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Authors
Vellaikal, Gopinathan
Washburn, Jack.

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

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 $2g/mm^2$. 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 appli-

cation 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 dis-

locations 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 speci-

mens. 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 recry-stallized specimen, will lie exactly on {111} planes; most of the dislo-cation 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 treat-

ment. 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 dislo-

cation motion or multiplication induced as a result of the drying opera-

tion 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 ex-

perimental 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 observa-
tions 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 dislo-
cations 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. There-

fore, 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 expec-

tation that few, if any, segments in an annealed dislocation network will

be completely free of jogs. It is also consistent with previous experi-

mental 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 macro-

scopic 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 becomes general 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

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

References and Footnotes

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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 sheets

Surface of observation of specimen

Stainless steel plate P

(a)   (b)
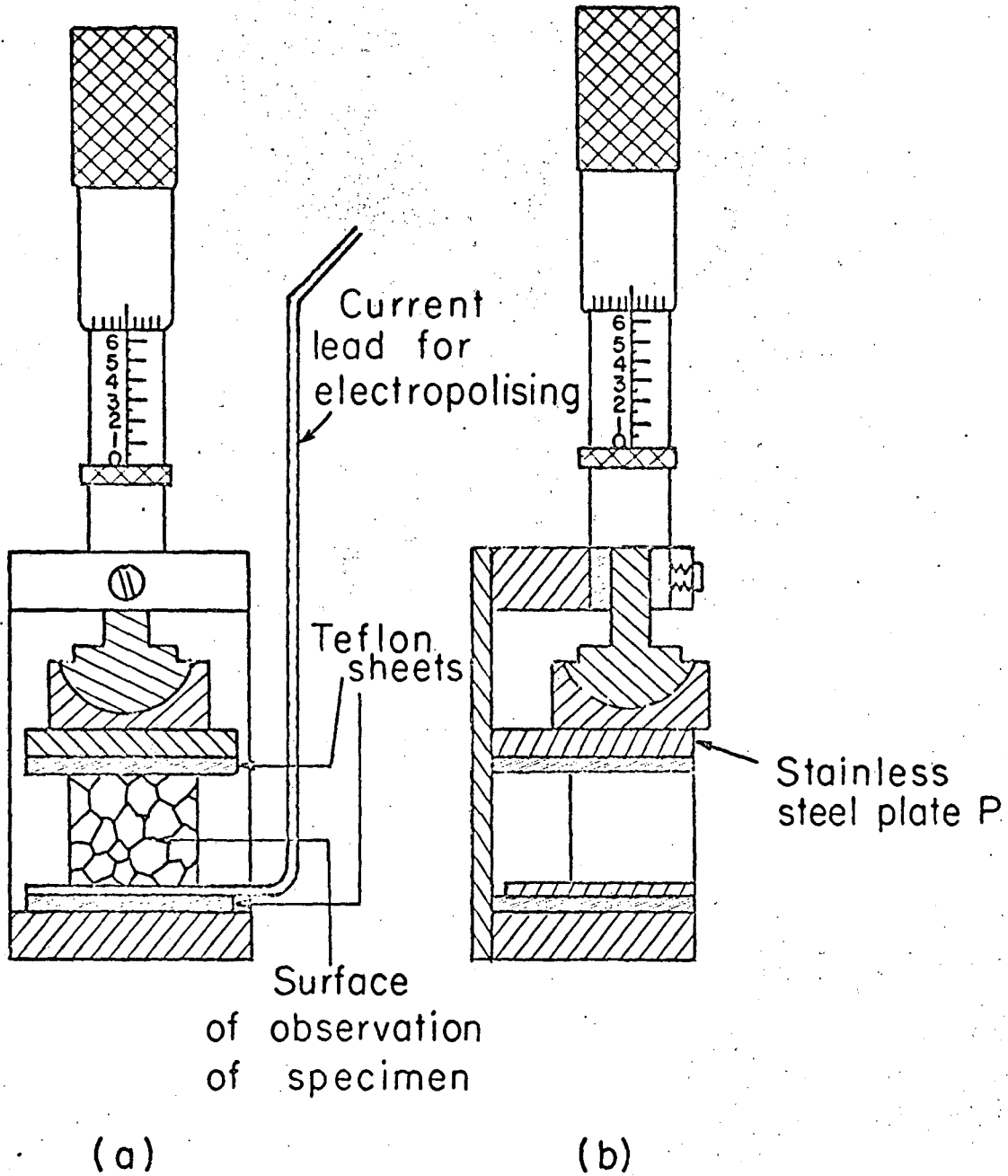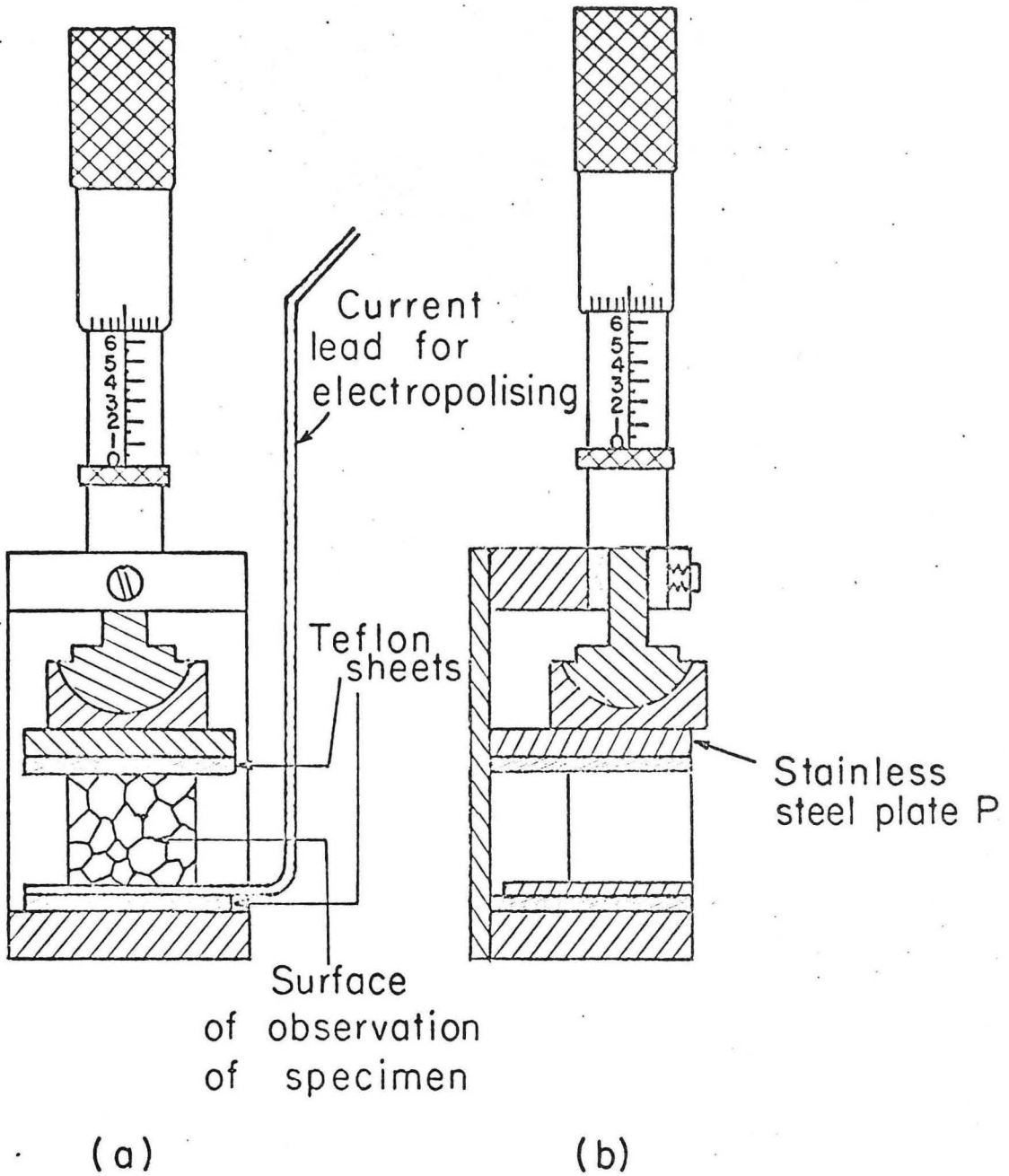
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Fig. 1
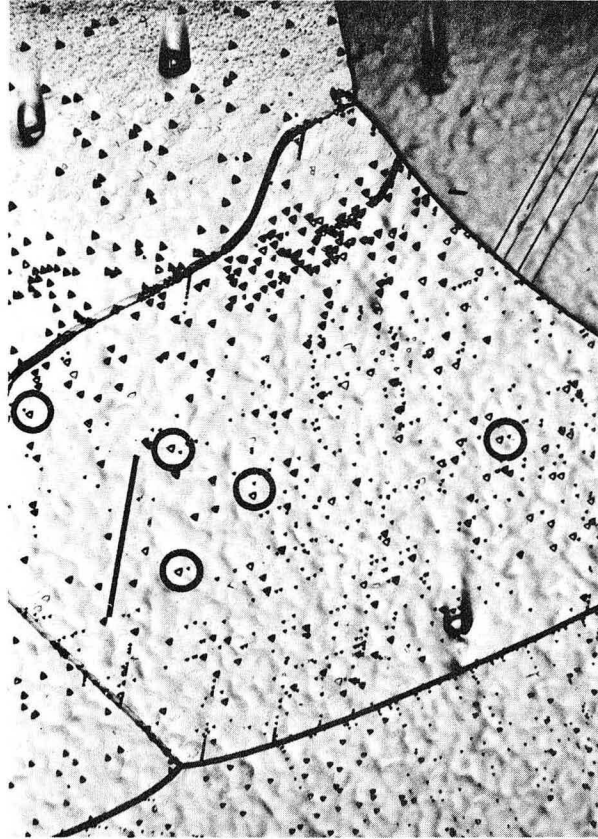
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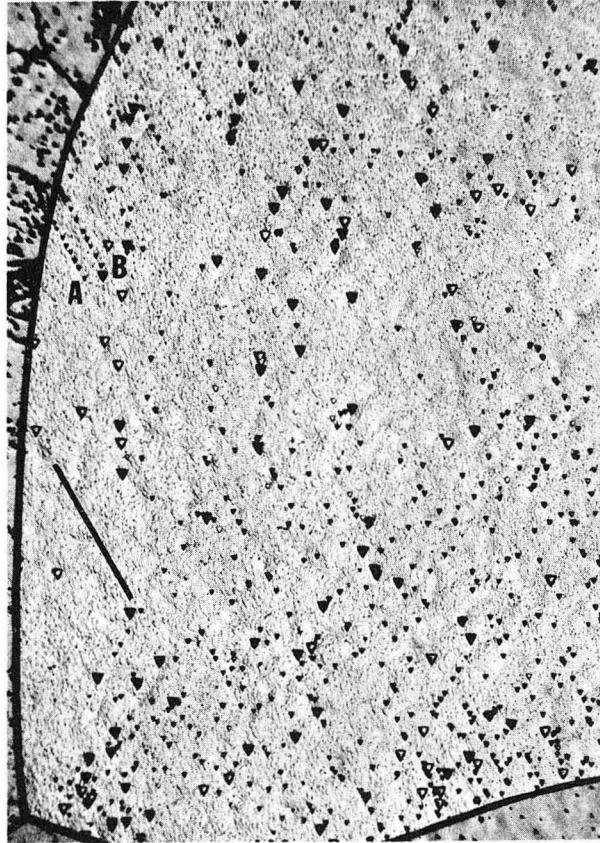
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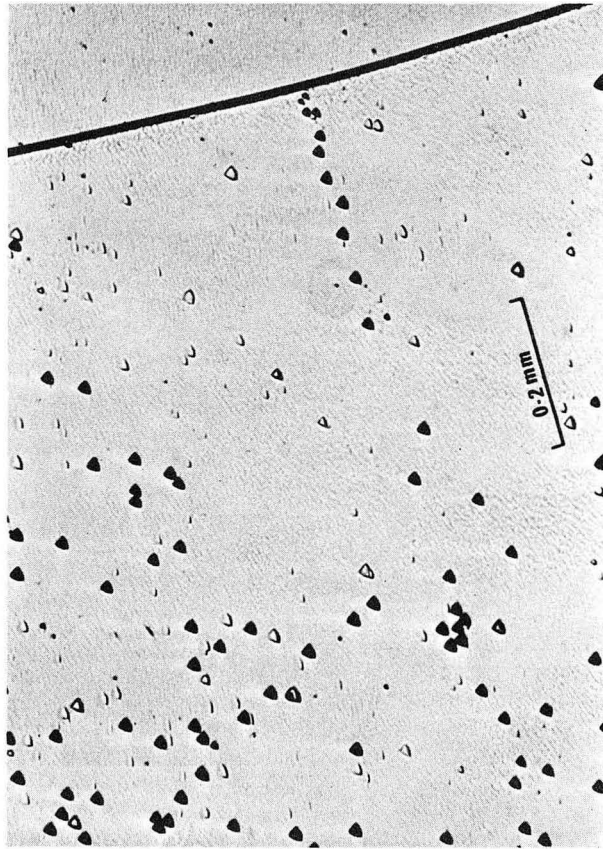
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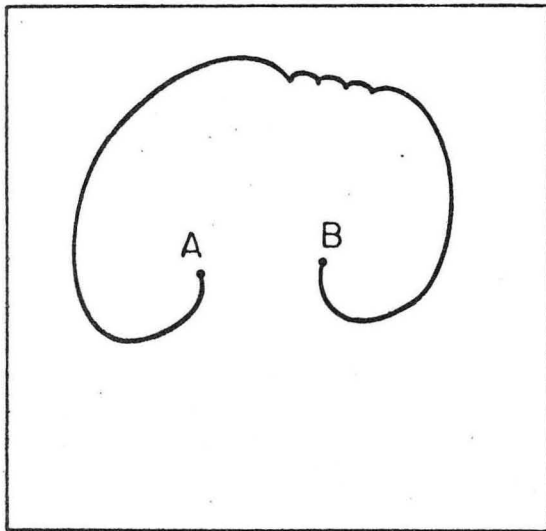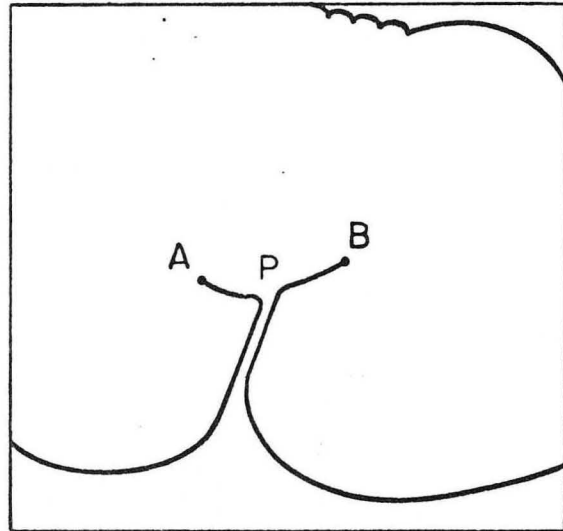Figure 4

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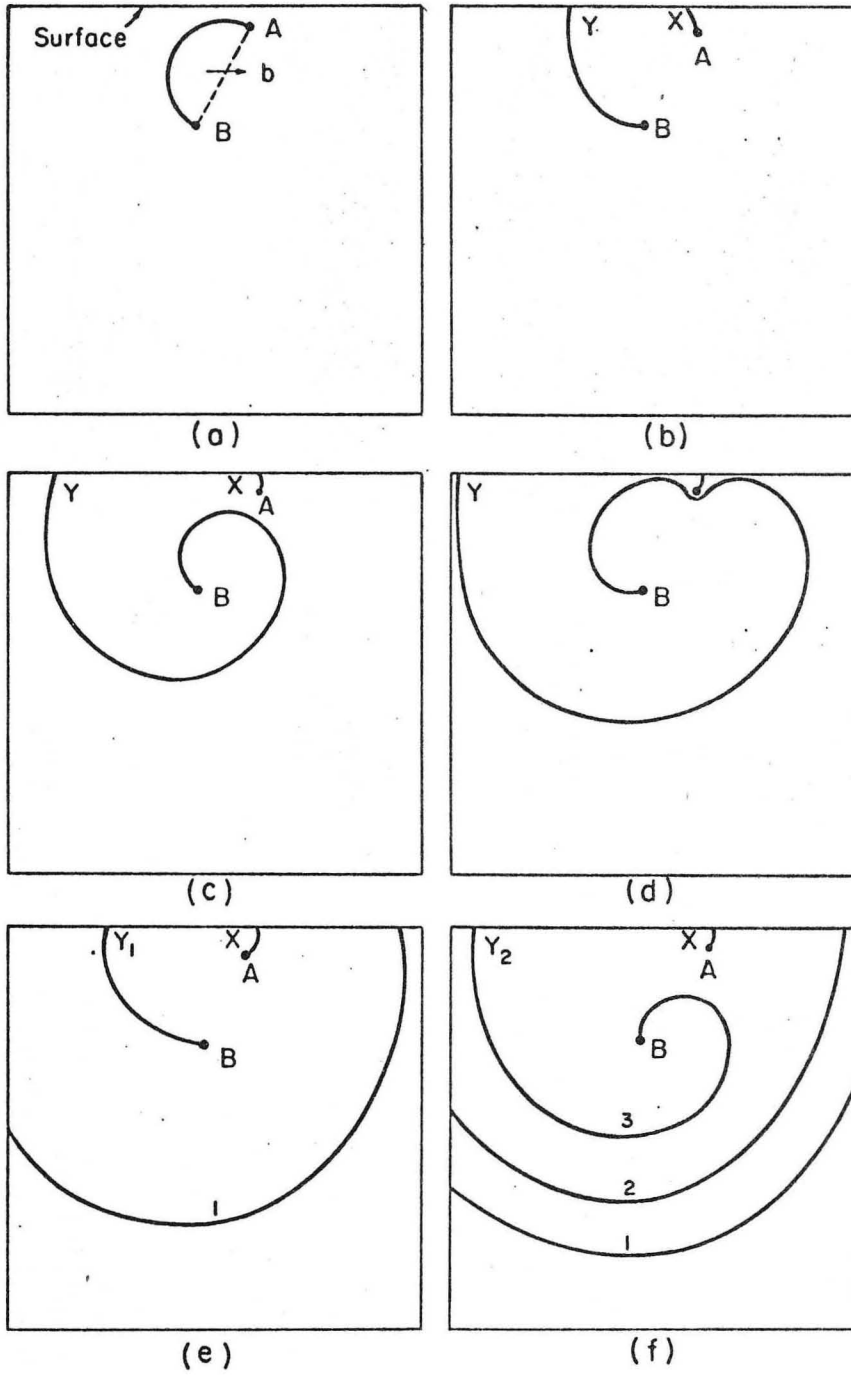
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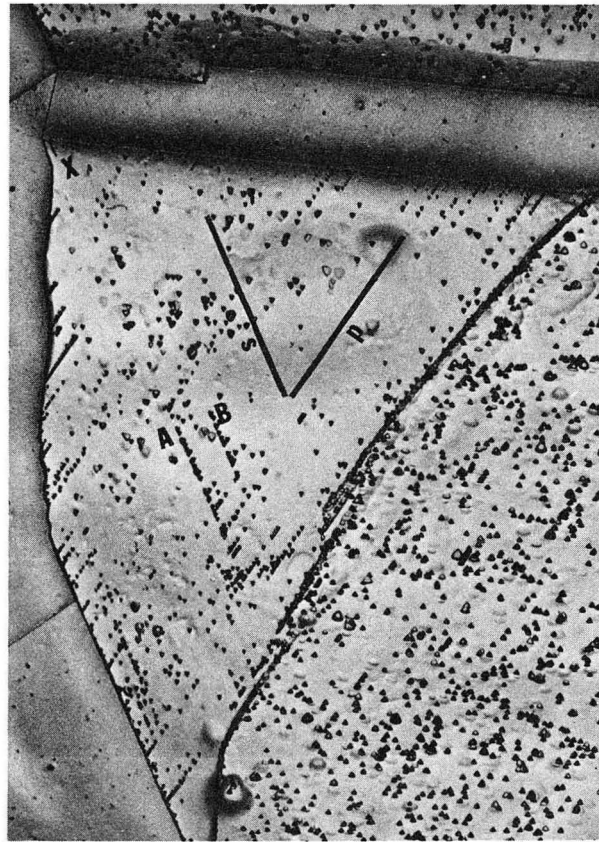
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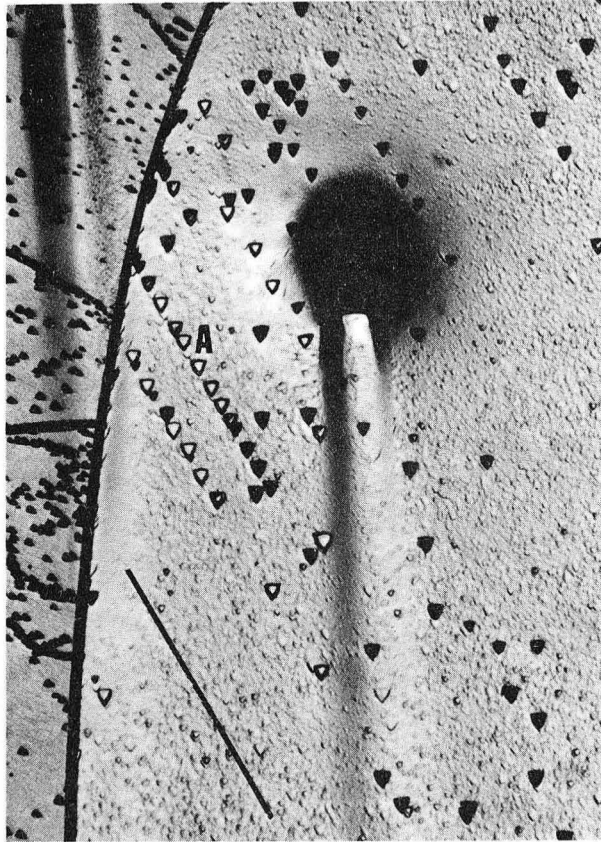
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Figure 7
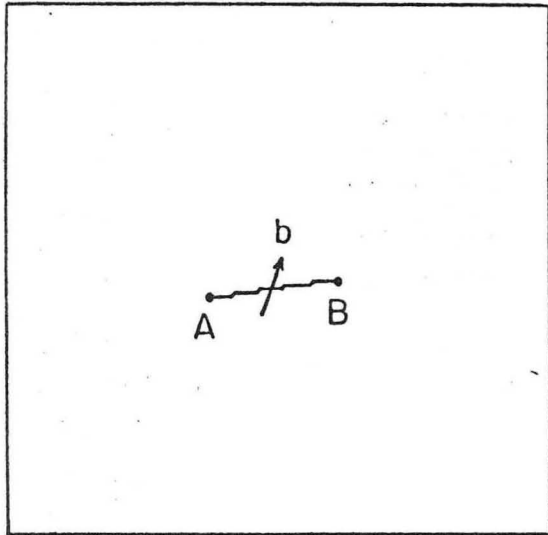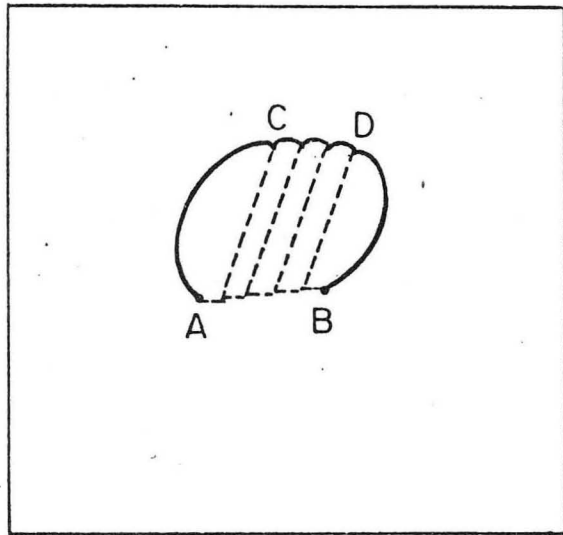
(a)　　　　　　　　　　　(b)

(c)　　　　　　　　　　　(d)

XBL6810-6962

Fig. 8

Fig. 9

XBL6810-6963

XBB 6811-7277

Figure 10