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ABSTRACT

Pre-yield dislocation motion and multiplication were studied in large grained polycrystalline copper by the etch pit technique. Specimens were loaded in compression and dislocation arrangements were observed in both the stressed and unstressed conditions. The dislocation loops generated by the first few active sources on both primary and secondary slip systems generally traversed the entire cross section of the grain. The observations were consistent with the idea that jog density is an important factor in determining the mobility of a dislocation segment. The external surface was found to be a preferential site for dislocation multiplication even when grown in segments, where they cut the surface, were immobile and stress concentrations were carefully avoided. It is suggested that the rapid increase in length per unit volume of moving dislocation line that is associated with macroscopic yield requires motion of dislocation segments of high jog density.

INTRODUCTION

It is well known that high sensitivity strain measurements or dislocation etch pit observations can detect small plastic strains far below the ordinary 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. More recently, Young and his coworkers[7,8] have made direct x-ray topography observations of dislocations in lightly deformed copper single crystals with extremely low dislocation density. In spite of these large number of investigations many aspects of pre-yield plastic deformation in copper such as the effect of a free surface, the details of dislocation motion and multiplication and, in particular, the phenomenon of macroscopic yielding are still only incompletely understood. In the present investigation an attempt has been made to get some additional information on these aspects by studying, using the etch pit technique, dislocation behavior in the very early deformation stages of large grained copper polycrystals. Apart from providing a natural extension of the many previous single crystal studies the choice of polycrystals was motivated by the prospects of observing dislocation arrangements in interior grains (thereby avoiding or separating any surface effects) and by the expectation that grain boundaries would help in detecting the very early stages of slip activity by serving as effective barriers to moving dislocations.

II. EXPERIMENTAL PROCEDURE

Since dislocations in copper can be revealed by etching only when a low index plane is parallel to the surface of observation special experimental techniques were needed to prepare specimens that had one or more surface grains in an etchable orientation. Since most reliable etchants have been developed only for planes of the {111} type in copper,[9] experiments were aimed at obtaining a specimen in which at least one grain would have a {111} plane parallel to the surface. Recrystallization of extruded OFHC copper rods resulted in the preferential formation of grains with a {111} type plane approximately normal to the extruding direction. Specimens were obtained by machining from 19 mm square extruded OFHC copper rods (analysis given in Table I), chemically polishing[10] a surface that was normal 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 and an average grain size of about 5 mm.

The specimens were compressed at room temperature using either an Instron machine or a micro-compression device schematically illustrated in Fig. 1. The device was made of stainless steel and was essentially like a C clamp in which by turning the micrometer head an increasing stress could be applied to the specimen placed between the bottom end plate and the modified spindle of the micrometer as shown in the figure. The ball and socket joint at the head of the spindle ensured proper alignment and a uniform distribution of the load. The stainless steel plate P kept flush against the vertical arm minimized any torque that might have been transmitted to the specimen during loading. The two teflon plates served to electrically insulate the specimen during electropolishing. Insulating lacquer was applied on all the surfaces of the specimen except the one on which observations were to be made and the one in contact with the stainless steel plate at the bottom through which electrical connection was made to the specimen. A rough estimate of the load applied by the device was obtained by finding the number of turns required on the micrometer to cause the same deflection of the end plates as that caused by hanging a known weight from one of the end plates. The particular advantage of the micro-compression device was that specimens could be etched and observed under the microscope while under stress. Also it enabled investigation of the dislocation distribution down to a limited depth below the surface while in the stressed condition by immersing the whole device in the polishing solution and re-etching. Furthermore, specimens could be deformed while immersed in the electropolishing solution and etched before and after drying so as to reveal possible effects of surface films or deposits that may be formed 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 0.1 amps/cm^2. The etchant used to reveal dislocations was the one developed by Livingston.[9] Unless otherwise mentioned the amount of material removed from the surface between successive etchings was always of the order of 5-10 microns. A double etching technique was used whenever the new positions of dislocations were to be related to the old ones. When interior grains were to be examined after deformation the removal of the necessarily large amounts of material from the surface without introducing any mechanical damage was accomplished in a modified polishing apparatus[11] using a solution of 1:1 nitric acid followed by the chemical polishing solution.

III. RESULTS AND DISCUSSION

The recrystallized grains had a relatively high perfection with dislocation densities always less than $10^4/cm^2$ and frequently of the order of $10^3/cm^2$. Dislocations were quite uniformly distributed and there were usually no sub-boundaries. Motion of the ends of dislocation segments at the surface could be detected only after the resolved shear stress was raised to about 15-20 g/mm^2. At this stress level there was usually also dislocation multiplication. This is clearly seen in Fig. 2 which shows a grain etched before and after application of a stress of 17 g/mm^2.[12] The black circles indicate a few typical cases of simple dislocation motion and the pileups, visible near the grain boundaries, indicate that dislocation multiplication has occurred. Additional examples of some early dislocation pileups can be seen in Figs. 3 to 5. Figure 3 shows a grain etched before and after applying a stress of 20 g/mm^2 and Figs. 4 and 5 show grains etched under a stress of 16 and 22 g/mm^2 respectively.

The fact that isolated dislocation pileups at the grain boundaries such as in Figs. 3 to 5 were frequently the very first indications of the occurrence of plastic deformation in polycrystals implies that the dislocation loops emanating from the first few sources to become active are able to glide over long distances. In most of the previous experiments using single crystals, where observations were usually made in the stress relaxed condition, dislocation multiplication would have been hard to detect in the early deformation stages because the first dislocation loops could have completely escaped through the surface. The present experiments also showed, contrary to usual reports, that dislocations of secondary systems sometimes also travel very long distances in the early stages of plastic deformation. Figure 6 shows a grain etched after application of a stress of 25 g/mm^2. The primary and secondary slip directions are indicated by the letters 'p' and 's' respectively. The group of dislocations marked X represents the first few dislocations sent out by a secondary source. These dislocations were able to travel all the way to the grain boundaries. However, at a later stage the great activity on primary planes results in numerous barriers to the motion of secondary dislocations so that long distance motion becomes impossible. Glide layers across which secondary dislocation loops spread at an early stage become clearly "decorated" with primary dislocations due to the larger number of interactions. A few good examples of such decorated secondary slip plane traces can be seen at A and B in Fig. 6.

There were three significant aspects about dislocation multiplication that were clearly evident from the examination of a large number of specimens. First, the sources for early dislocation multiplication were often not dislocations originally intersecting the surface as can be seen clearly from Fig. 3. Careful examination of the slip plane traces corresponding to pileups A and B along the entire length of the grain fails to show any large flat bottomed pits which might represent the original position of a surface-intersecting dislocation segment that could have moved and acted as a single ended Frank-Read source. Secondly, dislocation pileups were rarely seen in the interior grains at stress levels at which they were observed in large numbers in the surface grains. Finally, the early dislocation pileups usually contained dislocations belonging to distinctly different glide layers. Figures 4 and 5 are particularly good examples of such pileups. These observations as well as the generally higher stress (10-15 g/mm^2) needed for the motion of surface dislocation segments in the present work as compared to a stress of only 2-4 g/mm^2 in previous experiments[2,5,6] can be explained in terms of differences in specimen preparation techniques.

As discussed by Petroff and Washburn[5] the dislocation network in a crystal would tend, at high temperatures, to approach a configuration of metastable equilibrium by both conservative and nonconservative motion. Assuming that the energy associated with a jog is small, elastic strain energy will be minimized when dislocation segments approach linearity and nodes become symmetrical. In a metal of medium stacking fault energy like copper few if any dislocation segments, in an annealed or recrystallized specimen, will lie exactly on {111} planes; most of the dislocation segments will be jogged. Because of image forces dislocations intersecting a free surface tend to become normal to the surface, at least up to a certain depth, and hence consistently acquire a comparatively high jog density. Some of those dislocation segments with both ends fixed within the crystal are likely to have the lowest jog densities. When a new surface is exposed by extensive polishing just before application of a load, as has been done in previous experiments, some of the most mobile dislocations with low jog densities will also intersect the surface at small angles so as to be able to shorten their length by conservative motion. When a stress is applied some of these should start to move even at the smallest stresses but multiplication should not take place. This motion will not be reversible on removal of the applied stress since reverse motion involves an increase in the length of the segment. This explanation is consistent with the usual etch-pit observations[2,5,6] that the early dislocation motions are largely irreversible. As the stress is raised a gradually increasing fraction of the dislocations that intersect the surface should be expected to move as observed by Young[4] and by Petroff and Washburn.[5] In the present investigation where only about 5 microns of material was removed from the surface after the recrystallization treatment and prior to the initial deformation most of the dislocations cutting the surface should have been heavily jogged. Under these conditions their critical stress for motion should be higher than that for a few less jogged interior segments. Consequently some of the inner dislocation segments might be expected to move and multiply before motion of the surface segments. This indeed appeared to be the case. When dislocation pileups were first detected in a double etching experiment the sources were not always surface segments.

The second observation, that pileups were predominantly observed only on surface grains, implies that the surface of a crystal is indeed a region of enhanced slip activity in the very early stages of plastic deformation. There are several reasons why a surface might be expected to act as the site for early dislocation multiplication. Dislocations accidentally introduced at surfaces during normal specimen preparation and deformation techniques can act as sources for dislocation multiplication. Irregularities such as scratches and etch pits can provide stress concentrations at which plastic flow can be initiated. Films or deposits left behind in cases where the specimens are electropolished and dried before the deformation can cause stress concentrations. Even if an atomically smooth surface is achieved there might be enhanced dislocation activity near the surface because dislocations intersecting a free surface can theoretically start multiplying by the Frank-Read mechanism at stresses half that needed to operate interior sources of the same length.[13] In the present experiments these explanations are inadequate. The chances of accidentally introducing dislocations had been minimized because there was no acid-sawing or wheel-polishing after the recrystallization treatment. The final light electropolishing was carried out with the specimen already mounted in the microcompression device. Lack of correlation between some of the first pileups and any surface emergent segments of the grown-in network shows that most potential surface sources were inactive. In specimens which were loaded while immersed in the electropolishing solution and etched before and after drying there was no additional dislocation motion or multiplication induced as a result of the drying operation which suggests that surface deposits or films were not causing stress concentrations. Figures 7(a) and 7(b) show a grain etched in the two respective conditions under a stress of 17 g/mm^2. The only noticeable difference is an increase in the size of the etch pits in (b) as compared to (a). It is thus to be concluded that even under experimental conditions where accidentally introduced surface damage was practically eliminated and where the surface intersecting dislocation segments were relatively immobile the surface still acted as a preferential site for multiplication.

A tentative mechanism that can explain this observation is as follows. When a small stress is applied to an annealed crystal the first segments to glide should be those having the lowest jog density on the slip systems sustaining the highest resolved shear stress. Since most of the surface-intersecting dislocation segments would have been heavily jogged under the present experimental conditions the first segments to glide will be lightly jogged inner dislocation segments of greater than average length. Consider the expansion of such a segment AB shown in Fig. 8(a) under an increasing applied stress. If the average jog height is only a few angstroms they would either be dragged along with the moving dislocation causing the production of point defects or if the original segment is near enough to edge orientation they may move conservatively. In either case the stress required would depend on the separation between the jogs. After some motion two jog-free segments AC and BD would be generated as in Fig. 8(b) and their continued expansion would then lead successively to configurations shown in Figs. 8(c) and 8(d). If the initial jog density had been small the opposite segments of the expanding loop would approach each other in Fig. 8(d) on sufficiently close glide planes to form a stable dipole except in the special case where the initial segment is near pure edge orientation and therefore the dipole would be nearly pure screw. Formation of a dipole would usually cause the source to cease operating after it had sent out only one dislocation loop. Generation of a second loop would require an increase in the applied stress by an amount that could be as much as a factor of two. At a higher stress the segments AP and BP could again bow out extending the length of the dipole. It seems reasonable to suppose that most of the first sources to operate would be stopped by this mechanism after the formation of a single loop. To explain the formation of some pileups during early pre-yield deformation it is necessary to consider those conditions under which a jogged source segment might not be bisected by a dipole after generation of the first loop. One possible case is the operation of an interior segment of the network one end of which is very near the free surface as shown in Fig. 9(a). The intersection of the expanding loop with the surface creates a short and hence immobile dislocation segment AX and a long mobile segment BY as shown in Fig. 9(b). Continued expansion of BY should lead successively to configurations shown through (c) to (f). In (d) a short length of dipole is beginning to be formed near AX. In (e) the expanding loop has reached the surface recreating the source segment BY_1 and leaving a tripole at AX. However, the tripole will be unstable unless the number of jogs on the initial segment was very small. The two dislocations of opposite sign that lie on the same glide layer will usually annihilate leaving only segment AX. The source can then continue to operate like a single ended Frank-Read source as indicated in (f). The fact that most of the dislocation segments that were revealed by etching prior to loading did not undergo such multiplication is probably due to their being heavily jogged as a result of the high temperature anneal and the surface image forces. It is consistent with all the experimental observations to suppose that the first sources to send out more than one loop were longer than the average network length, that they lay nearly on {111} planes i.e. had a lower than average jog density and that they had one end near an external surface.

The presence in the early pileups of dislocations belonging to different glide planes cannot be explained by the above mechanism alone and it does not seem likely that it was due to cross-slip of some of the piled up dislocations. For the primary slip system of the present experiments the cross slip plane was always the one parallel to the surface of observation and consequently had a negligible resolved shear stress acting on it. Also dislocation configurations as in Figs. 4 and 5 are difficult to visualize as resulting from cross slip of the leading dislocations in a pileup. It is more likely that the dislocations in many early pileups have originated from different sources. The fact that such dislocation configurations were usually observed even in the first few isolated pileups indicate that some kind of cooperative source operation was involved in their formation. A likely possibility is that passage of a group of dislocations of like sign over a large area of the glide plane induces the emission of one or more loops from nearby parallel segments of the same Burgers vector which in the absence of the momentary stress concentration are below critical length for the applied stress. When there is a significant resolved shear stress acting on either of the other two coplanar Burgers vectors in the primary glide plane the above mechanism might also induce multiplication of these secondary dislocations. Dislocations of different Burgers vectors generated by this process are probably indicated by the two different kinds of pits seen in pileup A of Fig. 10 which shows a grain etched after applying a stress of 22 g/mm^2. Recent electron microscopy observations by Essmann et al.[14] on comparatively highly strained (4%) copper polycrystals have also indicated that slip often occurs in different directions on the same glide plane.

As already mentioned pileups were rarely found in the interior grains. It is not likely that this was due to the impossibility of examining interior grains without removing the applied stress. For surface grains there was generally only moderate reverse motion of dislocations on removal of the applied stress. Many pileups were stabilized by interactions with dislocations of a secondary system. If pileups had been present in the interior grains some of them almost certainly would have been stabilizied by such interactions and hence would have been detected even in the stress relaxed condition. It seems more likely that most sources in interior grains send out only a single dislocation loop during the pre-yield deformation for the reasons discussed in connection with Fig. 8. During their subsequent expansion over a large area of the glide plane such loops will interact with some segments of the grown-in dislocation network thereby reducing the average network length and causing a fairly uniform increase in dislocation density. The rapid increase in the number of moving dislocations that is associated with plastic yielding may then depend on the critical cross-over stress for the dipoles of widest spacing. When this stress is reached some sources should be able to operate as two individual single ended Frank-Read generators. They would then send out a number of loops on each of the two glide layers which are determined by the positions of the end points of the original network segment. Transmission electron microscopy observations by Essmann et al.[14] on the dislocation structure in the interior grains of copper polycrystals deformed to comparatively higher stress levels have shown accumulation of large numbers of dislocations of the same sign near grain boundaries. This implies that dislocation segments in the interior grains do eventually begin to generate many dislocations at higher stress levels and that these new dislocations are still able to travel all the way to the grain boundaries.

For a segment of given length the frictional stress required to start its bowing should increase linearly with increasing number of jogs. Therefore, the first segments to bow out would be those with lowest jog density. Most of these can send out only one loop because the dipole which is formed will have a small interplanar spacing and will be stable under the applied stress. As the number of jogs on the segment increases the stress to start bowing also increases; however the stress required to drive the two dislocations of the dipole past each other decreases since the spacing of the dipole will now be larger. There will be a critical number of jogs on a segment for which the stress to start its bowing and the stress for cross-over of the resulting dipole are equal. Only these segments will generally be able to send out a group of dislocation loops in regions away from a free surface. According to this mechanism of yielding it is necessary for the stress to be high enough to move heavily jogged dislocations before general multiplication can occur. Such a model is consistent with the many experiments which show that some dislocations move at stresses far below macroscopic yielding and also with the expectation that few, if any, segments in an annealed dislocation network will be completely free of jogs. It is also consistent with previous experimental results of Petroff and Washburn[5] which show that the stress needed to move heavily jogged dislocations in copper is of the order of the macroscopic yield stress.

IV. CONCLUSIONS

1. The dislocation loops generated by the first active sources both on primary and secondary slip planes traverse the entire cross section of the grains and pile up at the grain boundaries. In single crystals this first operation of sources might be missed entirely because all the dislocation loops would have escaped at surfaces.

2. At an applied stress level where grain boundary pile-ups containing 10 or more dislocations are numerous in surface grains none are found in interior grains.

3. The preferential multiplication of dislocations near an external surface takes place even when grown in dislocations are immobile where they intersect the surface and extreme precautions have been taken not to introduce any fresh dislocations or permit any stress concentrations.

4. Grown-in dislocations are relatively immobile where they intersect a {111} surface if extensive polishing is avoided following the last high temperature anneal. This is probably due to their tendency to lie at right angles to the {111} surface and therefore to have a high jog density.

5. All of the observations can be explained if it is assumed that all segments of the grown-in network contain jogs after a long anneal at high temperature.

6. Rapid multiplication of dislocations in interior grains probably becomesgeneral only after a stress is reached which permits motion of dislocation segments of high jog density. At lower stresses most potential sources should generate only a single loop due to formation of a stable dipole. This restriction should not exist for sources that lie very close to an external surface.

ACKNOWLEDGEMENT

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Table I. Analysis of the OFHC Copper

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

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FIGURE CAPTIONS

Fig. 1. Schematic drawing of the microcompression device. (a) as viewed from front (b) as viewed from side

Fig. 2. Dislocation motion and multiplication in a grain etched before and after application of a stress of 17 g/mm^2.

Fig. 3. Dislocation pileups in a grain etched before and after applying a stress of 20 g/mm^2. Note the absence of any large flat bottomed pits along the slip plane traces of the two pileups A and B.

Fig. 4. A typical early dislocation pileup in a grain etched under a stress of 16 g/mm^2.

Fig. 5. A typical early dislocation pileup in a grain etched before and under a stress of 22 g/mm^2.

Fig. 6. Decoration of secondary slip plane traces (at A and B) in a grain etched after applying a stress of 25 g/mm^2.

Fig. 7. (a). A grain subjected to a stress of 17 g/mm^2 while immersed in the electropolishing solution and etched before drying.
(b). Same grain etched after drying; there is no evidence of dislocation motion or multiplication.

Fig. 8. Conservative motion of a lightly jogged dislocation segment to form a single dislocation loop and a dipole.

Fig. 9. Mechanism for the generation of many dislocation loops from an interior source having one of its ends close to the surface.

Fig.10. Pileup (A) containing dislocations of different Burgers vectors in a grain etched after applying a stress of 22 g/mm^2.

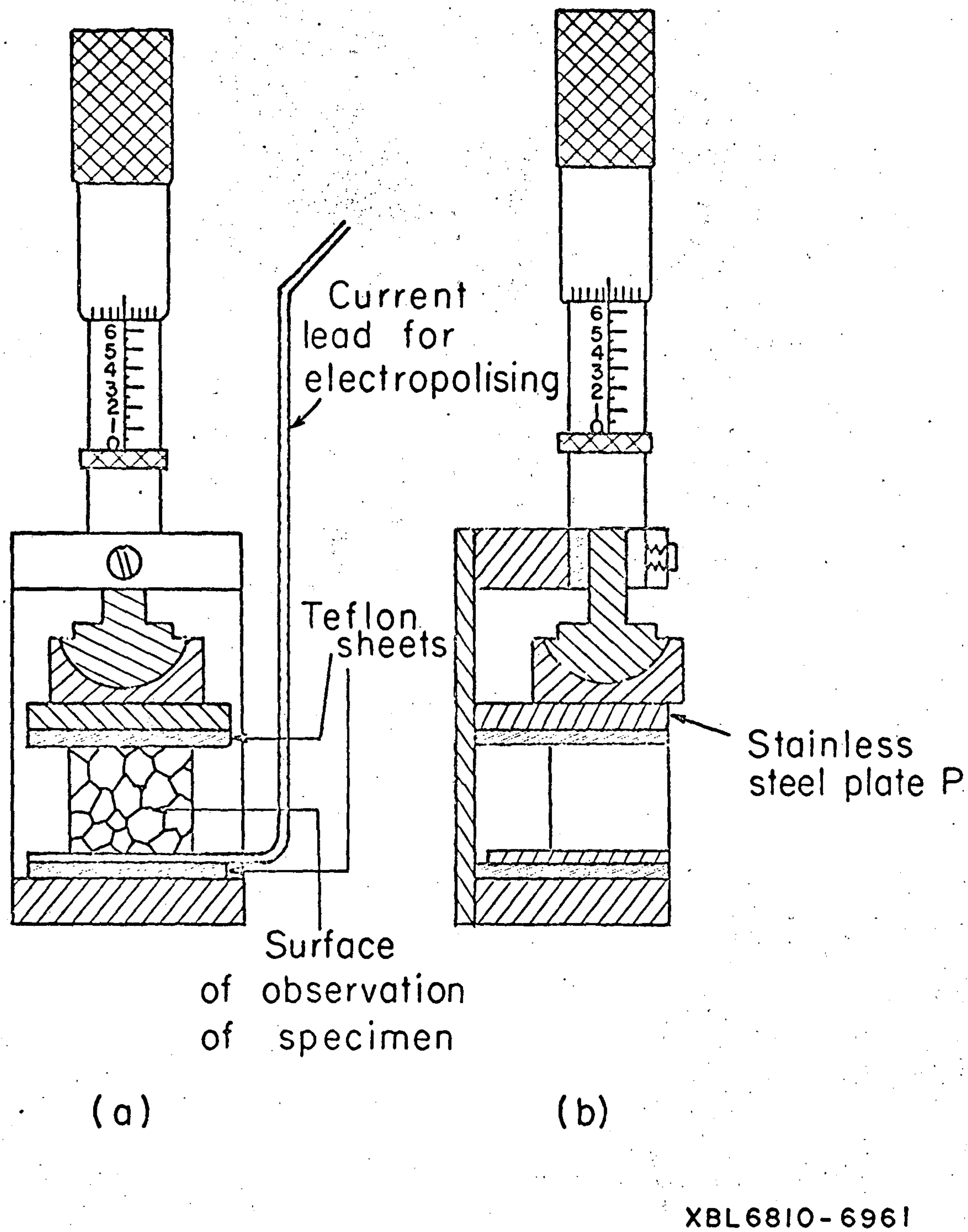
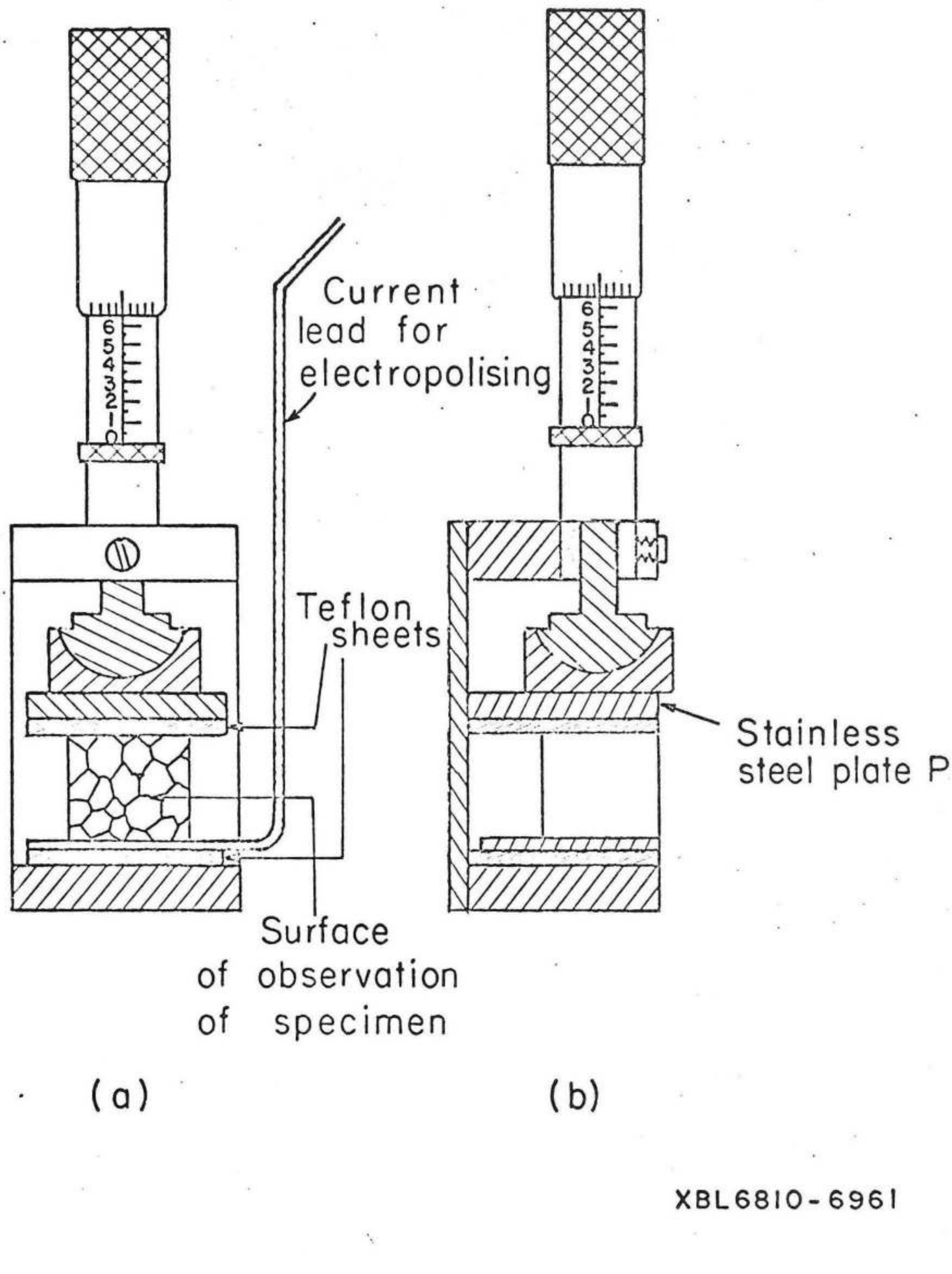

Current
lead for
electropolising
Teflon
sheets
Stainless
steel plate P
Surface
of observation
of specimen
(a)
(b)
XBL6810-6961

Fig. 1


Current
lead for
electropolising
Teflon
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Stainless
steel plate P
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of observation
of specimen
(a)
(b)
XBL6810-6961

Fig. 1

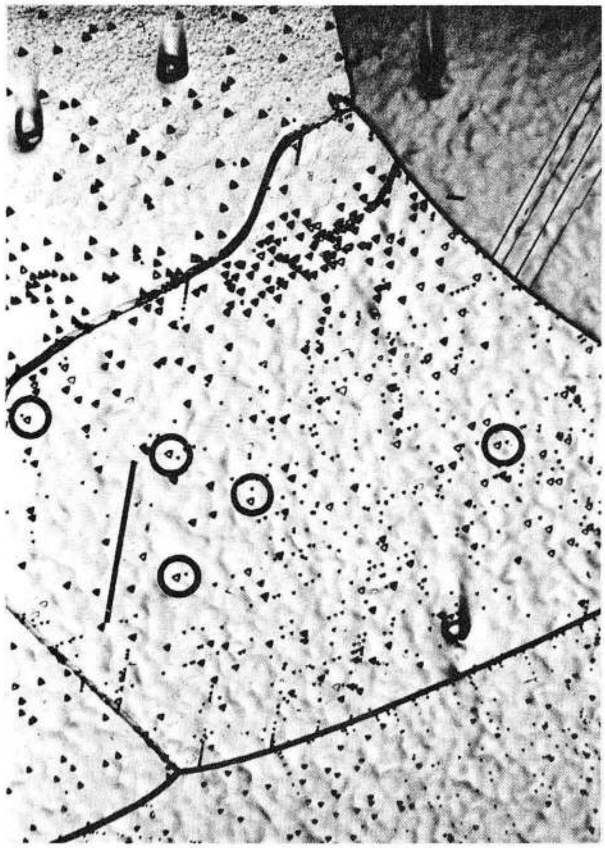

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Figure 2

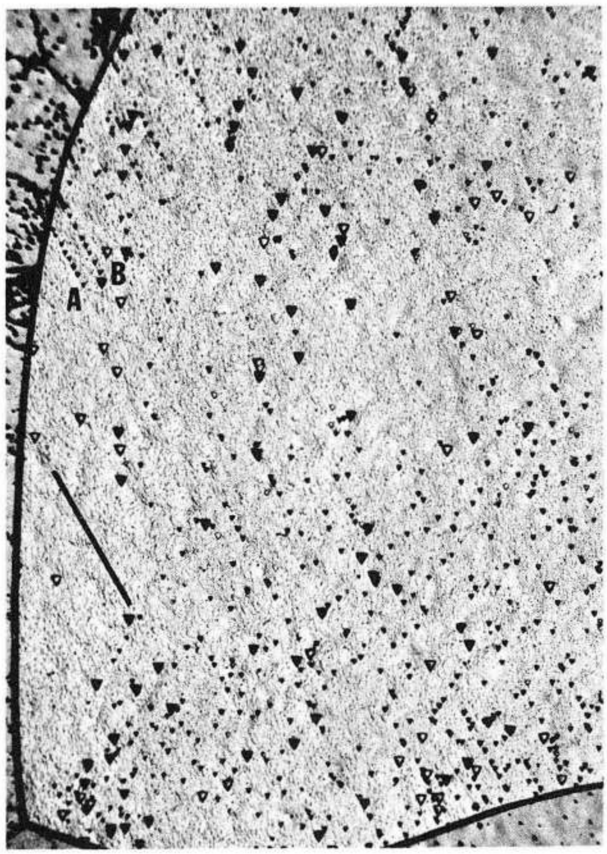

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Figure 3

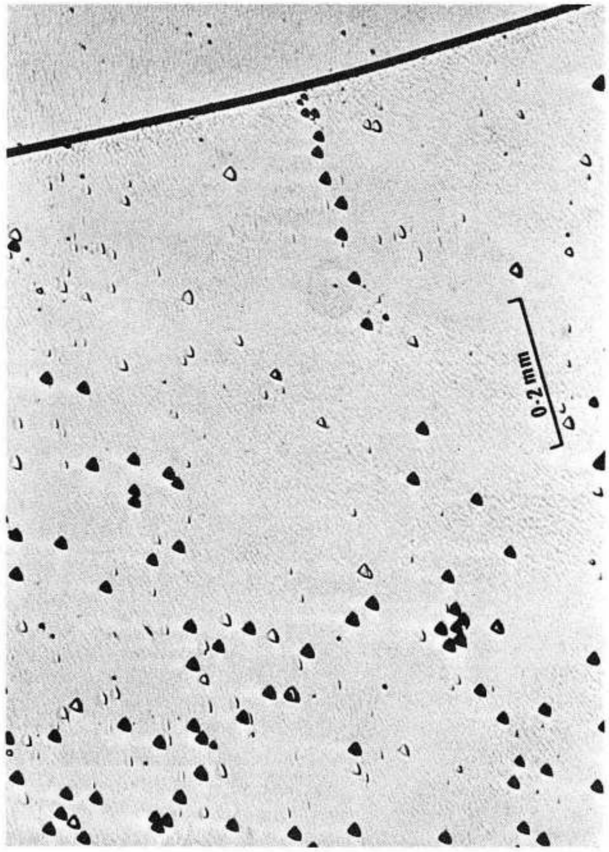

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Figure 4

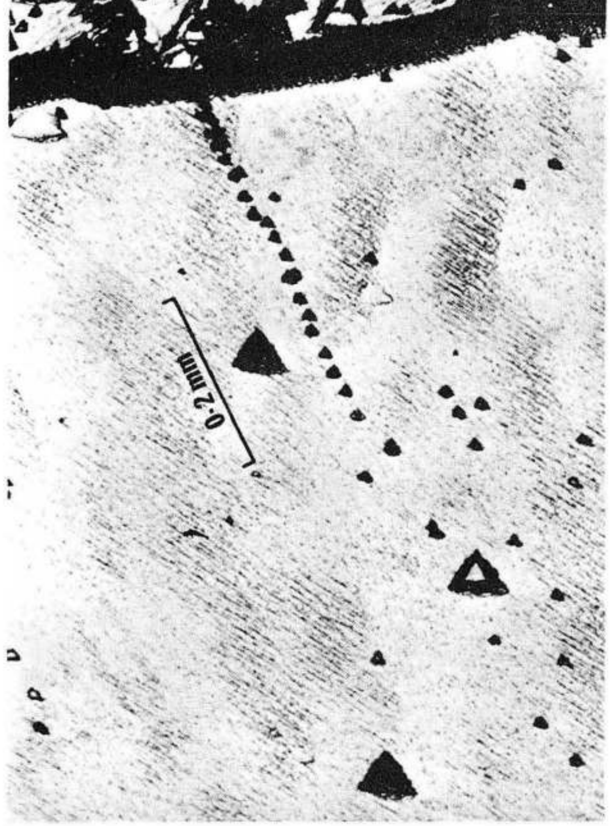

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Figure 5

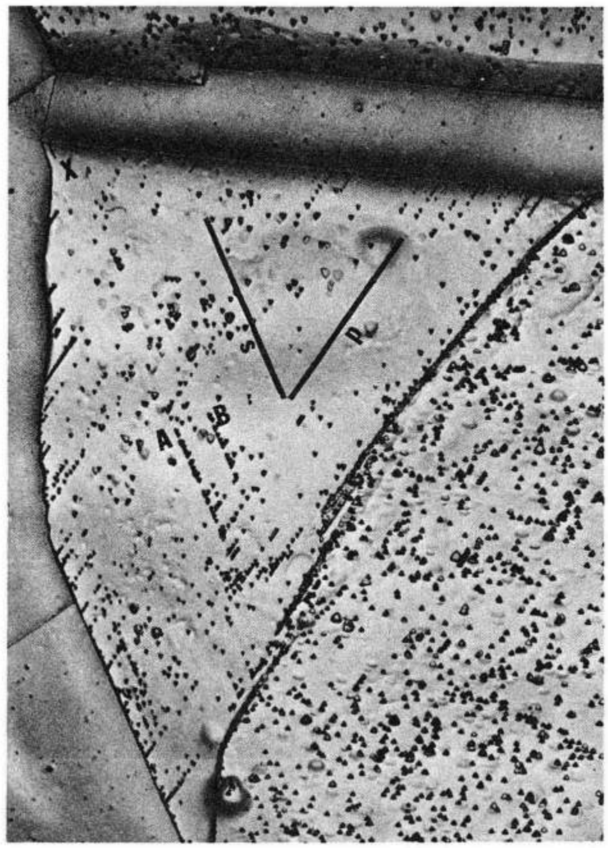

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Figure 6

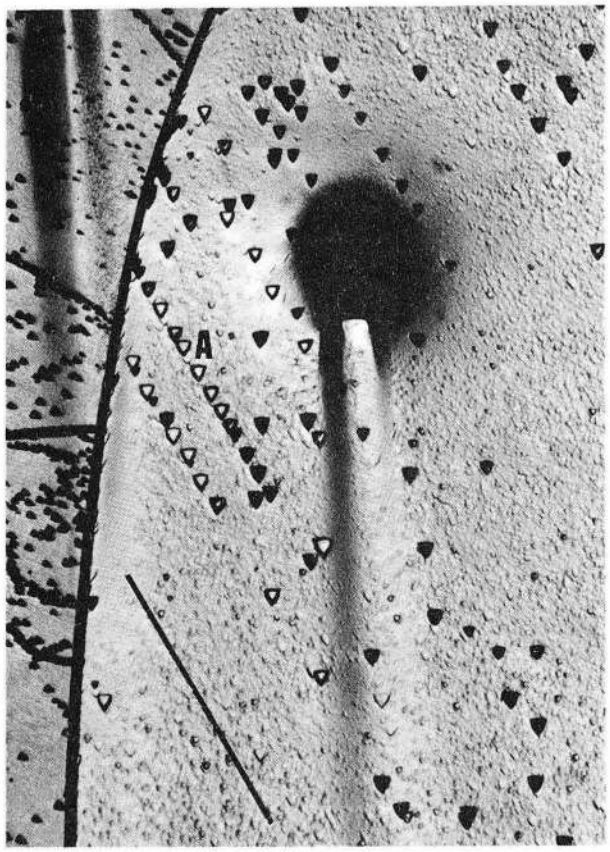

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Figure 7

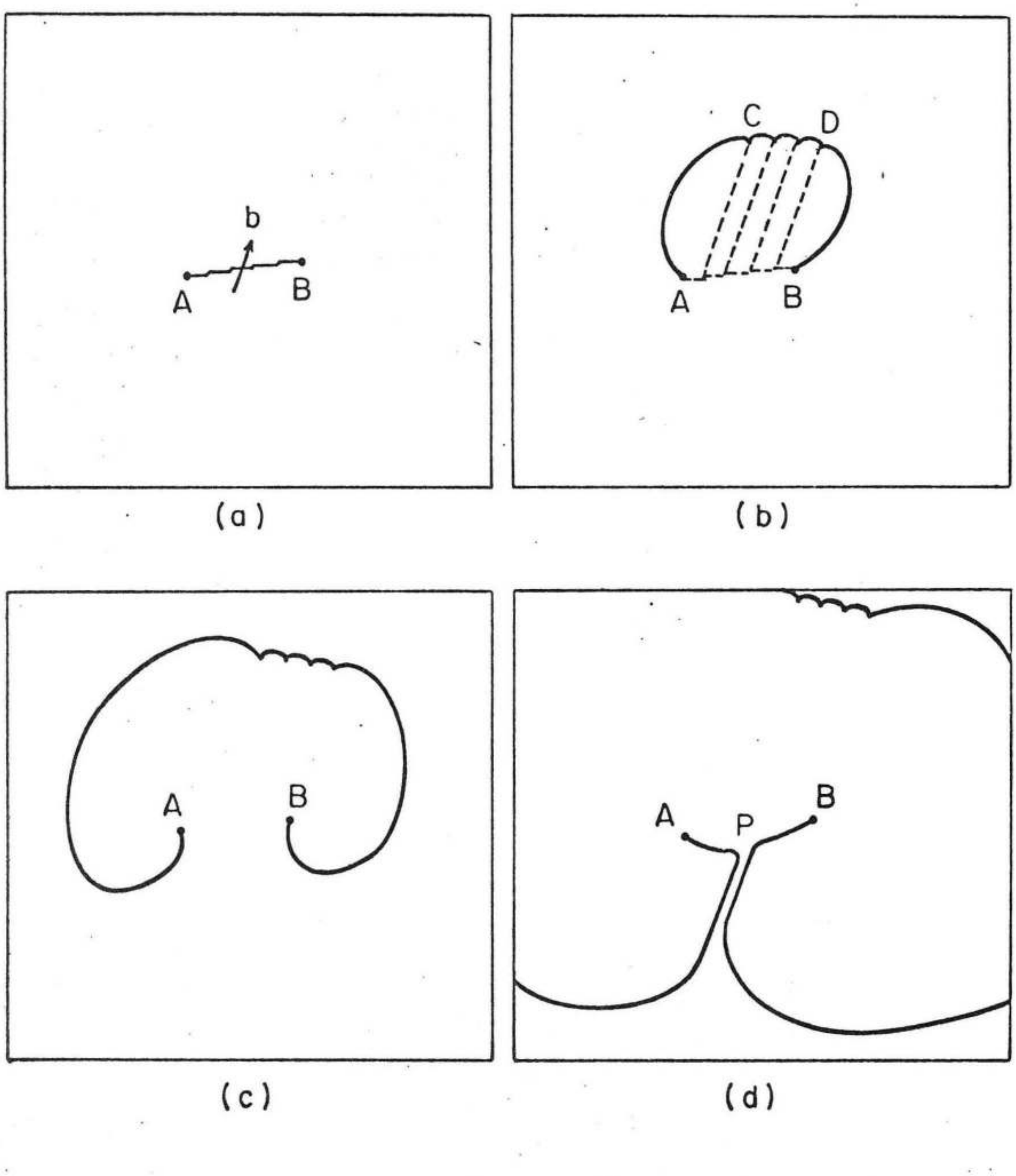

Fig. 8

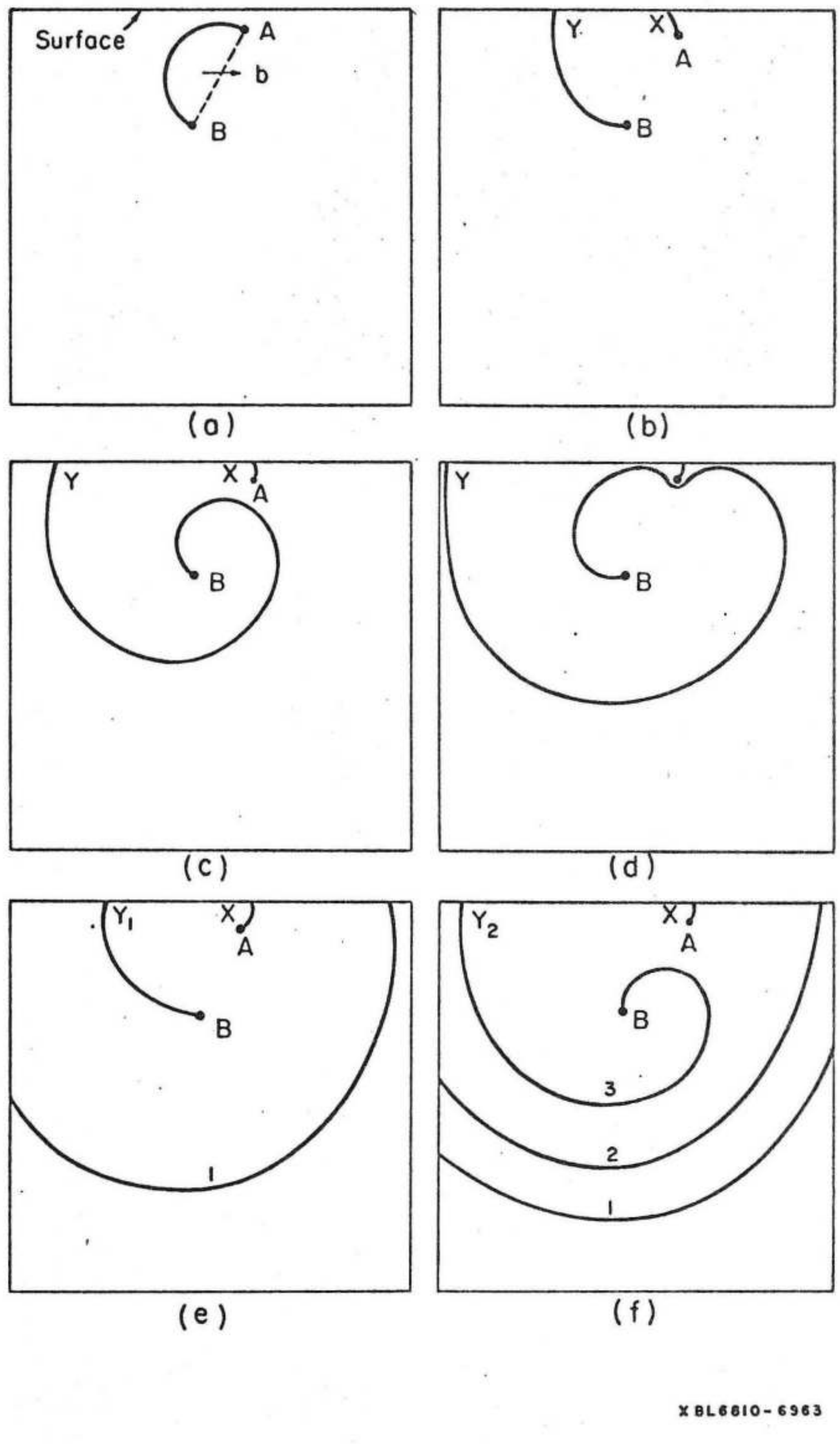
Surface
A
b
B
(a)
Y
X
A
B
(b)
Y
X
A
B
(c)
Y
B
(d)
Y_1
X
A
B
1
(e)
Y_2
X
A
B
3
2
1
(f)
XBL6810-6963

Fig. 9

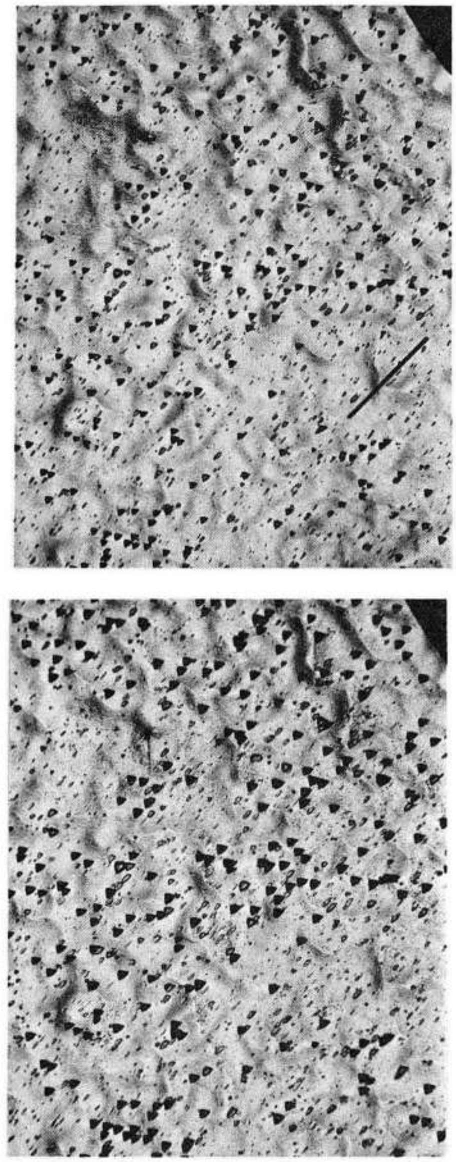

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Figure 10

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