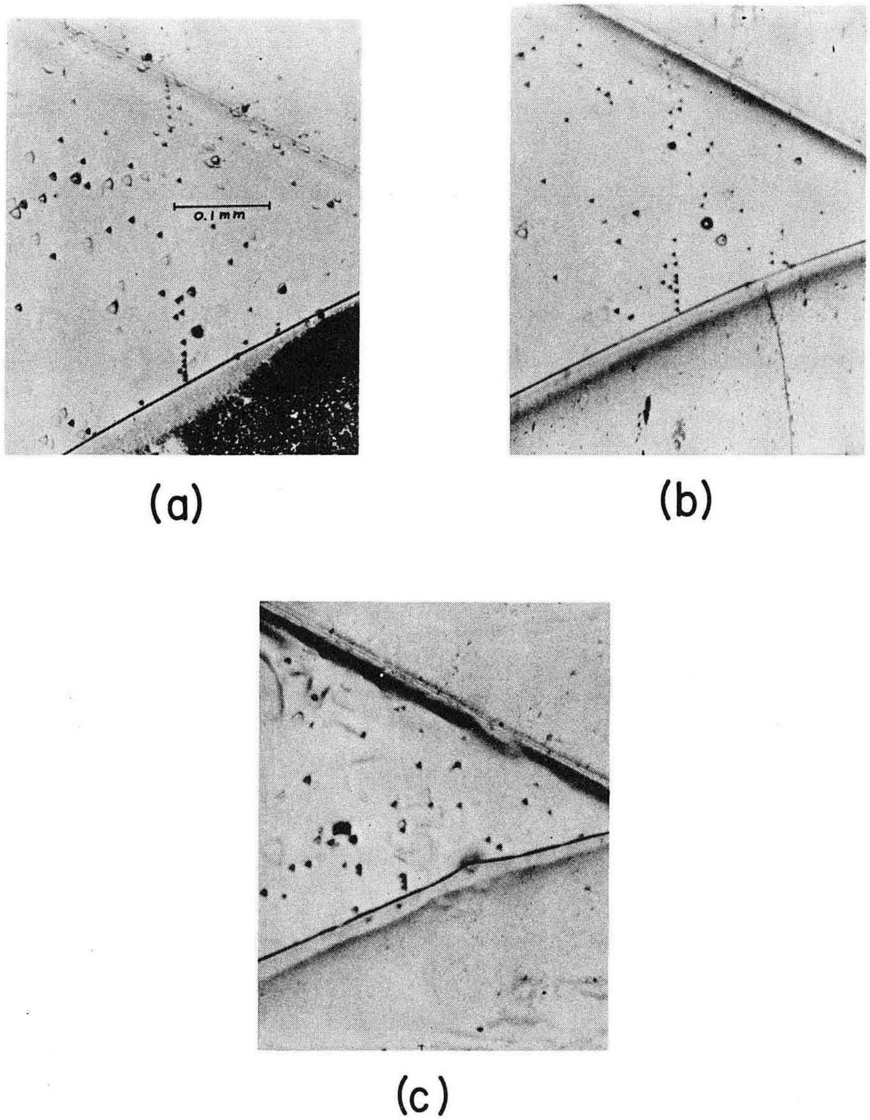


(b)



XBB 688-4990

Figure 20



XBB 689-5506

Figure 21

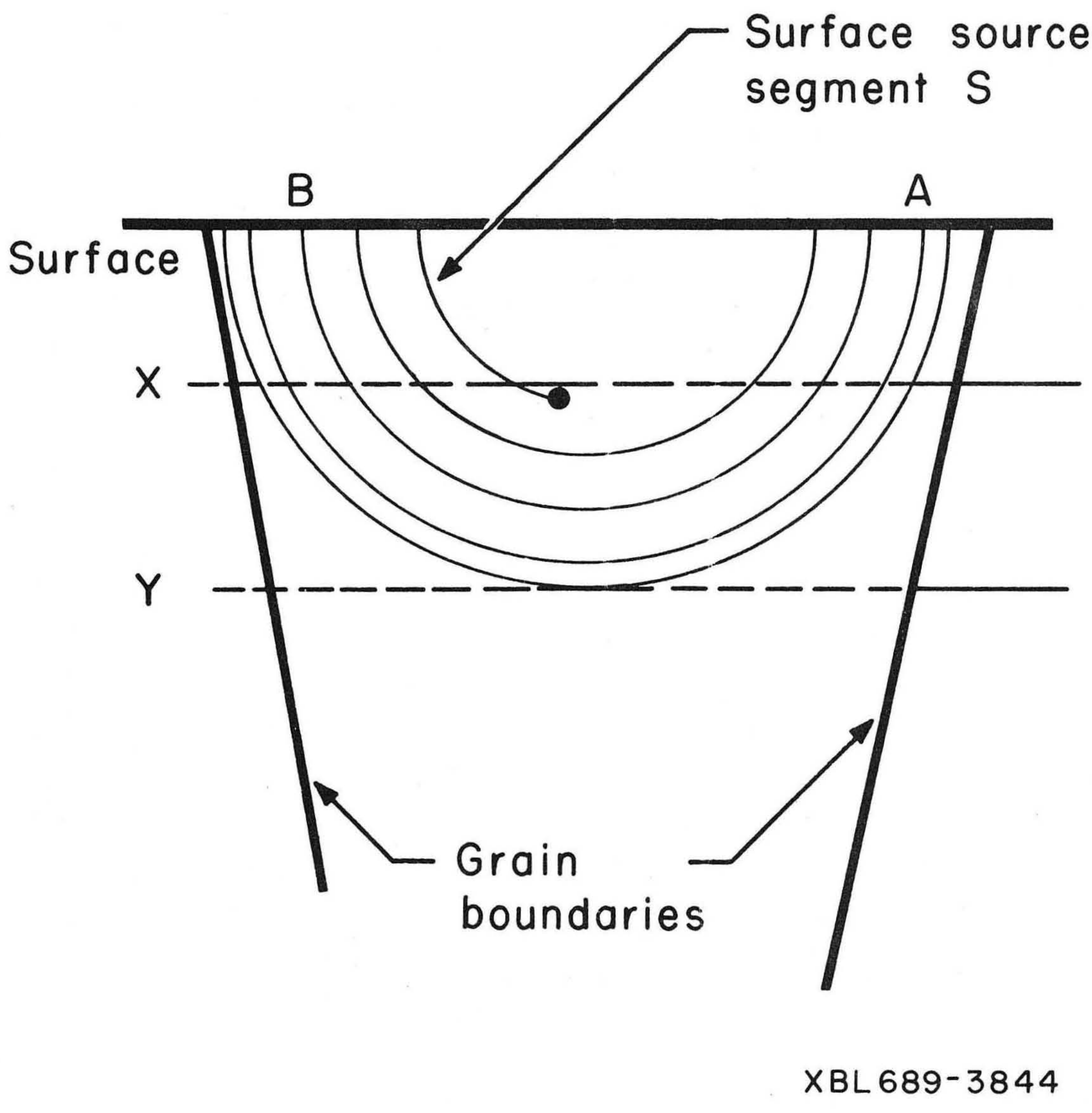

Surface source
segment S
B
A
Surface
X
Y
Grain
boundaries
XBL689-3844

Figure 22

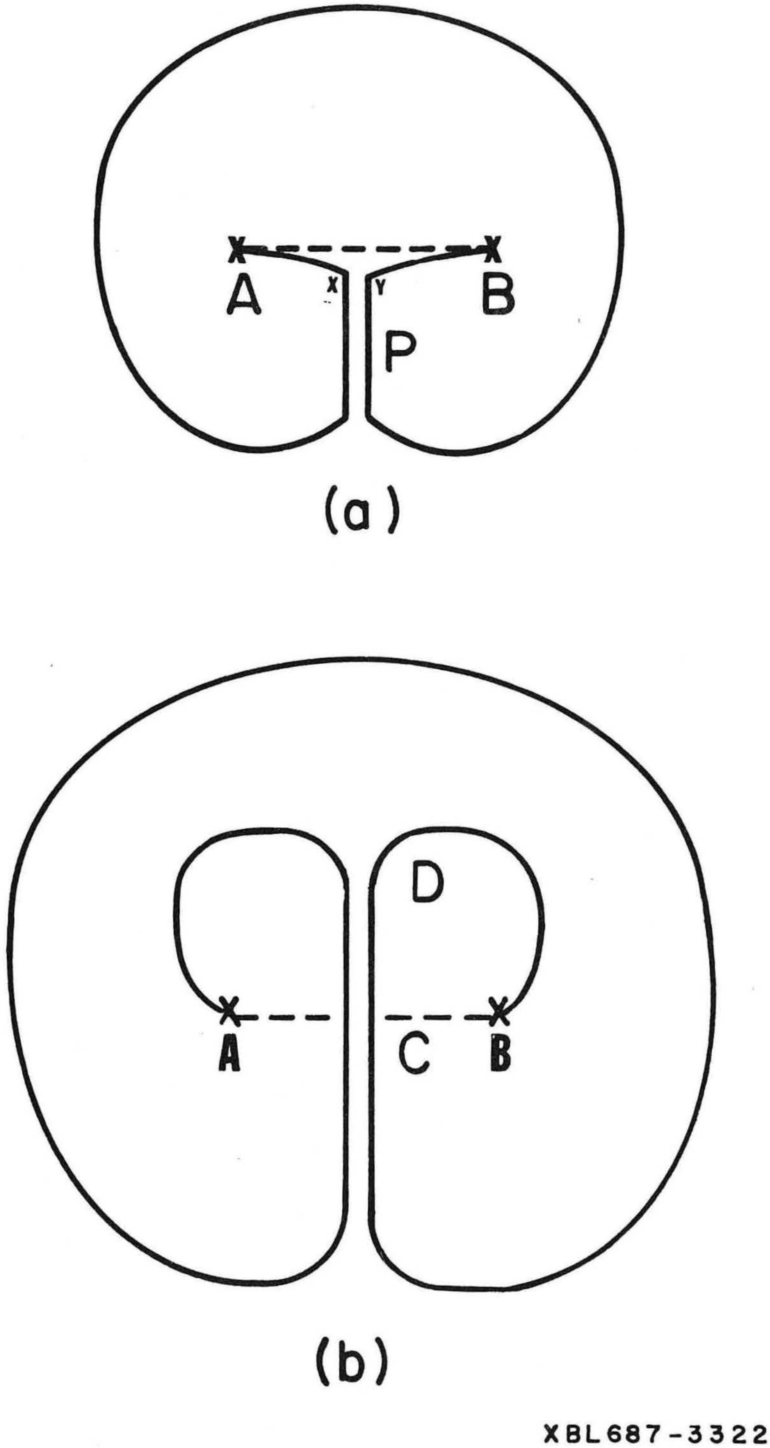
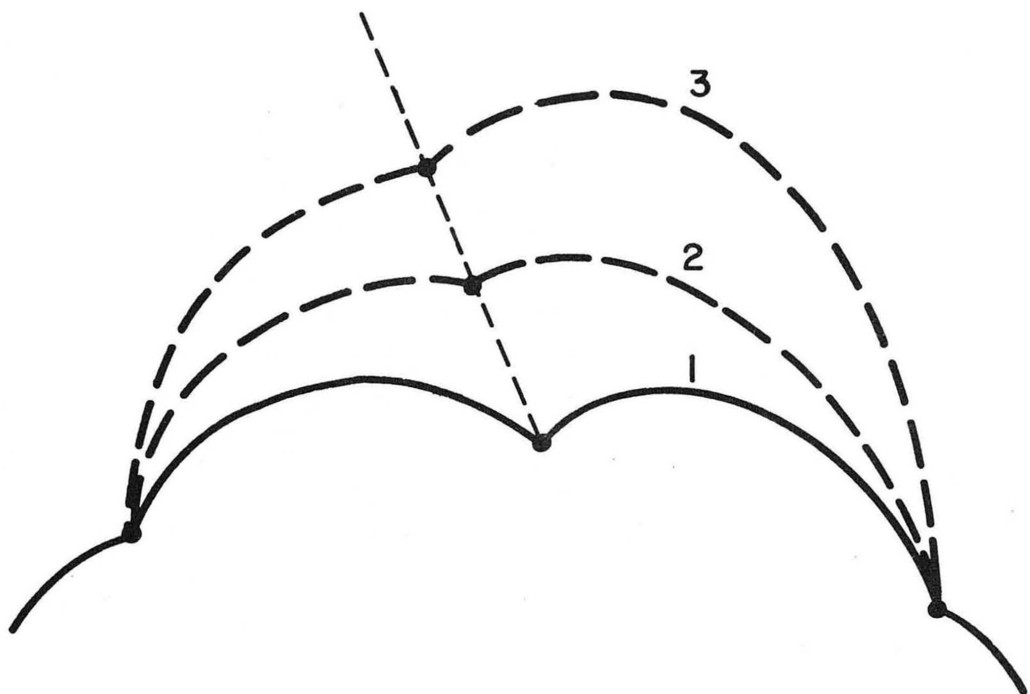
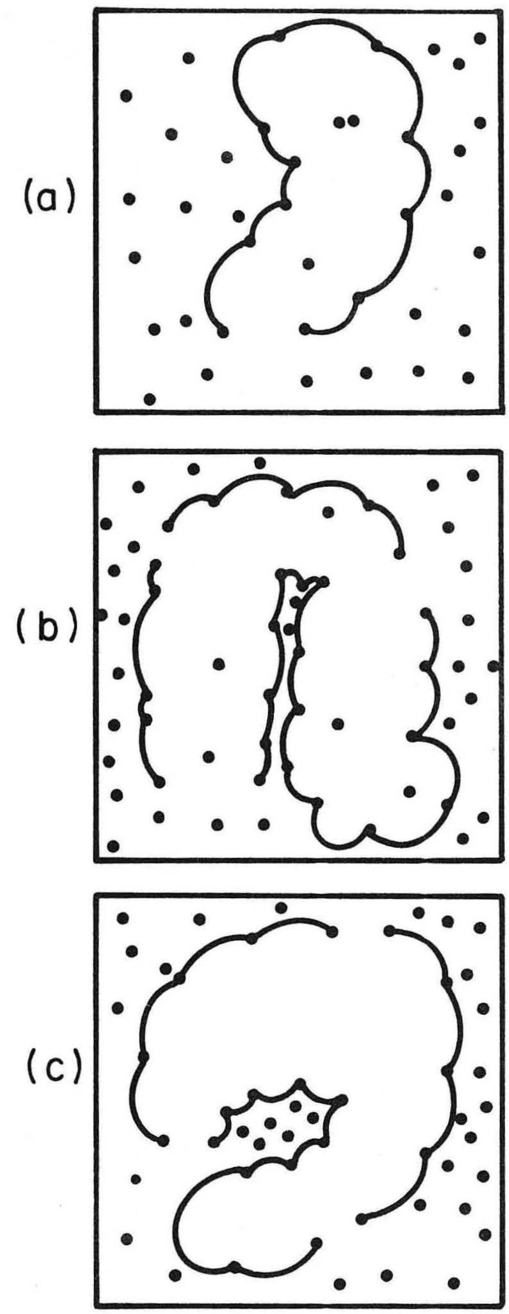


Figure 23



XBL687-3328

Figure 24



XBL687-3329

Figure 25

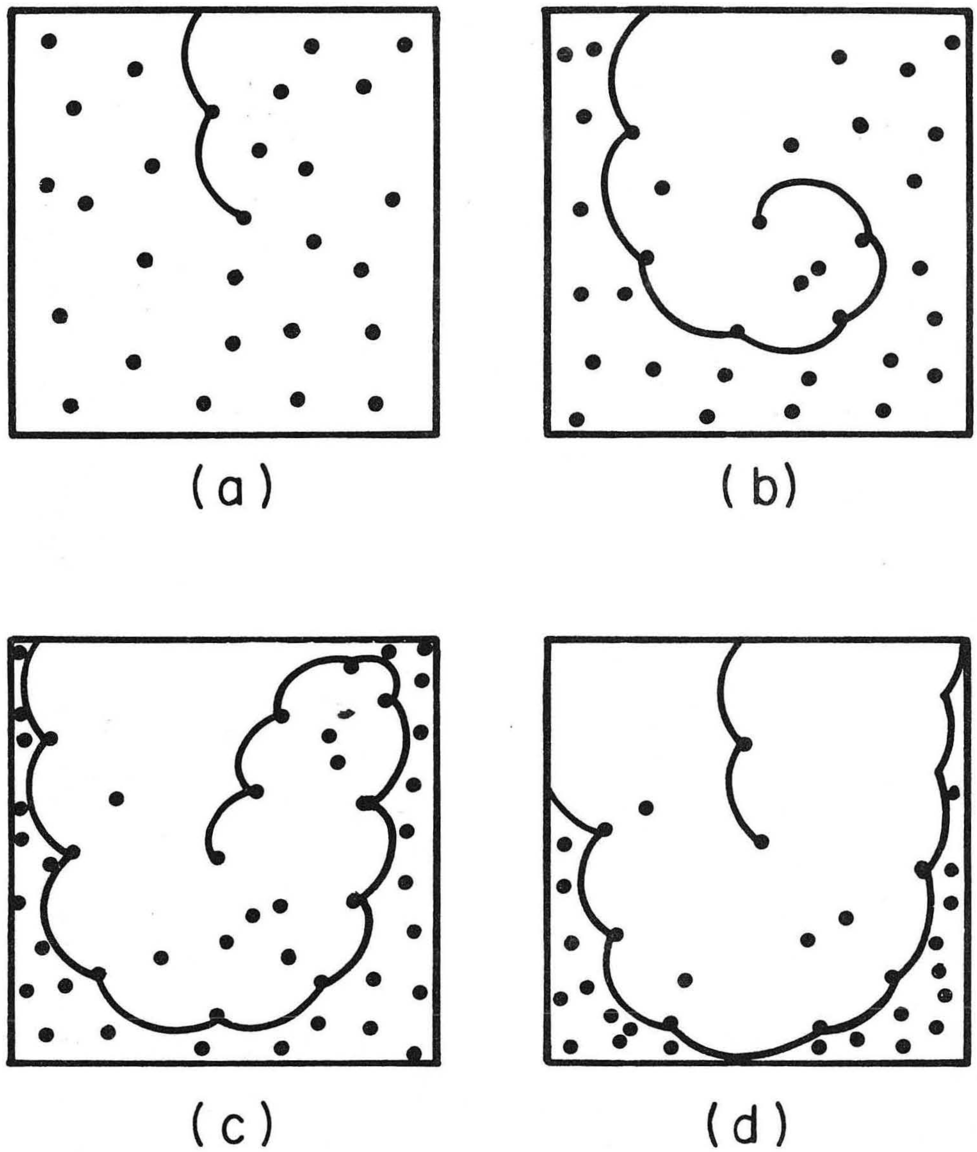
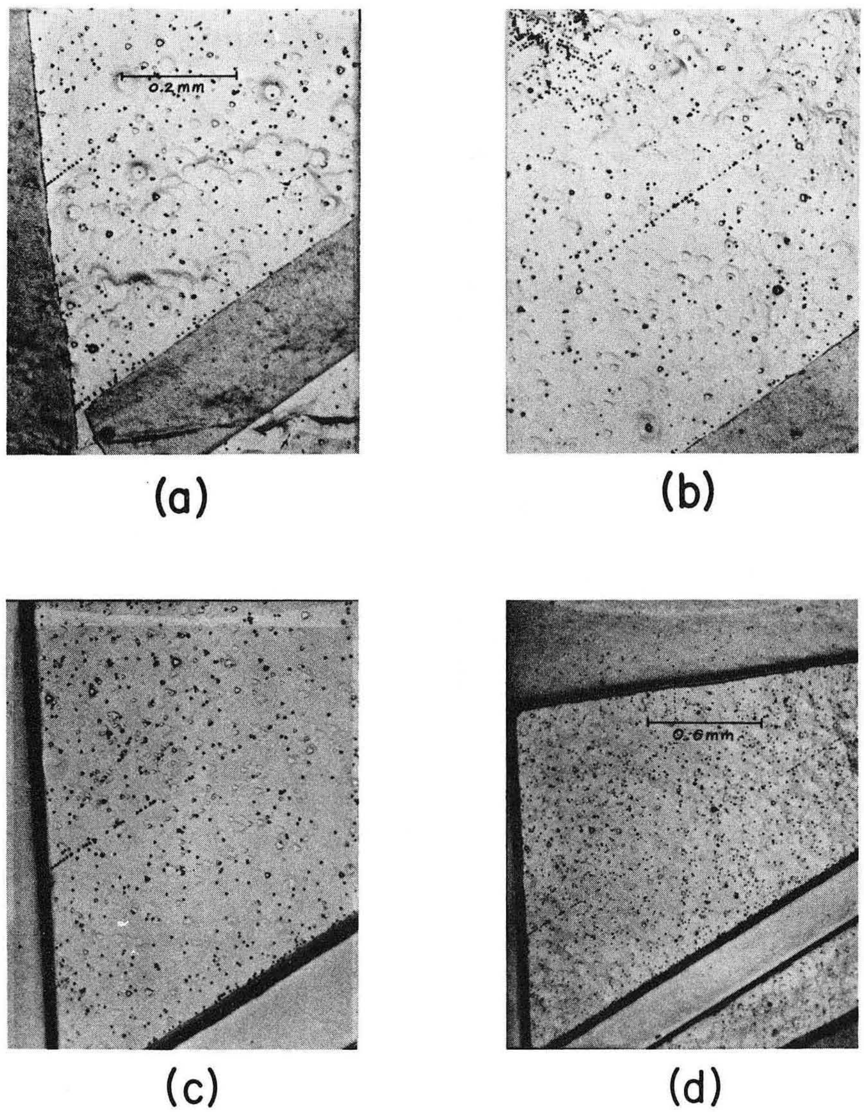


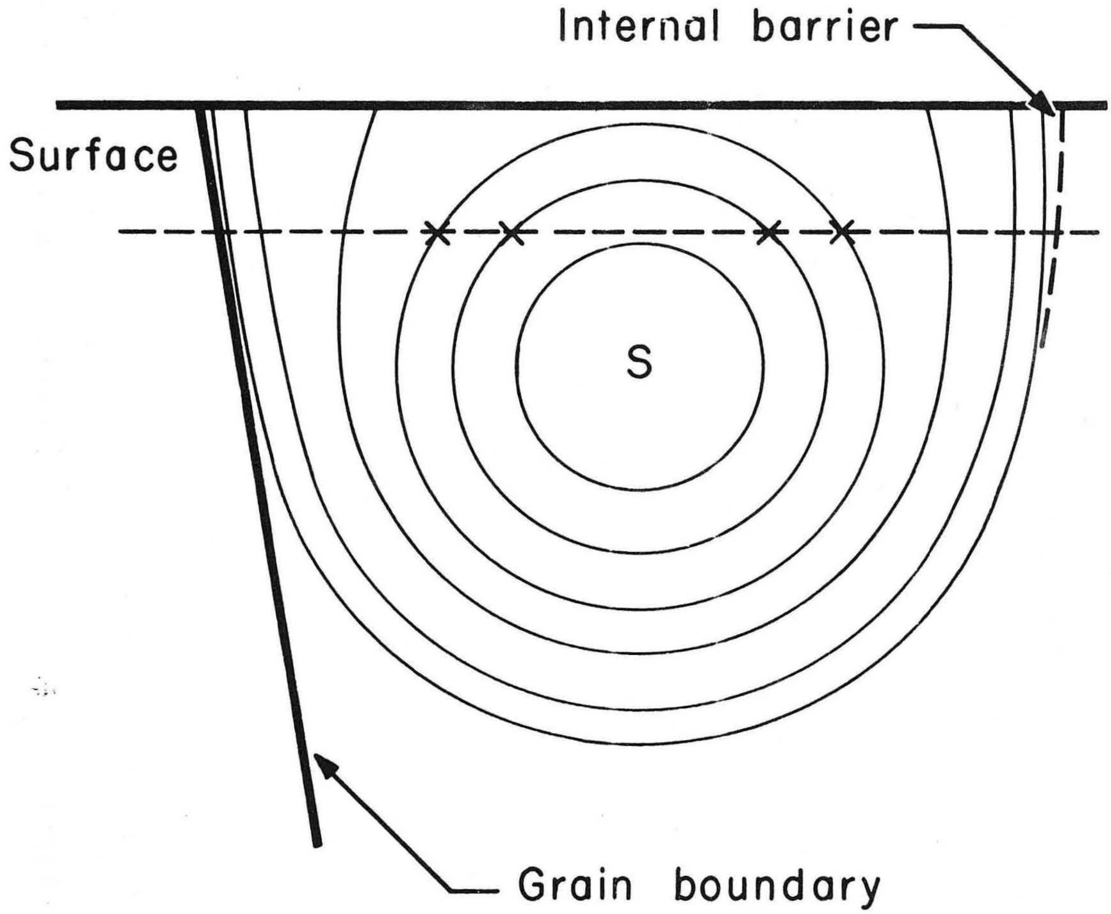
Figure 26

XBL 687-3330



XBB 689-5507

Figure 27



XBL689-3843

Figure 28

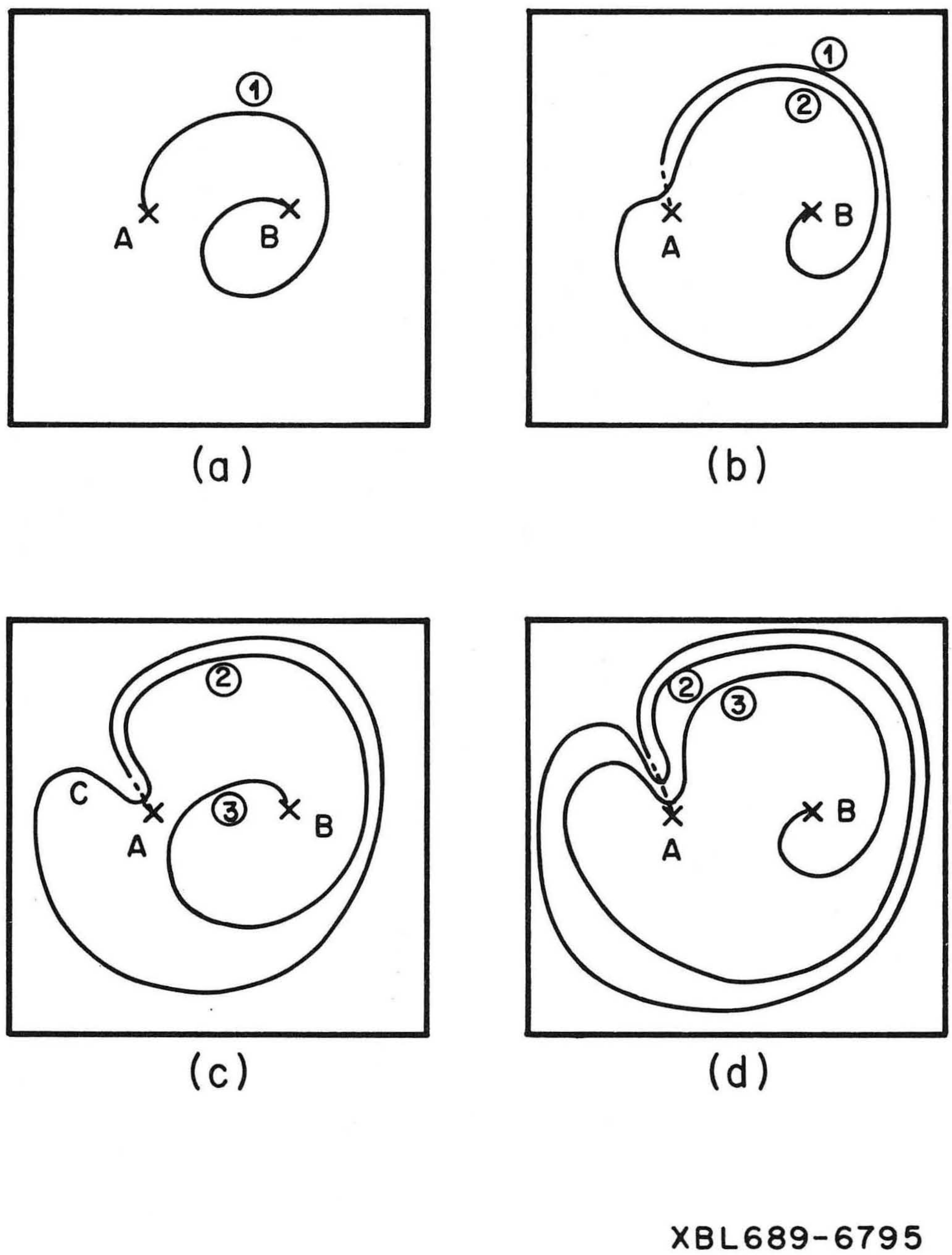
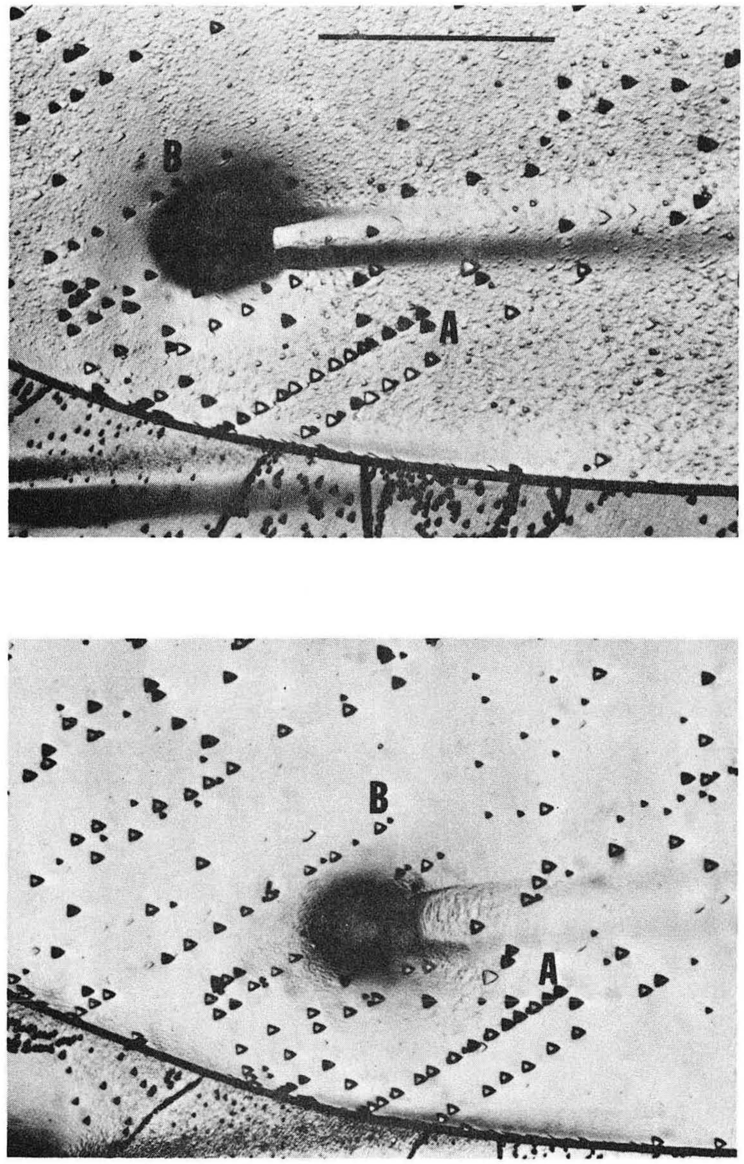


Fig. 29

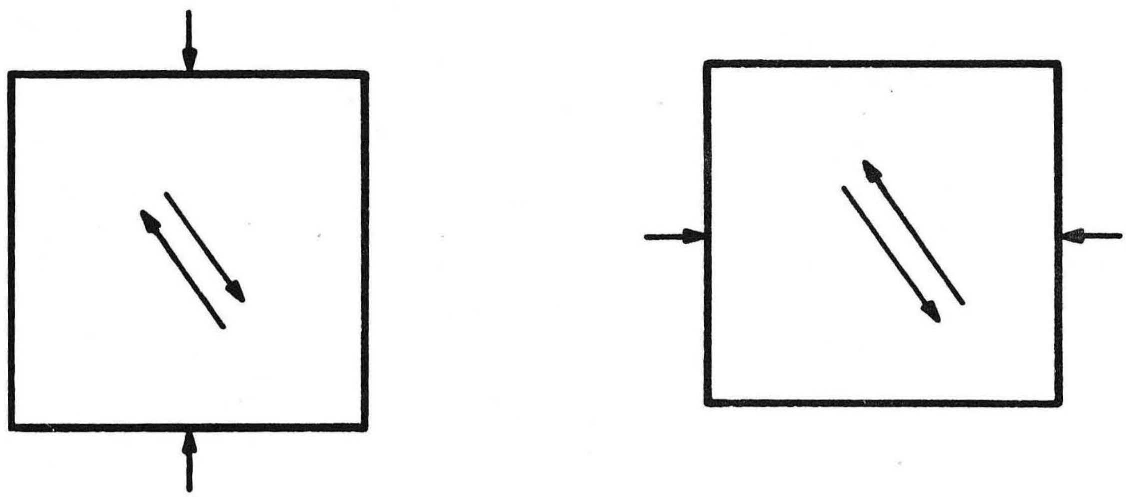
(a)

(b)



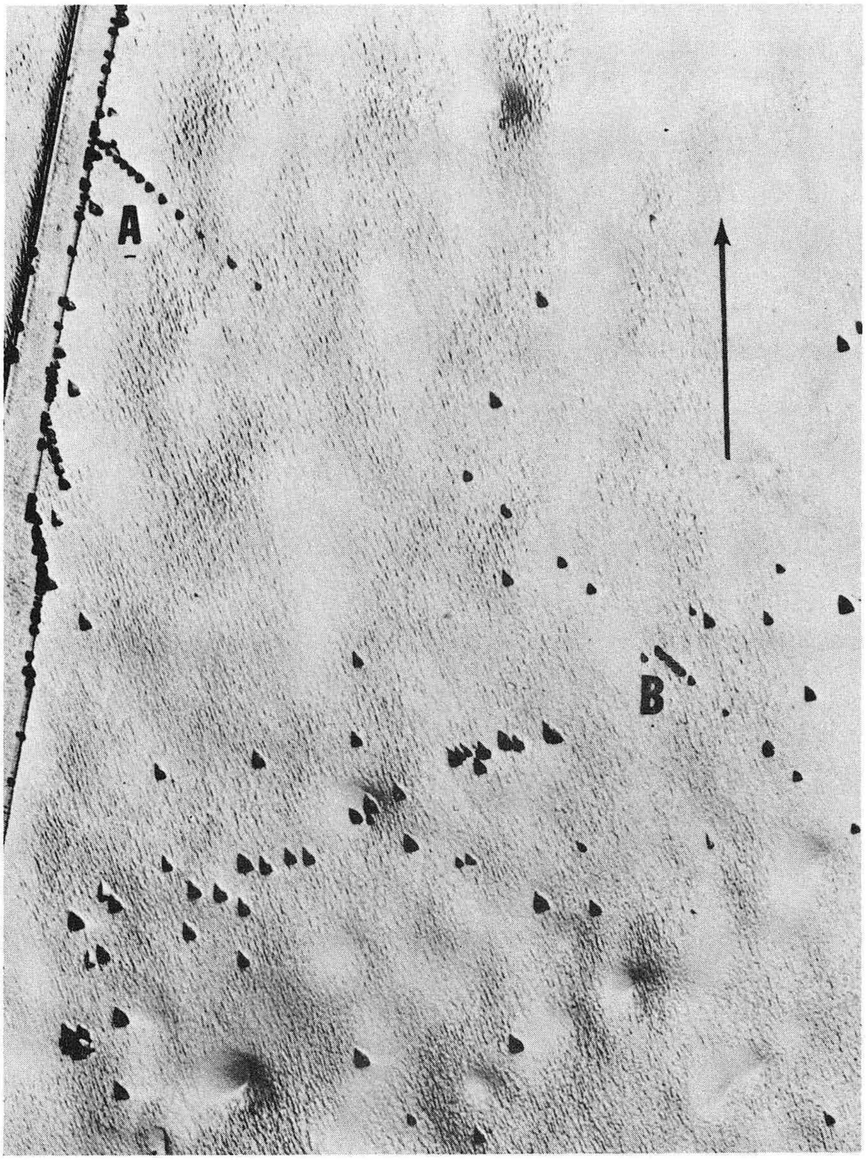
XBB 688-4989

Figure 30



XBL687-3325

Figure 31



XBB 688-4978

Figure 32

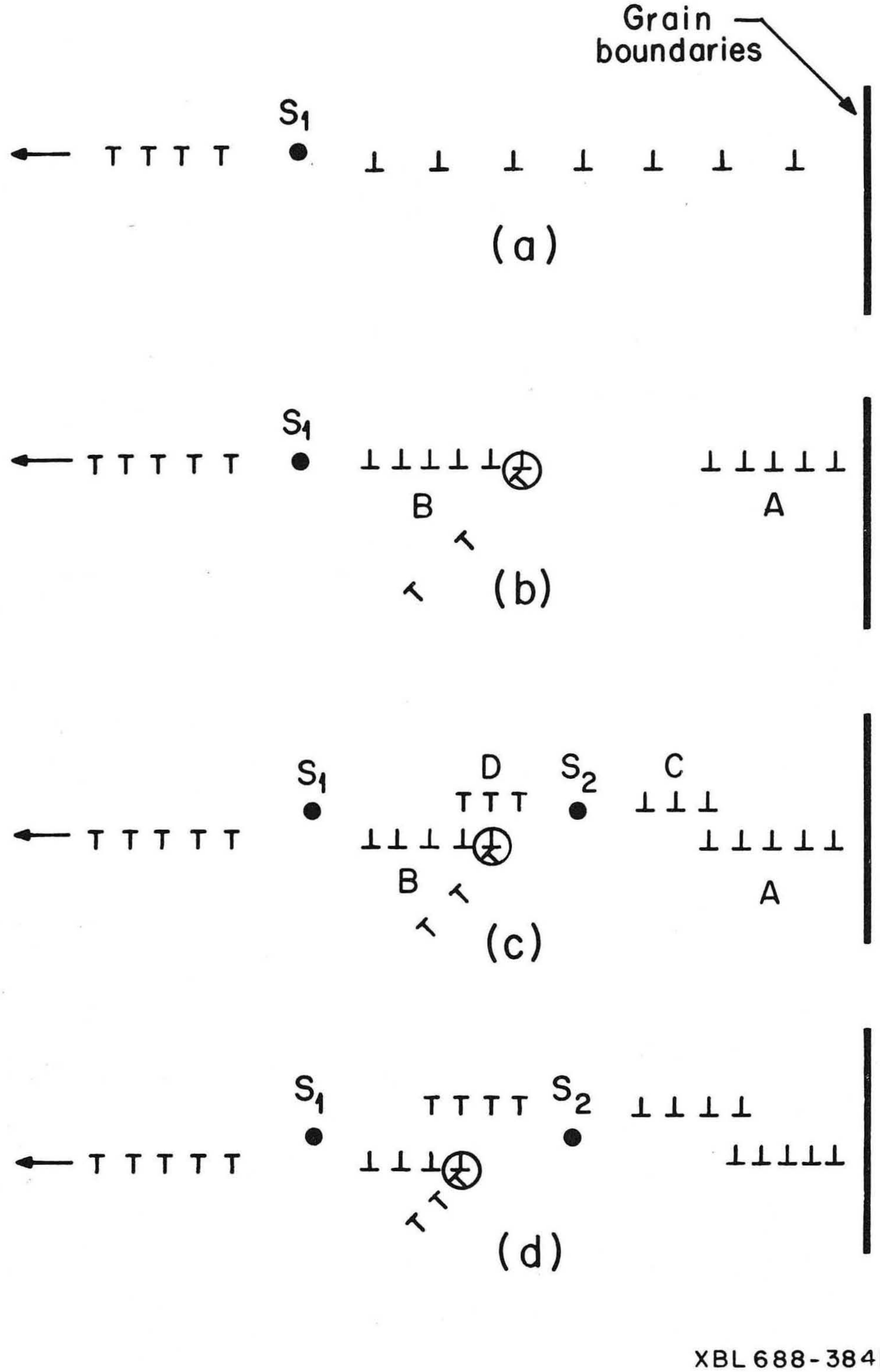
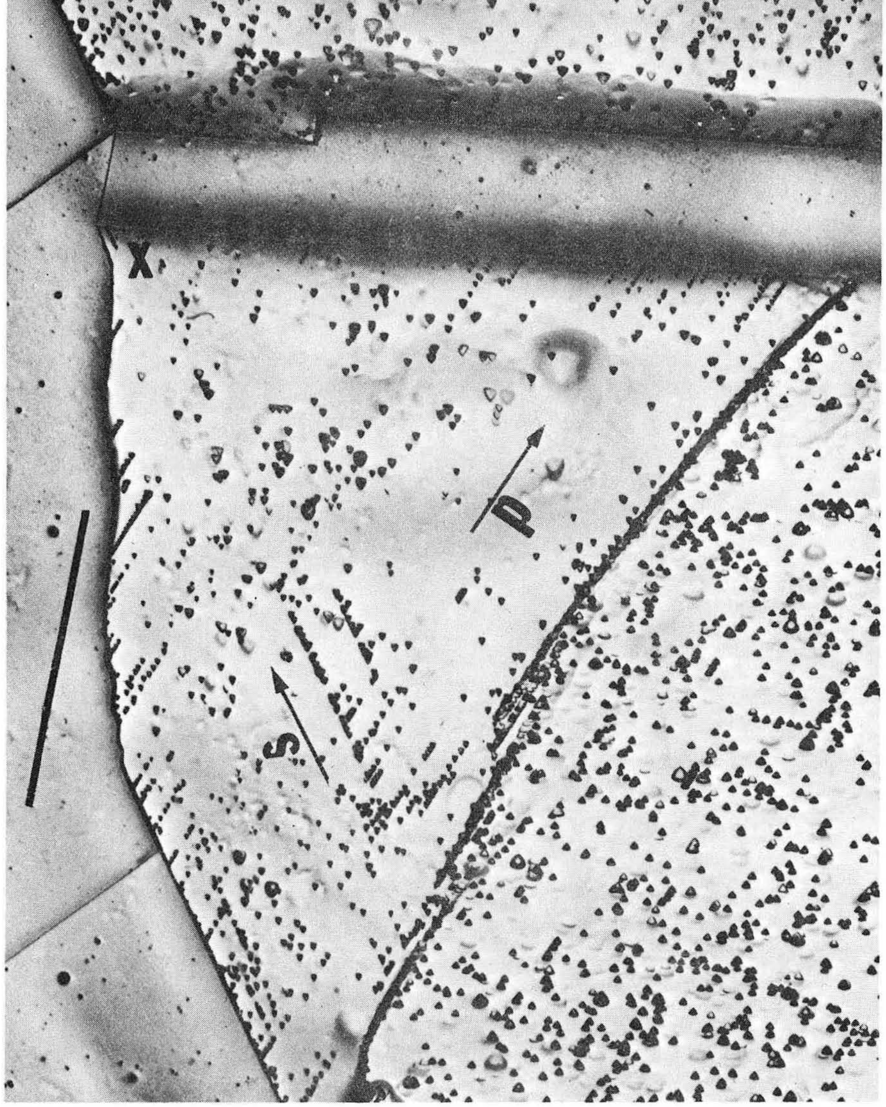
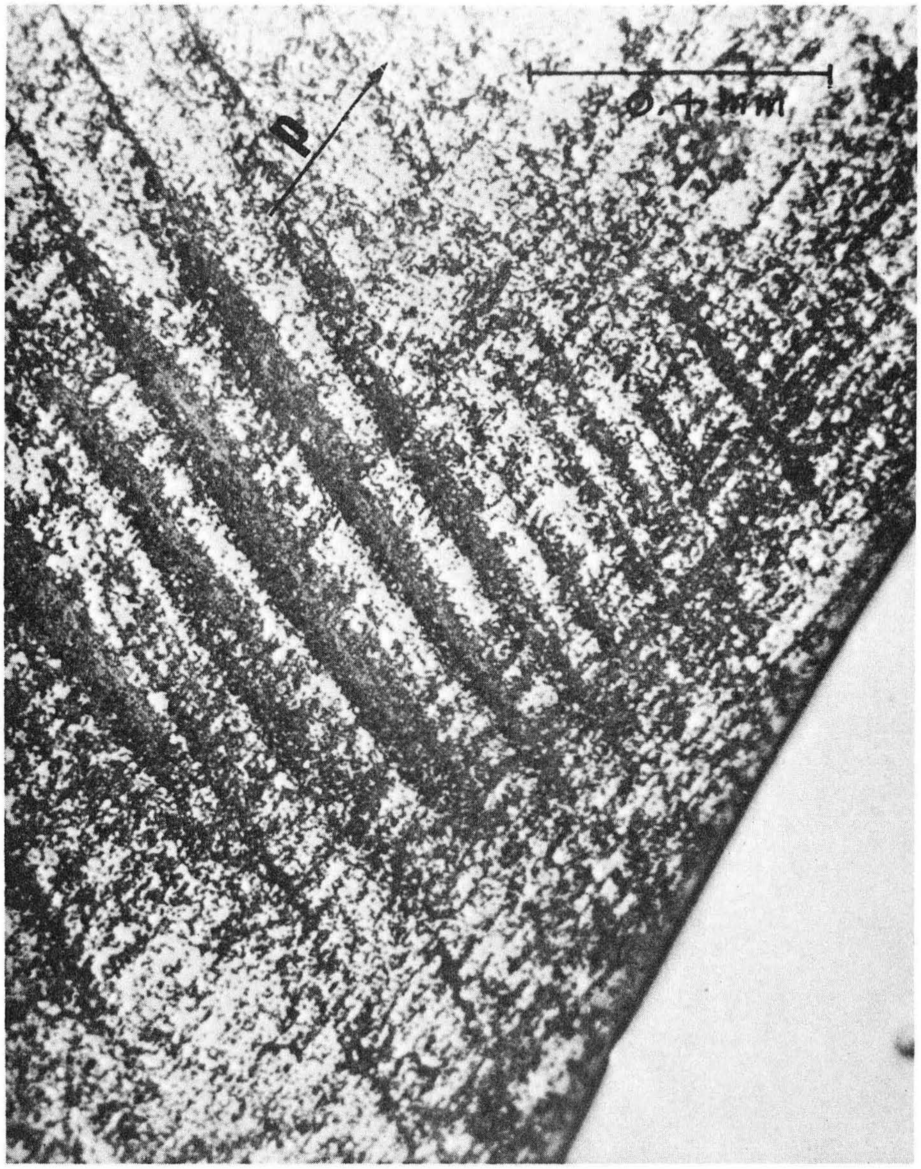


Figure 33



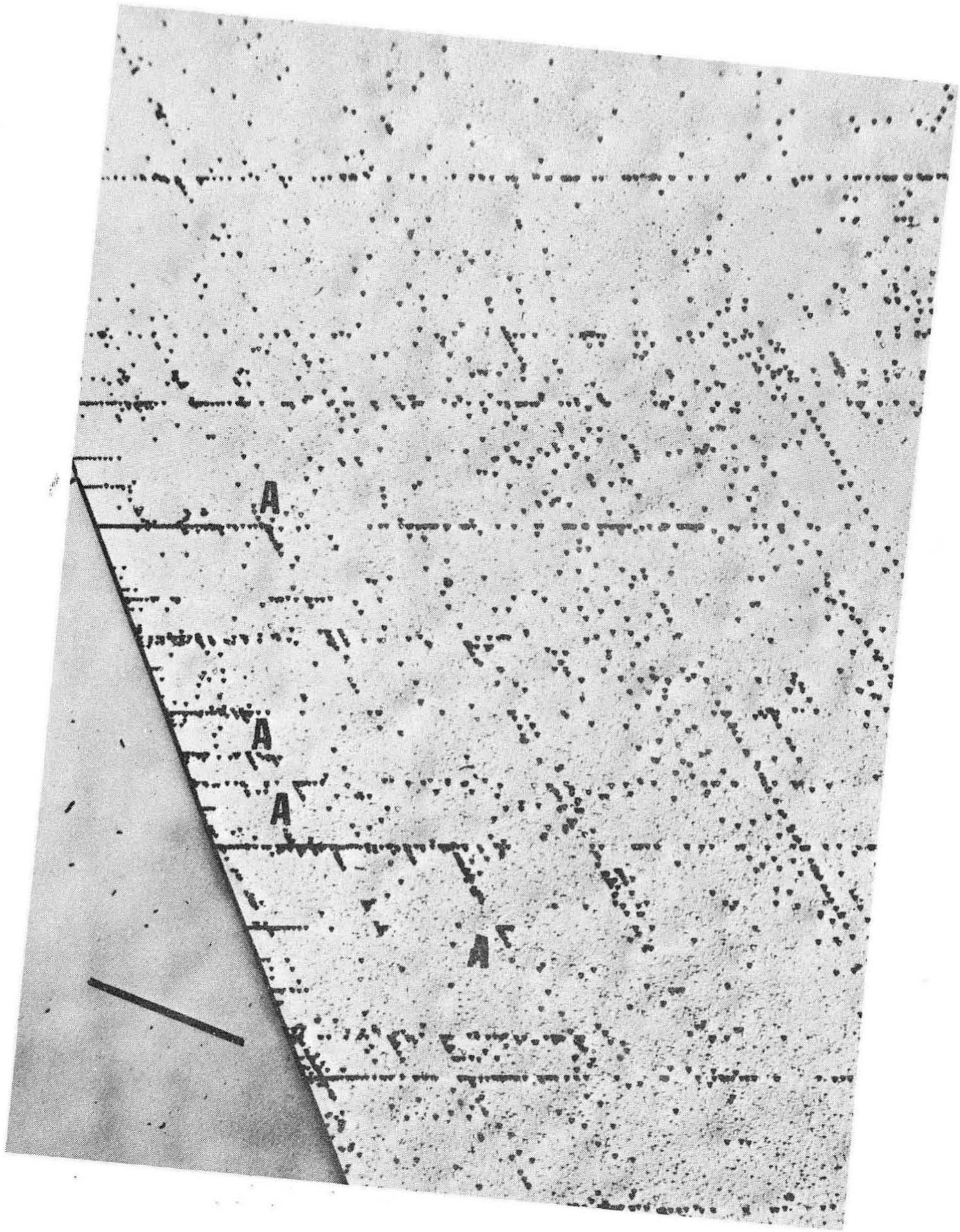
XBB 688-4975

Figure 34



XBB 688-4976

Figure 35



XBB 688-4974

Figure 36