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Authors

Strudel, J.L.
Washbum, J.

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University of California

Ernest O. Lawrence Radiation Laboratory

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J. L. Strudel and J. Washburn

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

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ABSTRACT

Observations have been made by transmission electron microscopy on the interactions between faulted dislocation loops and moving dislocations in high purity aluminum. Thin single crystals were quenched from 650°C and 540°C into water at 0°C. In both cases most of the loops inclosed a stacking fault. Their average diameter was 250 Å in the first case, 900 Å in the second case. Surface orientation for all foils was (110) which made possible easy identification of Burgers vectors for both loops and moving dislocations. When a moving dislocation came close to or in contact with a loop the stacking fault was always completely destroyed and part or all of the loop was brought to the surface of the foil by prismatic glide.

1. INTRODUCTION

Clustering of vacancies in quenched or irradiated fcc metals leads to the formation of spherical voids, stacking fault tetrahedra, or dislocation loops. These defects can cause moderate increases in the yield stress which are explained in order of magnitude by the theoretical models of Seeger (1958) and Friedel (1963). In some cases there is also a concomitant decrease in the strain hardening rate during the initial stages of plastic deformation (Maddin and Cottrell, 1955). This might be expected because it is known that small amounts of plastic deformation after quenching and aging of pure aluminum can completely remove the dislocation loops (Vandervoort and Washburn, 1960).

Direct observations of interactions between moving dislocations and loops in aluminum and with stacking fault tetrahedra in gold have been made by Hirsch and Silcox (1958). There are many different kinds of interactions to be expected depending on the relative Burgers vectors and spatial relationships between the loop and the moving dislocation. Some of these have been considered for the fcc structure by Saada and Washburn (1963): Contact interactions between moving dislocations and large imperfect loops of Burgers vector $\frac{a}{3}\{111\}$ are of two different types:

(case 1.) When the loop does not lie on either of the two possible glide planes the moving dislocation can dissociate in the stacking fault of the loop into a Frank sessile dislocation and a Shockley partial. The stacking fault is swept away by the glide of the Shockley partial and the dislocation line acquires a segment that does not lie on the original glide surface. The following reactions take place:

Dissociation of the moving dislocation in the stacking fault of the loop:

$$\frac{1}{2}[1\bar{1}0] \rightarrow \frac{1}{3}[1\bar{1}1] + \frac{1}{6}[1\bar{1}\bar{2}]$$

Recombination of the Shockley partial with the Frank sessile at the perimeter of the loop:

$$\frac{1}{6}[1\bar{1}\bar{2}] + \frac{1}{3}[1\bar{1}1] \rightarrow \frac{1}{2}[1\bar{1}0] .$$

(case 2.) When the loop lies on one of the two possible glide planes of the moving dislocation, the latter dissociates in the stacking fault of the loop into two Schockley partials which remove the stacking fault each on its side. The dislocation reactions are:

Dissociation of the moving dislocation in the stacking fault of the loop:

$$\frac{1}{2}[1\bar{1}0] \rightarrow \frac{1}{6}[1\bar{2}\bar{1}] + \frac{1}{6}[2\bar{1}1]$$

Recombination at the perimeter:

$$\frac{1}{3}[11\bar{1}] + \frac{1}{6}[1\bar{2}\bar{1}] \rightarrow \frac{1}{2}[10\bar{1}]$$

and

$$-\frac{1}{3}[11\bar{1}] + \frac{1}{6}[2\bar{1}1] \rightarrow \frac{1}{2}[0\bar{1}1] .$$

The loop becomes two dislocation segments of different Burgers vector that may or may not lie in the glide plane and are connected to the moving dislocation at two nodes. This configuration should act as a strong anchor point on the moving dislocation.

The purpose of this paper is to give some direct experimental evidence for the above interactions and to consider further the conditions under which a Frank sessile loop will be eliminated by a moving dislocation.

2. EXPERIMENTAL PROCEDURE

Frank sessile loops were obtained in 99.999% aluminum single crystals by quenching from 650°C or 540°C followed by aging at 20°C. The former treatment produced a high density of small loops (250Å average diameter) and the later gave large loops (800Å to 1200Å diameter).

Use of single crystals near (110) orientation facilitated the determination of the Burgers vector of loops and moving dislocations. In this orientation two of the four {111} planes, $(\bar{1}11)$ and $(1\bar{1}1)$, lie approximately at right angles to the foil surface so that loops lying on them are seen edge-on with the Burgers vector at right angles to the projection of the loop. Dislocations that move to produce slip traces always lie on one of the other two glide planes, (111) or $(11\bar{1})$, and have one of the four Burgers vectors $\frac{1}{2}[\bar{1}01]$, $\frac{1}{2}[01\bar{1}]$, $\frac{1}{2}[101]$ or $\frac{1}{2}[011]$. The diffracting planes for a (110) foil surface are $(\bar{1}11)$, $(1\bar{1}1)$ and (001). None of the four sets of Frank sessile loops have Burgers vectors that lie in any of these planes. Therefore they are always in contrast regardless of which plane is in diffracting position.

Single crystal strips were grown under vacuum from 99.999% pure aluminum in a graphite mold packed with graphite powder. Seed crystals were welded to the polycrystalline blanks to produce the desired (110) surface orientation.

Mechanical polishing was necessary to provide a suitable surface for uniform chemical polishing. The following solution was used at 90°C to thin the crystals to 0.25 mm.

Phosphoric acid (86%)	850 cm^3
Sulfuric acid (96%)	130 cm^3
Nitric acid (70%)	20 cm^3

The strip was then annealed at 640°C in air for 24 hours and furnace cooled. Rapid quenching was achieved by pulling the specimen into a 1-meter-deep water quenching bath from a special furnace having its hot zone only about one inch from the surface of the water.

After an aging period of 1 to 20 hours at room temperature, the crystal was electrochemically polished at 4°C in a 20-80 perchloric acid-ethyl alcohol solution to which 5 cm^3/100 cm^3 butylcellosolve was added.

Flakes were obtained which were washed thoroughly with 200 proof alcohol before drying.

Specimens were observed by transmission electron microscopy in a Siemens Elmiskop I operated at 100 kV. Use of the Stereo-tilting stage enabled various diffraction contrast conditions to be obtained.

3. RESULTS AND DISCUSSION

3.1 General features of slip traces

Most of the dislocation lines that were seen moving in the thin foils had a relatively high velocity ($> 5 \times 10^{-3}$ cm/sec). Hence, details of the interactions between loops and moving dislocations were rarely observed directly. However, the slip traces left behind them exhibited features that resulted from these interactions. Most of the interpretations proposed in this paper have been deduced from this evidence.

A variety of slip trace contrast effects, which depend on the diffraction conditions, are observed when dislocations in thin foils move during observation. The entire projection of the slip plane usually becomes darker (Fig. 1) or lighter (Fig. 7) than the background, the greatest contrast being near the intersections of the glide surface with the top and bottom surfaces of the foil. The edges of a slip trace mark the paths of the dislocation ends along opposite surfaces of the foil. The persistence of slip trace contrast varies greatly from metal to metal; traces last only a few minutes in aluminum. It has been suggested (Hirsch, 1959; Whelan, 1959; Howie and Whelan, 1962) that these contrast effects may be due to the presence of a surface layer of alumina or other compounds left after the electropolishing treatment used to prepare the thin foil. This layer may prevent the slip step caused by the moving dislocation from reaching as a whole and immediately the surface of the foil. What is effectively a long dislocation must therefore be left in or just under the oxide layer.

Slip traces are also left by dislocations that intersect only one surface of the foil. For example, the imperfect loop P (Fig. 2) has been converted into a perfect one which has subsequently moved along its glide cylinder to one surface of the foil leaving the slip trace at P' (Fig. 2). See Fig. 2.

When moving dislocations react with loops, the slip trace of the dislocation generally shows indentations like E, F, J (Fig. 1).

3.2 Interpretation of the observed interactions

Where dislocations that have moved are visible at one end of a slip trace, the most common orientation is near pure screw. Somewhat less frequently the dislocation takes the orientation of the close packed <110> direction which lies in its glide plane at 60° from its Burgers vector. For example, the moving dislocation in Fig. 5 has Burgers vector BA; both its primary and cross-slip planes can be deduced from Fig. 5. Segment NM lies approximately parallel to its Burgers vector and segment ML is at 60° to it. These two orientations can be taken as limits for our models.

Thompson's notation (1955) will be used to represent the Burgers vectors and glide planes (Fig. 3). In the schematic drawings AD will be taken as the <110> direction which lies approximately at right angle to the plane of the foil. To describe the different kinds of interactions BD and a are assumed as the Burgers vector and glide plane of the moving dislocation. The dislocation is assumed to move on its glide plane from the left to the right.

3.3 Interactions in which the loop does not lie on either glide plane of the moving dislocation

In this case, the stacking fault is removed by a single Shockley partial that has to sweep accross the whole area of the loop and reach the edge where it combines with the Frank sessile dislocation. Experimental observations suggest that this process can take place in three different ways.

(a) Formation of one turn of a helix on a screw dislocation:

Different stages of this process can be seen on Fig. 4a, b, c, d, where the dislocation line L, close to screw orientation, was seen moving slowly and coming into contact with the perfect loop P which must have had the same Burgers vector as the moving dislocation. Later it came in contact with imperfect loop I (Fig. 4c). One turn of a helix was formed each time the dislocation line touched a loop - the final shape of the dislocation line reveals two turns of a helix (Fig. 4d). The same interaction is shown by Fig. 5a, b and described schematically in Fig. 5c.

If the moving dislocation comes into contact with the half of the loop for which the two Burgers vectors are at an acute angle (like $\vec{DB}$ and $\vec{D\delta}$ on Fig. 5c), the Shockley partial -- can bow out and sweep the stacking fault as shown in (111) projection in Fig. 5c. Subsequently the line tension acting on the dislocation line tends to extend the helix which becomes elongated as shown in Fig. 5a on segment MN. If the applied stress continues to move the dislocation the helix will not remain within the foil but will be pushed, along its glide cylinder toward the surfaces. As parts of the helix reach the foil surfaces both indentations and changes in the glide plane of the moving dislocation can be produced as at C'C'' (Fig. 5b).

This kind of interaction seems to be the most common one in aluminum thin foils. The following cases can be considered as modifications.

(b) Interaction which destroys only part of the loop:

When the angle between the Burgers vector $\vec{BD}$ of the moving dislocation and the Burgers vector $\vec{D\delta}$ of the one half of the loop that is involved in the reaction is obtuse, the Shockley partial must glide across the loop as shown on Fig. 6. This geometry is less favorable to the formation of an elongated turn of a helix and regardless of its orientation the dislocation - edge or screw - will tend to cut through the loop leaving a smaller loop behind. The other part of the loop becomes a part of one turn of a helix on the dislocation and leaves an indentation on the slip trace where it reaches the surface.

This kind of interaction occurred with loop F (Fig. 7a) which gave rise to indentation I in the slip trace (Fig. 7b) and a smaller loop P was left behind. Similarly loop G (Fig. 7b) gave rise to indentation J in the slip trace (Fig. 7c), a smaller loop Q was left behind and had partly reached one of the surfaces of the foil when the picture was taken. Note that residual loop, P, visible on Fig. 7b has already vanished on Fig. 7c a few seconds later. This suggests that smaller loops may often be left after an interaction but since they are always perfect loops they usually glide to one of the foil surfaces. Residus were hardly ever observed for interactions with small loops (diameter < 300 Å). N and Q (Fig. 8a) simply gave rise to indentations N' and Q' (Fig. 8b).

(c) Interaction without direct contact:

Figure 5b at Q' and Fig. 8b at E' show a peculiar kind of contrast which seems to be a superposition of a regular slip trace and a trace like that shown in Fig. 2b where a perfect loop has reached the surface by prismatic glide. This suggests that sometimes an imperfect loop is converted to a perfect one and glides to the surface without actually coming into contact with the moving dislocation.

3.4 Interactions in which the plane of the loop is parallel to the Burgers vector of the moving dislocation

In this case there is no long range interaction between the loop and moving dislocation because their Burgers vectors are at right angles. Two different situations can be distinguished.

(a) The loop does not lie on the glide plane of the dislocation:

When a dislocation of Burgers vector $\vec{BD}$ intersects a loop lying on plane C, it will split into two partials:

$$\vec{BD} \rightarrow \vec{B\gamma} + \vec{\gamma D}$$

which will combine separately with $\pm\ \vec{\gamma C}$ of the loop:

$$\vec{B\gamma} + \vec{\gamma C} \rightarrow \vec{BC}$$

on one side and

$$\vec{C\gamma} + \vec{\gamma D} \rightarrow \vec{CD}$$

on the other side. The reaction results in formation of nodes N_1 and N_2 as shown by Fig. 9a. A dislocation that has been held up by this type of interaction can be seen at G in Fig. 1.

If the dislocation line is then pulled away from the loop, the result of the interaction is to change the loop from a Frank sessile to one of the perfect prismatic type. The nodes N_1 and N_2 and the segment joining them will tend to glide toward the nearest edge of the loop. (Fig. 9c) The final Burgers vector of the loop will be either BC or CD. Fast moving dislocations usually left no trace of this kind of interaction. They simply passed around the loop leaving it as a perfect loop but not causing it to glide to the surface. This interaction is probably illustrated in Fig. 7c and d where imperfect loop S becomes perfect loop S' when the dislocation causing slip trace T intersected it.

(b) The loop lies on a plane parallel to the glide plane of the dislocation:

In this case there is little probability of direct contact between loop and moving dislocation because they would have to lie on the same atom layer. However, if the dislocation passes close to the loop, its stress field can cause nucleation of a Shockley partial in the loop (Yoshida, Kiritani and Shimomura, 1963) which has the same result as direct contact. This interaction may have taken place in Fig. 8 where the dislocation L on the left of Fig. 8a passed by loop M which is seen in good contrast. The slip trace due to motion of dislocation L to the right is seen in Fig. 8b. The loop which is still present at M' now has a different Burgers vector and is almost completely out of diffraction contrast.

4. CONCLUSIONS

Large Frank loops interact with moving dislocations in two different ways. Both lead to a complete removal of the stacking fault and change in the Burgers vector of the loop from $\frac{a}{3}\langle 111\rangle$ to $\frac{a}{2}\langle 110\rangle$.

When the loop lies on one of the glide planes of the moving dislocation, the latter reacts with the loop so as to leave a perfect loop with a Burgers vector that is not parallel to that of the moving dislocation. When an imperfect loop does not lie on either glide plane of the moving dislocation, the latter combines with the loop which becomes a helical segment on the moving dislocation.

These observations show that whenever a moving dislocation intersects or nearly intersects a large imperfect loop in aluminum the stacking fault is destroyed, the loop being converted to a perfect loop. If the Burgers vector of the resulting loop is the same as that of the moving dislocation then the loop often becomes a helical segment of the moving dislocation. In a thin foil perfect loops and helical dislocation segments can be easily eliminated by prismatic glide to the foil surface. The results suggest that in a bulk crystal they would be swept into the subgrain boundaries or into the regions of highest dislocation density where they would become a part of the three dimensional dislocation tangle or would be left as perfect loops.

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Figure Captions

Fig. 1. Typical slip traces in a 5000Å thin foil from a (110) single crystal quenched from 650°C into 0°C water. Slip traces exhibit indentations due to interactions with loops at E, F, and J.

Fig. 2. a and b. Prismatic glide of loop P towards one surface of the foil after its Burgers vector has been converted from $\frac{1}{3}\langle 111\rangle$ to $\frac{1}{2}\langle 110\rangle$.

c. Sketch showing prismatic glide of a perfect loop.

Fig. 3. Tetrahedron of {111} planes showing notation for Burgers vectors and planes.

Fig. 4. a, b, c, d. Successive stages of interactions between dislocation line L and loops $\mathring{I}$ and P.

Fig. 5. a and b. Interaction between a moving dislocation LMN and imperfect loops which do not lie on either glide plane of the moving dislocation. See text for explanation.

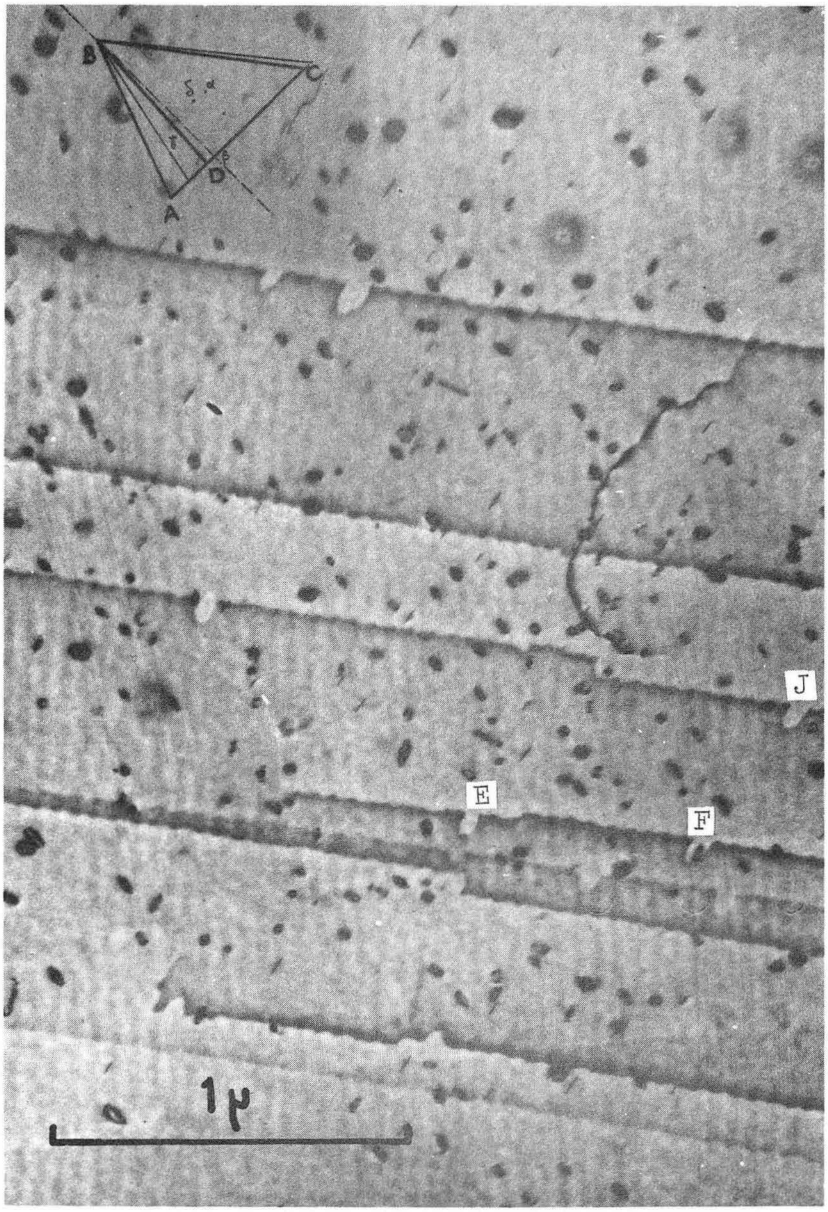
c. Sketch showing how the interaction taking place in Fig. 5 a, b leads to the formation of a helical segment on a moving dislocation.

Fig. 6. Interaction between a moving dislocation and a Frank sessile loop in which a smaller perfect loop is left behind.

Fig. 7. a, b, c and d. Examples of the interaction described schematically in Fig. 6.

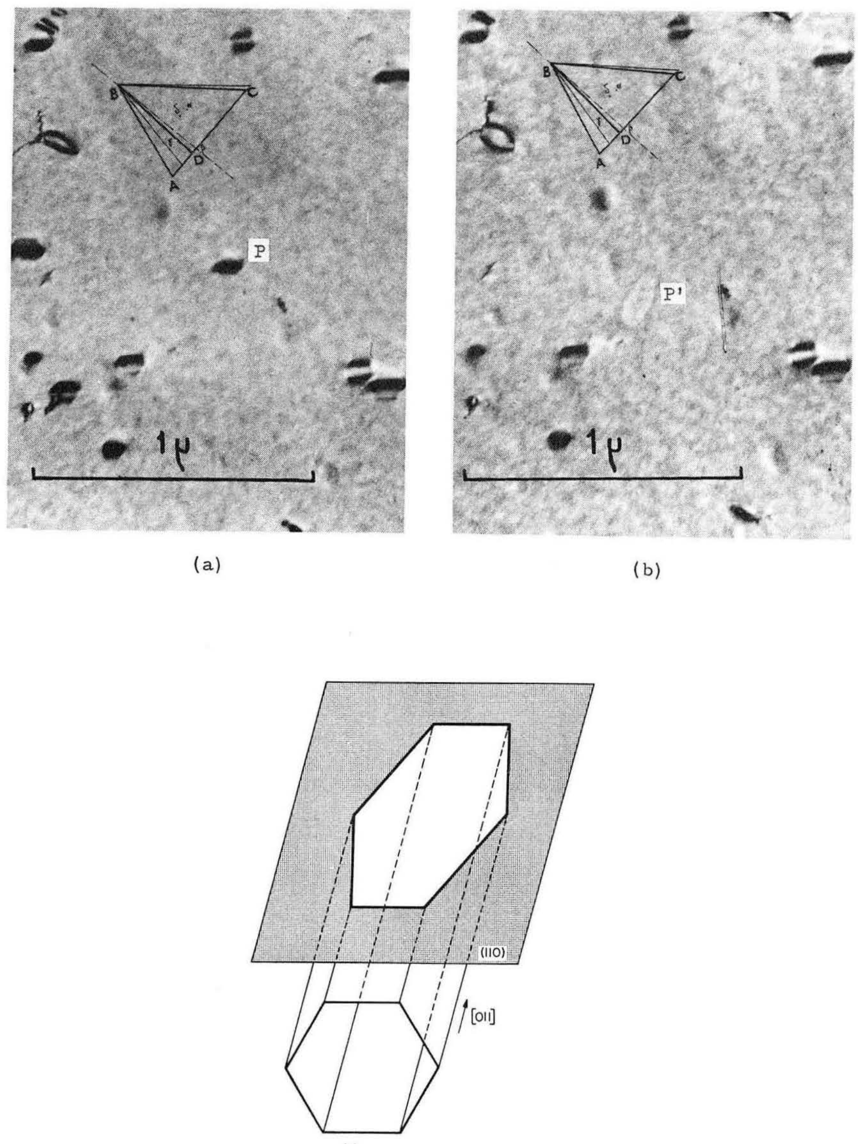
Fig. 8. a, b. Interactions with small loops produced by quenching from 650°C into 0°C water.

Fig. 9. a, b, c. Interaction in which the Burgers vector of the moving dislocation is parallel to the plane of the loop. Nodes N_1 and N_2 are formed and tend to be eliminated by glide towards one edge of the loop. A possible example of this case is shown at G in Fig. 1.



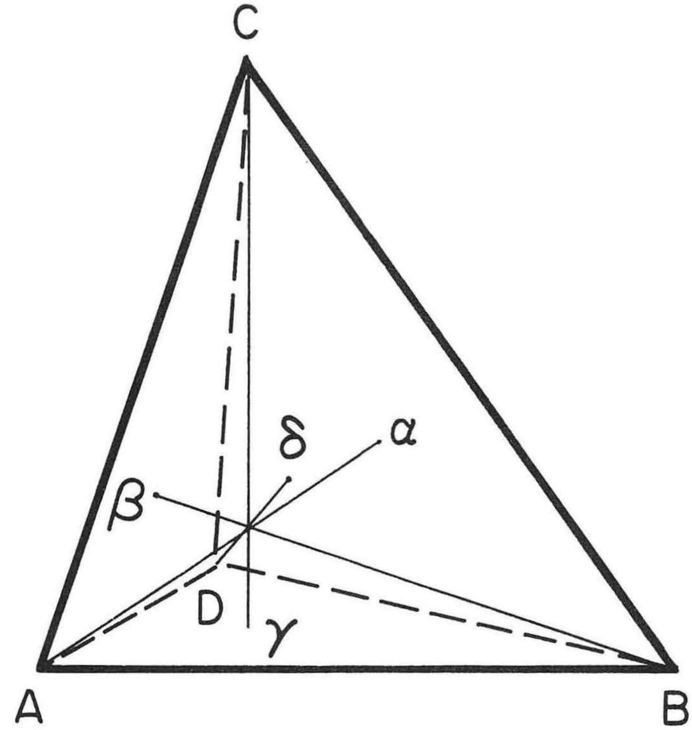
ZN-3978

Fig. 1



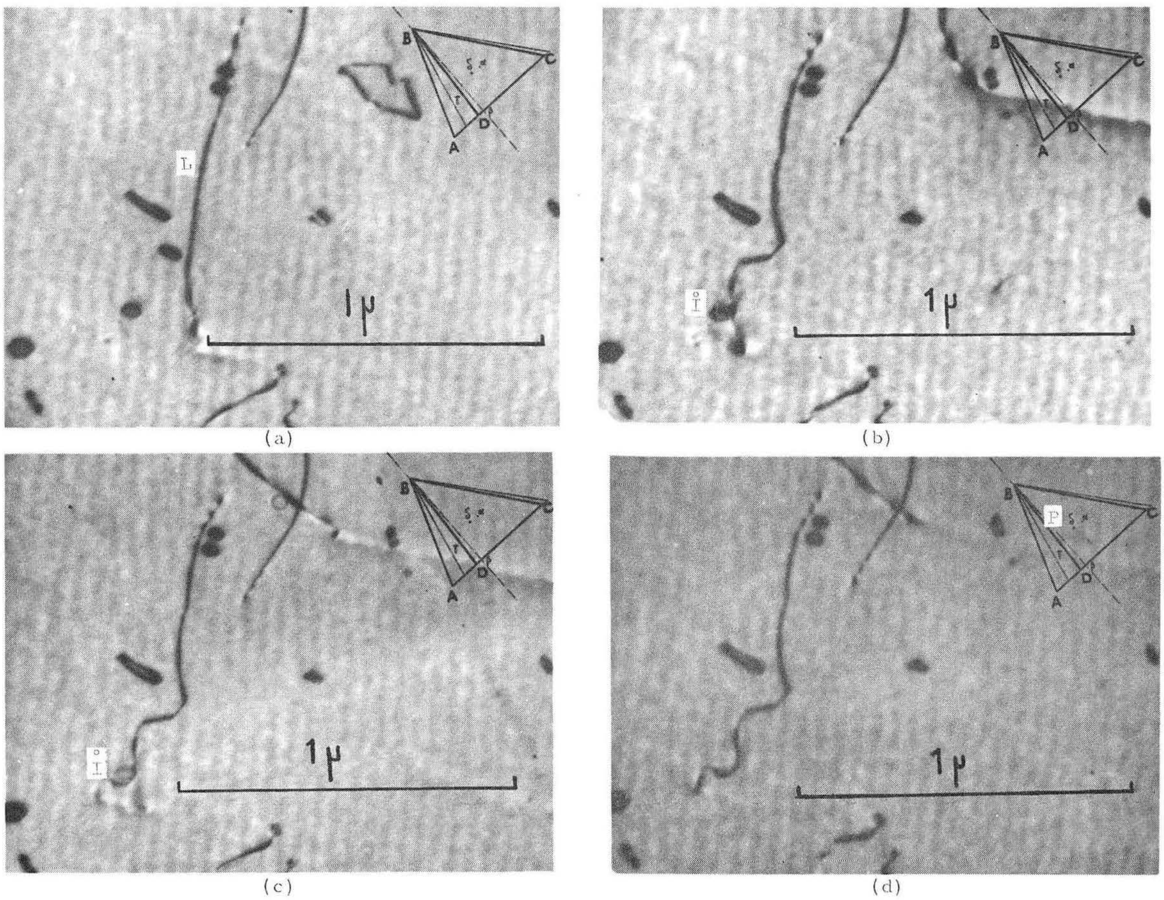
ZN-3980

Fig. 2



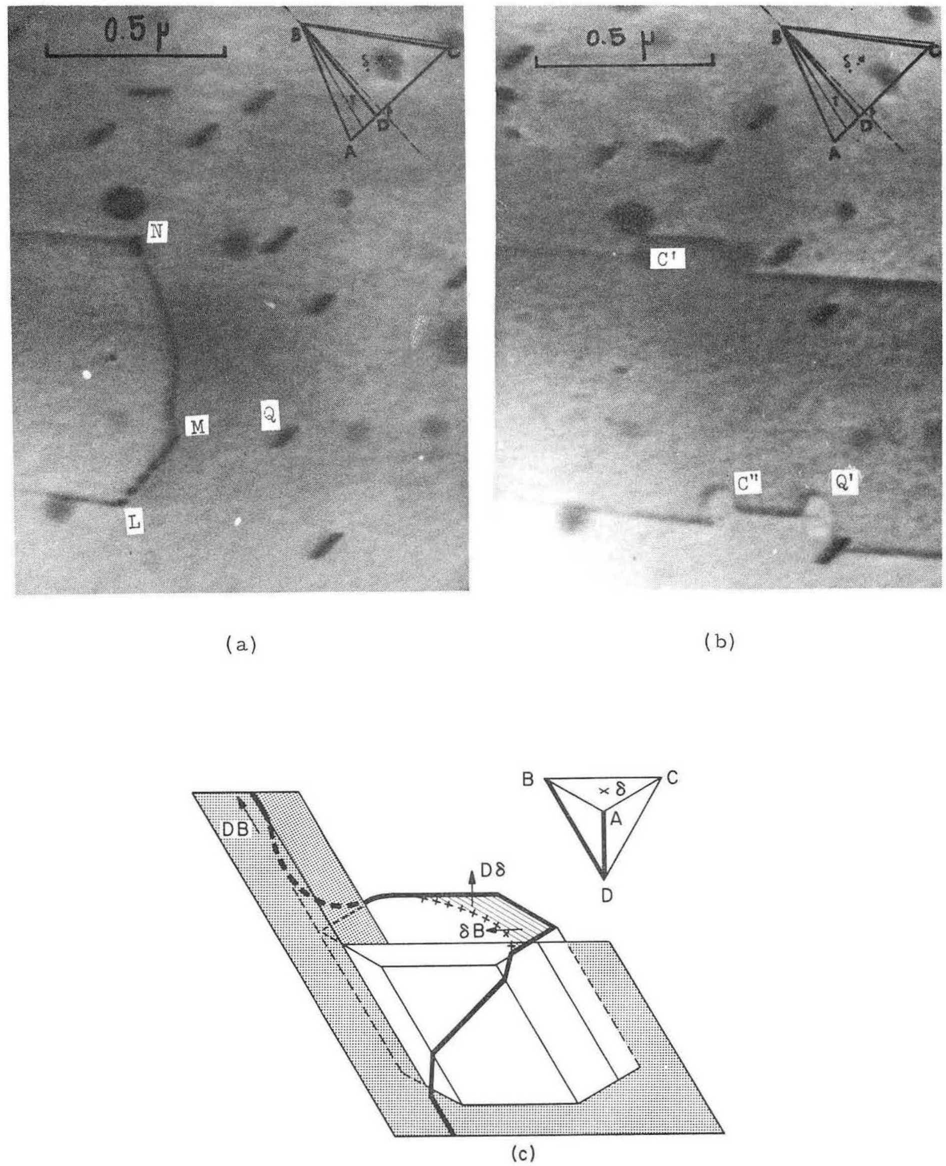
MU-30933

Fig. 3



ZN-3979

Fig. 4



ZN-3981

Fig. 5

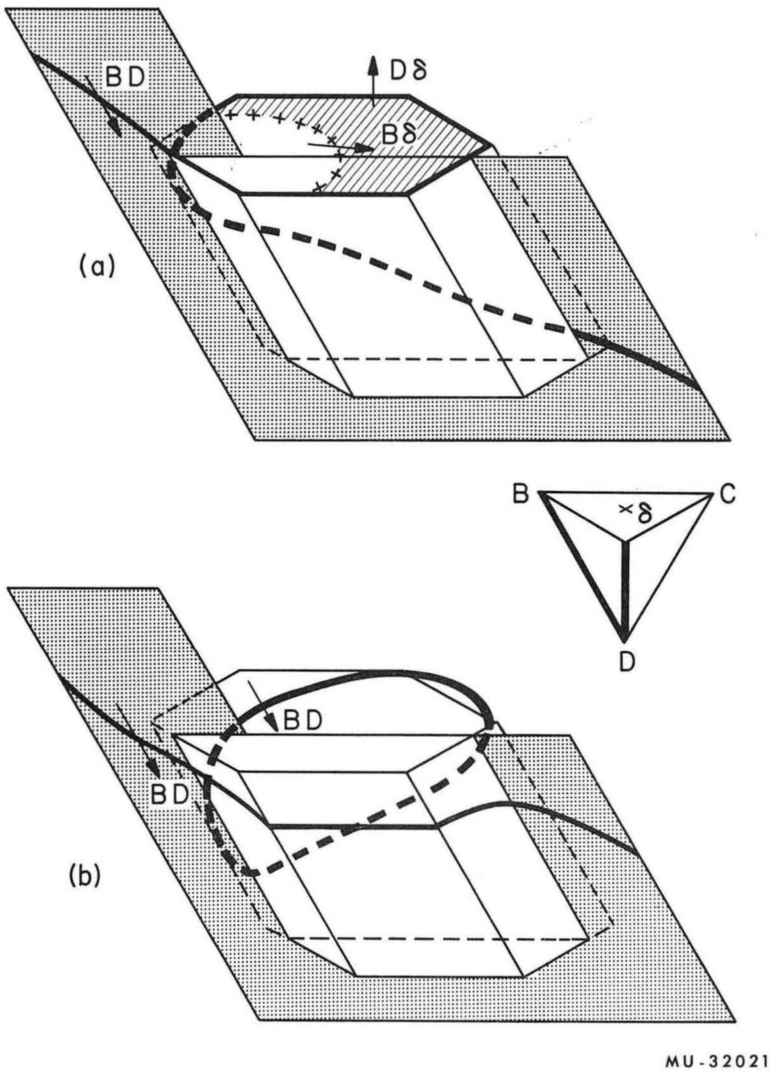
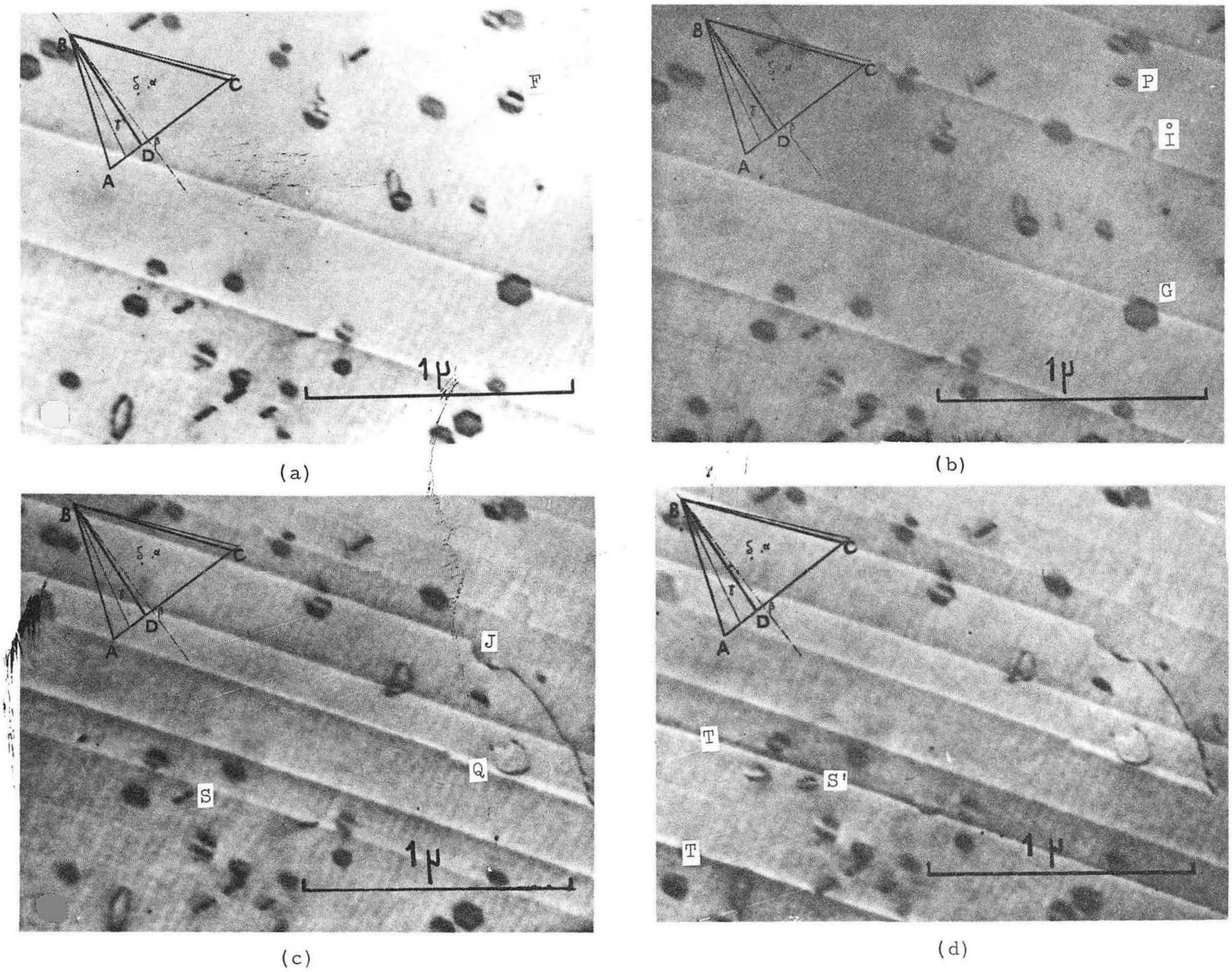
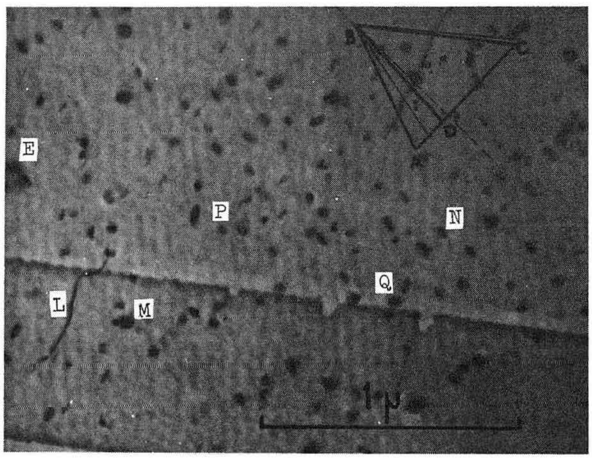


Fig. 6

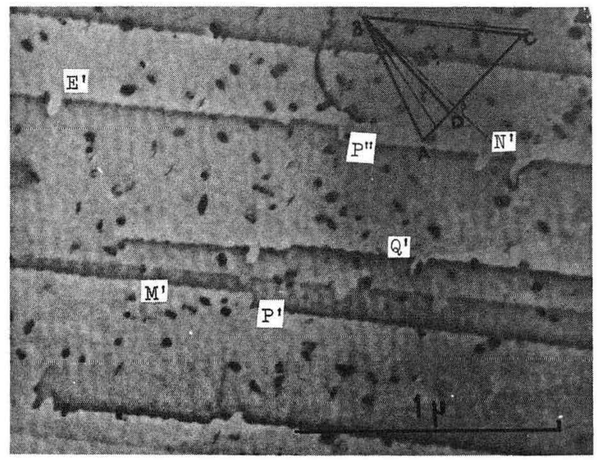


ZN-4010

Fig. 7



(a)



(b)

ZN-3982

Fig. 8

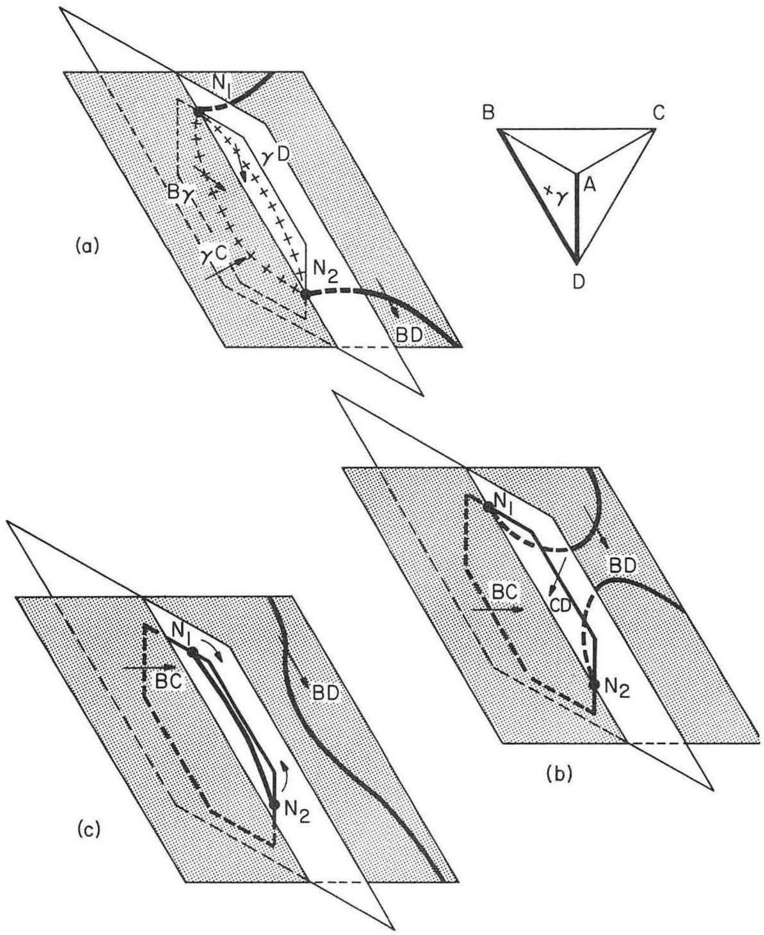


Fig. 9