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FATIGUE DEFORMATION OF MAGNESIUM OXIDE

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INTRODUCTION

The mechanism of plastic deformation of magnesium oxide and lithium fluoride has been studied by etch pit(1-8) and transmission electron microscopy(8-12) techniques. As a result of these experiments, the relationships between the behavior of individual dislocations and flow and fracture characteristics are probably better understood for LiF and MgO than for any metal.(13-22) However, even for these materials, there remain questions that are unresolved.

For example, in order to be able to relate yielding to the stress dependence of dislocation velocity, without making arbitrary assumptions, it is necessary to know the total length of moving dislocation line and how it varies with increasing strain. This parameter has not yet been measured. For example, it cannot be obtained by counting etch pits after a given strain. The transmission electron microscope observations show that a large fraction of the etch pits within a slip band must correspond to immobile dislocations, close pairs of dislocations, and prismatic dislocation loops. The etch pit density is a measure of the damage that has been left behind by moving dislocations as well as of the moving dislocations themselves.

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As has been pointed out previously, plastic deformation in magnesium oxide is concentrated at any instant at the surface of growing slip bands.[6,16] The rate at which the length of moving dislocation can increase in this material must therefore be influenced by the mechanism that operates to spread slip from one plane to a nearby parallel one. The double-cross-slip mechanism is the one most often proposed. However, there are other possibilities.[11] For example, if any region of the glide plane is cut by a large enough number of excess dislocations of one sign, there may be no need for double-cross-slip. Moving dislocations in this case can find helical paths that lead to spread of slip continuously to new parts of the crystal without any glide in the {100} cross-slip plane. This possibility seems particularly likely for specimens containing a network of subgrain boundaries.

The object of the present experiments[23] was to further study the mechanism of slip band growth in magnesium oxide. By using an alternating applied stress, very wide slip bands can be grown at a stress about equal to the yield stress as measured in a tension test.[24] Interactions between widening slip bands on intersecting systems can be studied without the development of stress concentrations severe enough to cause fracture.

EXPERIMENTAL TECHNIQUES

Thin sheets of magnesium oxide (approximately 1" x 1/2" x 0.01") were cleaved from large single crystals obtained from the Norton Company. These specimens were subsequently chemically polished with 85% orthophosphoric acid at 100°C, to remove the damage introduced by cleaving and to eliminate cleavage steps. The specimens were deformed in fatigue by cantilever bending. In these experiments both the surfaces of the

specimens experienced tensile and compressive stresses during each cycle. The maximum plastic strain was fixed at about 0.1% per cycle. None of the specimens fractures during a test. However, a large number of slip bands were always formed. After various numbers of cycles, specimens were etched in boiling nitric acid for 3 seconds to produce dislocation etch pits for optical observations.

Thin foils for transmission electron microscopy were prepared by a method that has been described previously.[10] Observations were made using a Hitachi HU-10 electron microscope operated at 100 Kv.

RESULTS AND DISCUSSION

The deformation took place primarily by growth of slip bands on the two {110} slip planes lying at 45° to the surfaces of maximum stress and parallel to the axis of bending. However, some slip occurred on the {110} planes at 90° to the top surface and at 45° to the axis of bending. Figure 1 is a schematic illustration of the mode of deformation. Figures 2 and 3 show both types of slip bands in regions that were almost completely covered by slip. Only a few thin strips of undeformed crystal remain. The magnification marker is drawn parallel to the band axis in all figures. Wide bulges have developed along the 90° slip bands wherever they cross a strip of undeformed crystal. This irregular slip band shape is possible for fatigue deformation because the net strain within a slip band can be small or even zero. Therefore, the end of a band is not necessarily a region of high stress concentration. Figure 4 is an example of simultaneous growth of wide bands on intersecting planes. Interference among the different systems has led to highly irregular slip band shapes.

The damage within the 45° bands, as revealed by transmission electron microscopy, is shown in Figures 5 and 6. The damage is of the same type as that found in slip bands that are formed during unidirectional strain:[9,10] i.e. edge dislocation pairs and elongated prismatic loops of various lengths with the spacing between the two dislocations of opposite sign varying from a few angstroms to a few hundred angstroms. The mechanism of formation of this kind of dislocation substructure has been discussed in previous papers.[9,10,11,12] The total density of dislocations was low compared to typical slip bands formed at 20°C by unidirectional stress application. Also, a greater fraction of the dislocations present were in screw or nearly screw orientation. In Figs. 5 and 6 the dislocation pairs extending in the horizontal direction are in edge orientation. Dislocation lines in the vertical direction with ends terminating at top and bottom surfaces of the foil lie in screw orientation. Even these were often paired as at "A" in Fig. 6.

Distribution of shear strain: The mode of deformation in these specimens suggests that even during fatigue straining moving dislocations are concentrated at the surfaces of the slip bands, i.e. (at the interfaces between the deformed material within the band and the unstrained crystal to either side). This conclusion is supported by the following observations:

(1) In specimens that were observed after various numbers of stress cycles, it was found that the number of growing slip bands tended to remain constant. As the crystal became nearly filled with slip by the growing together of bands on the 45° systems, then narrow bands on the 90° systems that had not previously been active, started to widen, resulting in the irregular shaped 90° bands shown in Figs. 2,3, and 4.

(2) The distribution of damage at the intersections of two orthogonal 90° bands also suggested that moving dislocations were only at the slip band interfaces. When moving dislocations with Burgers vectors at right angles cut through each other, jogs are formed on each. Therefore, it would be expected that where many such intersections occur there should be a greater than average density of edge dislocation pairs and prismatic dislocation loops. If moving dislocations are only at the slip band interfaces then at any instant during growth of intersecting orthogonal bands this increased damage will only be formed along the four lines of intersection between the four slip band interfaces. As both slip bands widen, these four lines of intersection will move away from each other generating two surfaces that contain a higher density of damage than any other part of the intersection. The etching grooves AB and CD that mark these surfaces are clearly visible in Fig. 7. If moving dislocations were not concentrated at the slip band interfaces, it would be difficult to explain these etching grooves. The increased damage should, in that case, be evenly distributed over the volume of the intersection.

Mechanism of slip band growth: Continuous widening of slip bands during fatigue is clear evidence that motion of a single dislocation across a glide plane leaves behind damage that tends to prevent its return glide over the same area, and that the dislocations left within the deformed volume are quite immobile. If this were not so, then in an experiment such as these where the strain amplitude is fixed, slip bands would stop growing wider when the total length of moving dislocation line became great enough to be able to produce the required strain by short back-and-forth motions over the same area. Apparently, a dislocation loop

that starts to spread over a slip plane on the first half cycle is often not quite able to return to its original position on the second half of the first cycle. On the next forward half cycle it reaches some still unswept parts of the glide plane and the process repeats until the entire area of the plane has been transversed. Slip must then spread to a nearby parallel plane.

The results are also interesting with regard to the mechanism of slip band widening. In order for slip to spread from one plane to another and therefore result in widening of a slip band, some segment of an expanding loop must move to a distance off the original glide plane of about:

$$h = \frac{Gb}{8\pi (1-\nu)\tau},$$

where G is the shear modulus, b is the Burgers vector, ν is Poisson's ratio and τ is the applied stress. If the stress is 10 kg/mm^2, this distance, h, for MgO is about 250 Å or about 80b. At this distance from the original plane the applied stress can move a dislocation through long distances without its getting stuck due to elastic interaction with the dislocation segments still lying on the old glide plane. The exact mechanism by which part of an expanding loop reaches a position many interatomic distances away from its initial plane of glide is still not definitely known although numerous possibilities have been discussed. (For a review, see references 11 and 25).

These results suggest that the external surface of the specimen may play a more important role than has usually been supposed. For example, the local widening of 90° bands in Figs. 2 and 3 where they cross narrow strips of undeformed crystal and also the existence of those undeformed volumes themselves as illustrated in Fig. 1 can be explained if it is

assumed that frequently the only place that dislocations can move far enough off the original glide plane to start a new slip layer is near an external surface that is parallel to the Burgers vector. In the case of 45° bands, this surface would be at the edges of the specimen. Therefore, when an undeformed strip is cut off from both edges by development of heavy enough 90° bands, there is no way for it to be filled in by further widening of the 45° bands. The fact that these strips often did not fill in with 45° slip is clear evidence that the number of sites on a slip band interface at which a new layer of slip can be started is not large. Since these undeformed strips were always in regions that were cut off from the edges by intersecting 90° bands, it suggests that the widening of 45° bands usually started at the edges.

Whereas the 45° bands could not grow to fill in these undeformed volumes, the 90° bands often did grow resulting in the kind of local widening illustrated by Figs. 2, 3, 4, and 7. This also would be expected according to the surface hypothesis because for these bands the Burgers vector was parallel to the top surface. The fact that the distance to which slip in the wide parts of the 90° bands has propagated into the 45° bands on either side is exactly symmetrical with respect to the position of the undeformed strip at the surface shows that continuous widening took place within the undeformed crystal and very near to the external surface. (The undeformed slab of crystal projects below the surface at 45°.)

Another interesting slip band structure that was sometimes observed is illustrated by the 45° bands in parts of Fig. 8. Particularly near the top of the photomicrograph a very regular spacing of glide layers within the band can be seen. The spacing between these rows of pits is

about an order of magnitude larger than the minimum spacing discussed previously. This type of slip band structure may be evidence for the helical ramp mechanism of slip band widening described in the introduction. (See also reference 25) For this mechanism, a very regular spacing that depends on the subgrain structure through which the slip band is growing would be expected.

ACKNOWLEDGMENT

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Figure Captions

Fig. 1 Schematic illustration of the widening of slip bands with increasing numbers of stress cycles - bottom end of specimen was clamped - top end was displaced alternately in both directions.

Fig. 2 Local widening of 90° bands at strips of undeformed crystal between wide 45° bands.

Fig. 3 Wide regions on parallel 90° bands that have grown together. The undeformed strip of crystal that allowed local widening has, in this case, subsequently filled in by further widening of the 45° bands.

Fig. 4 Irregular widening of two orthogonal sets of 90° bands.

Fig. 5 Transmission electron micrograph showing long dislocation dipoles, elongated prismatic loops and screw dislocations with cusps in a 45° slip band.

Fig. 6 Transmission electron micrograph showing dislocation pairs, A, that are nearly in screw orientation.

Fig. 7 Intersection of orthogonal slip bands; etching grooves AB and CD reveal increased density of dipoles and prismatic loops due to screw dislocations on the two systems cutting through each other.

Fig. 8 Regular spacing of active glide planes in a wide 45° slip band (near top of photomicrograph).

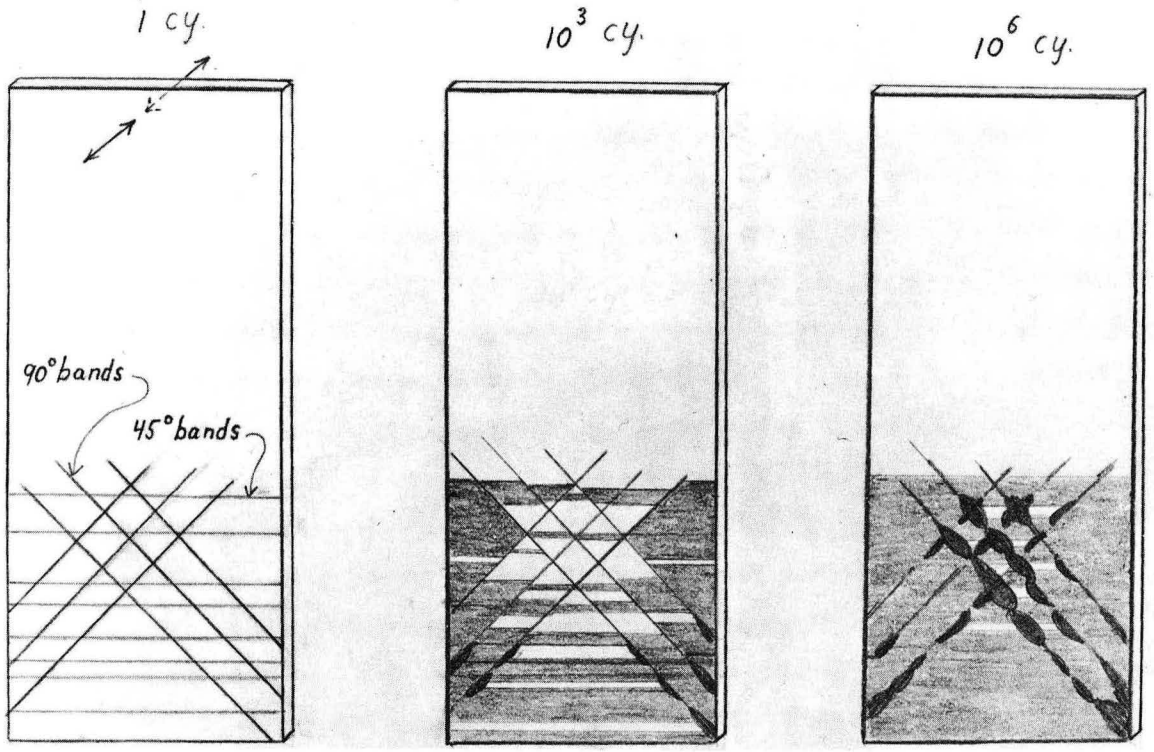

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Fig. 1

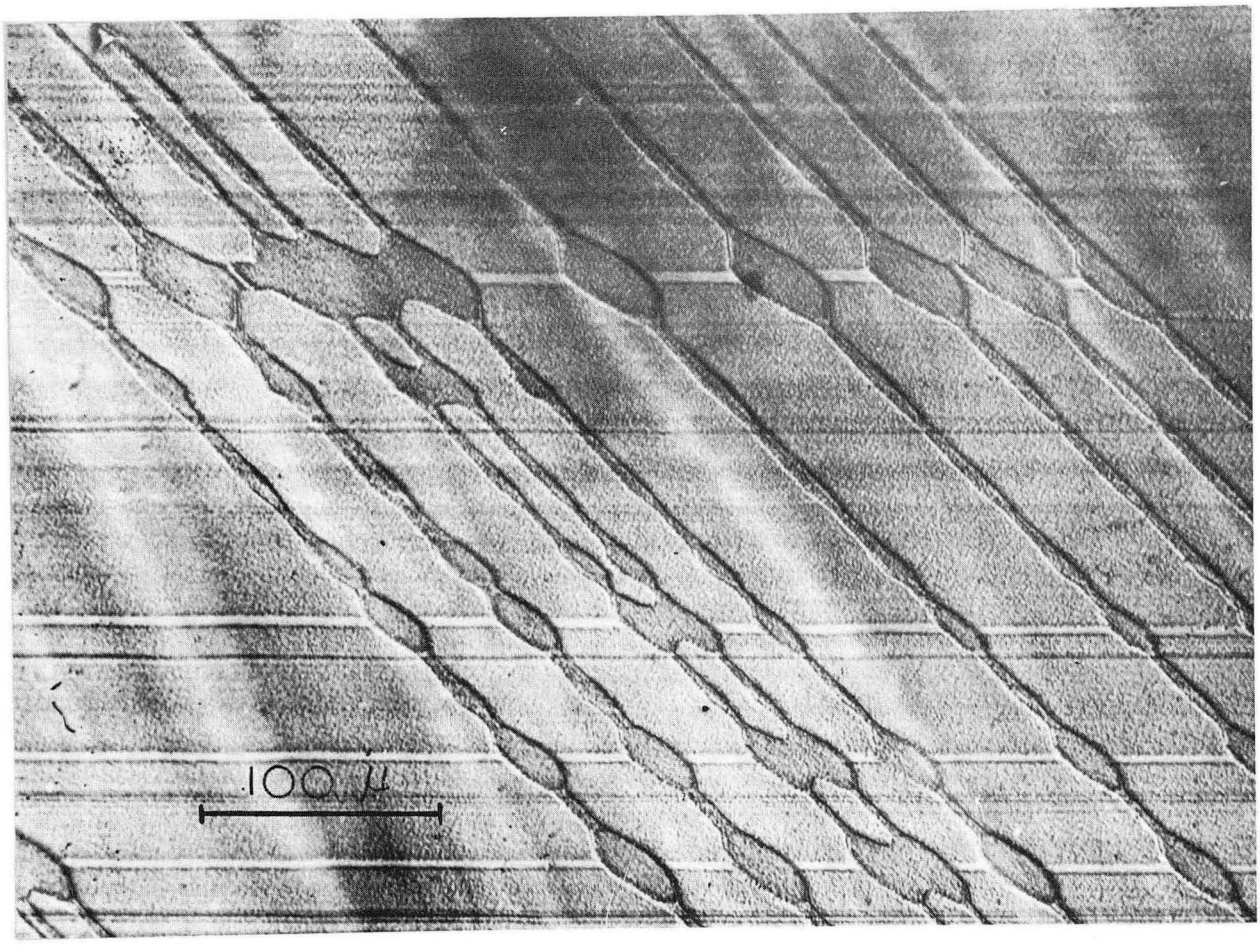

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Fig. 2

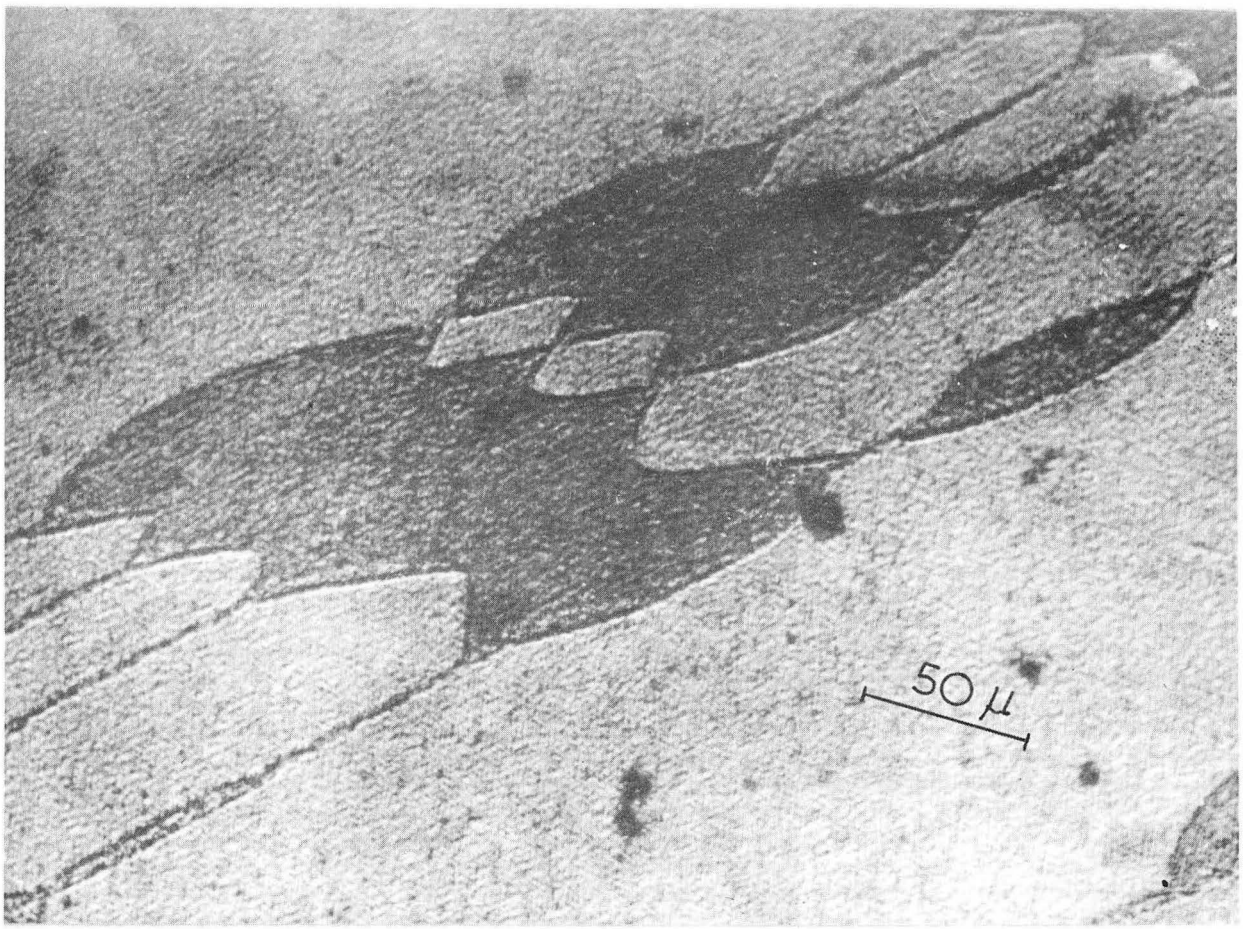

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Fig. 3

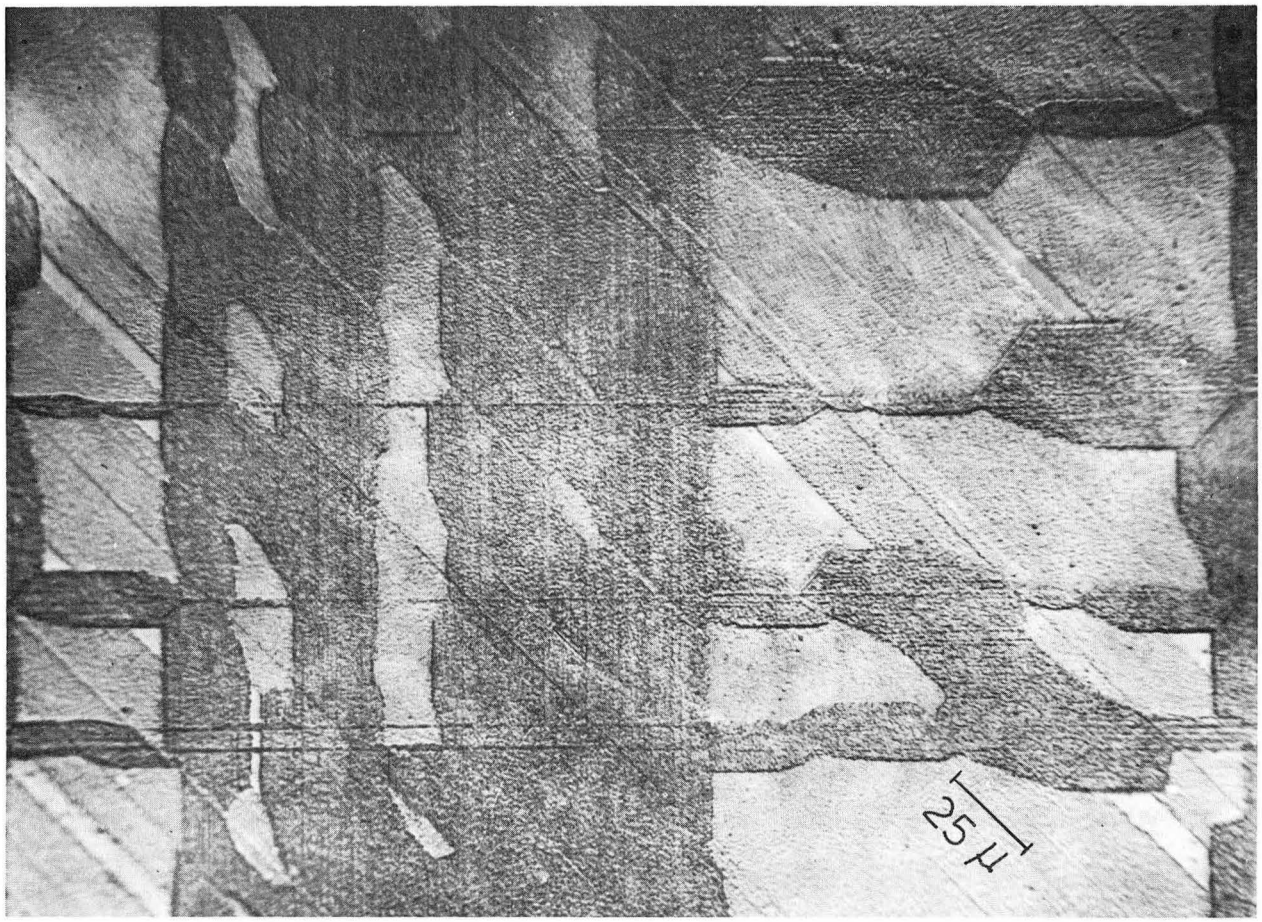

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Fig. 4

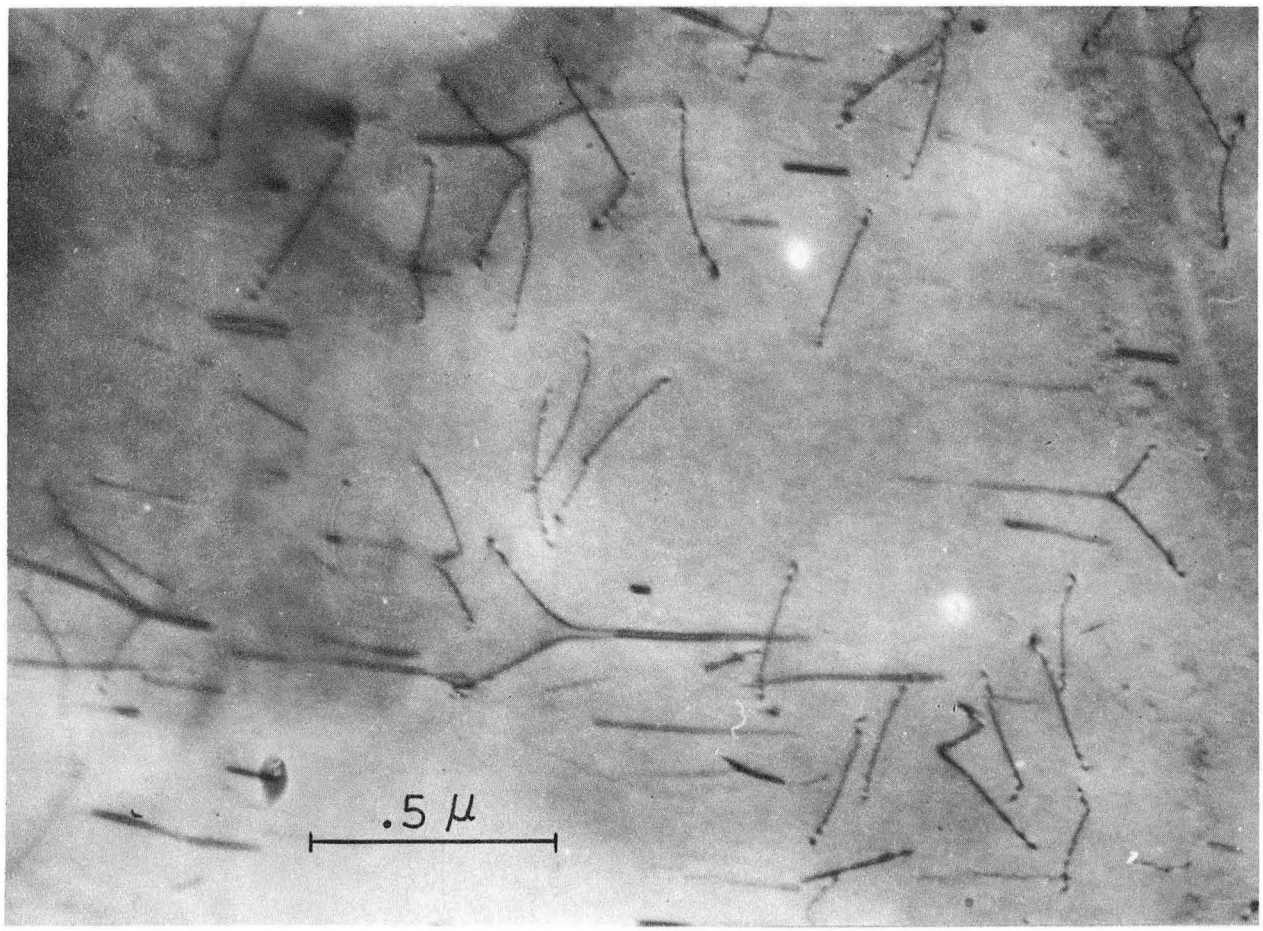

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Fig. 5

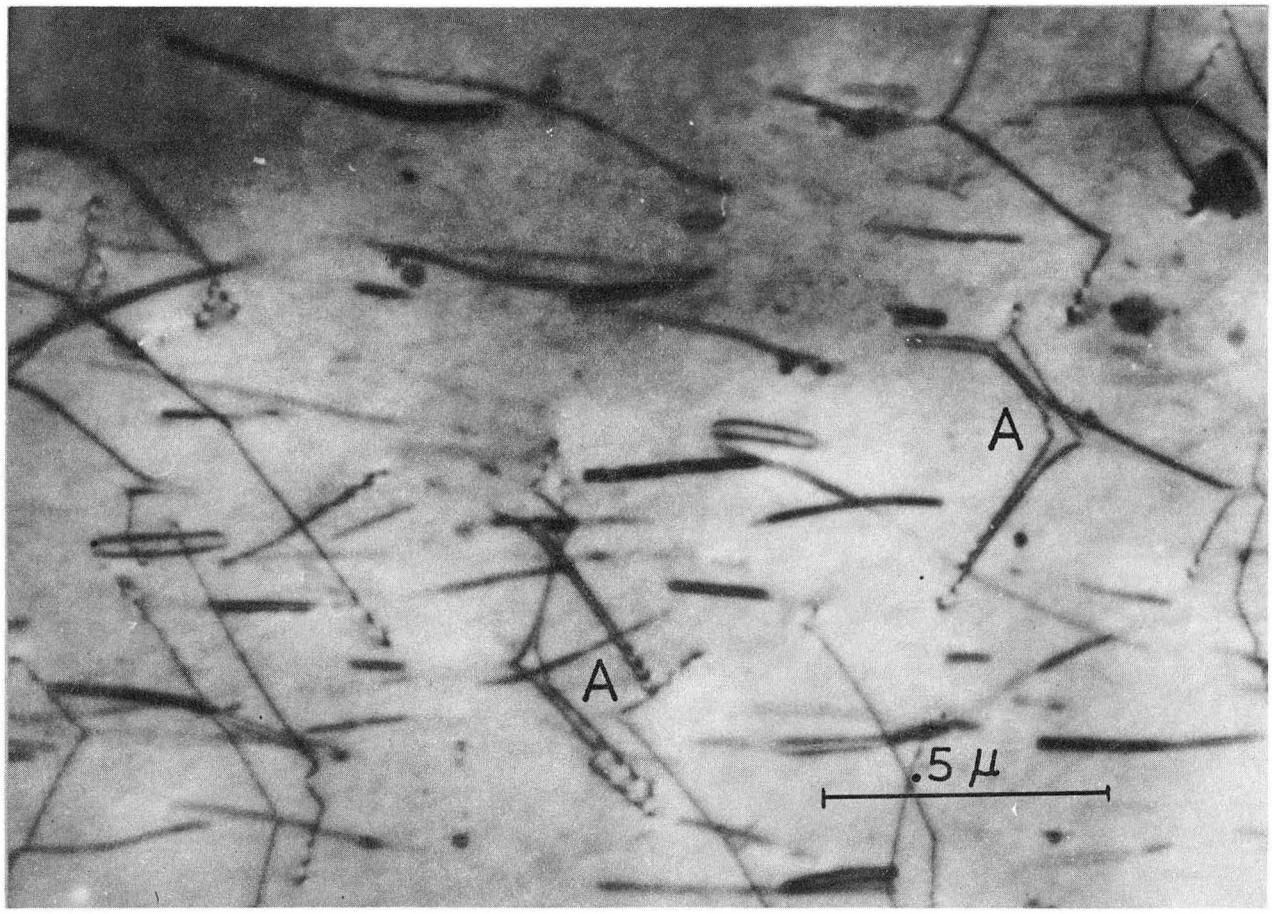

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Fig. 6

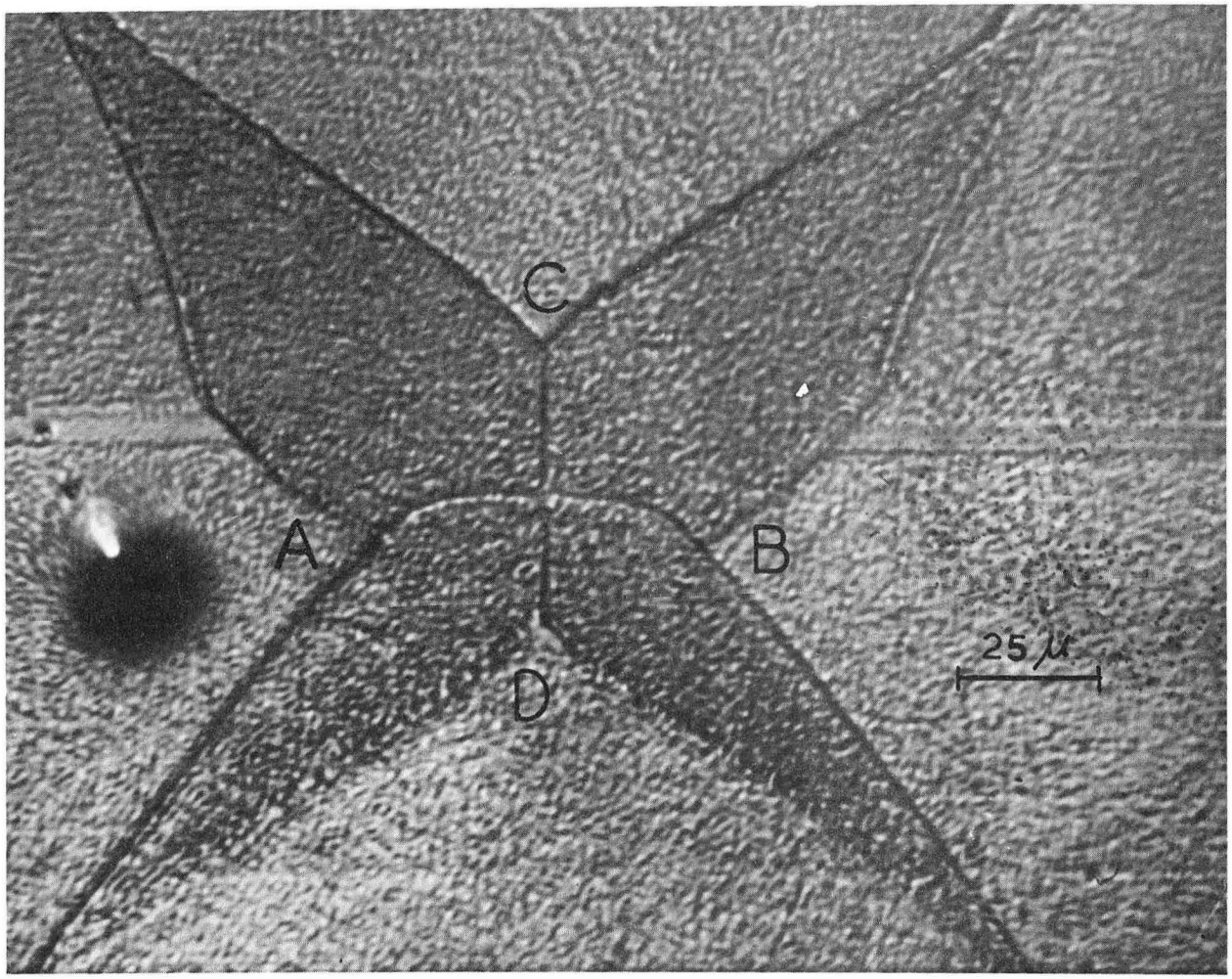

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Fig. 7

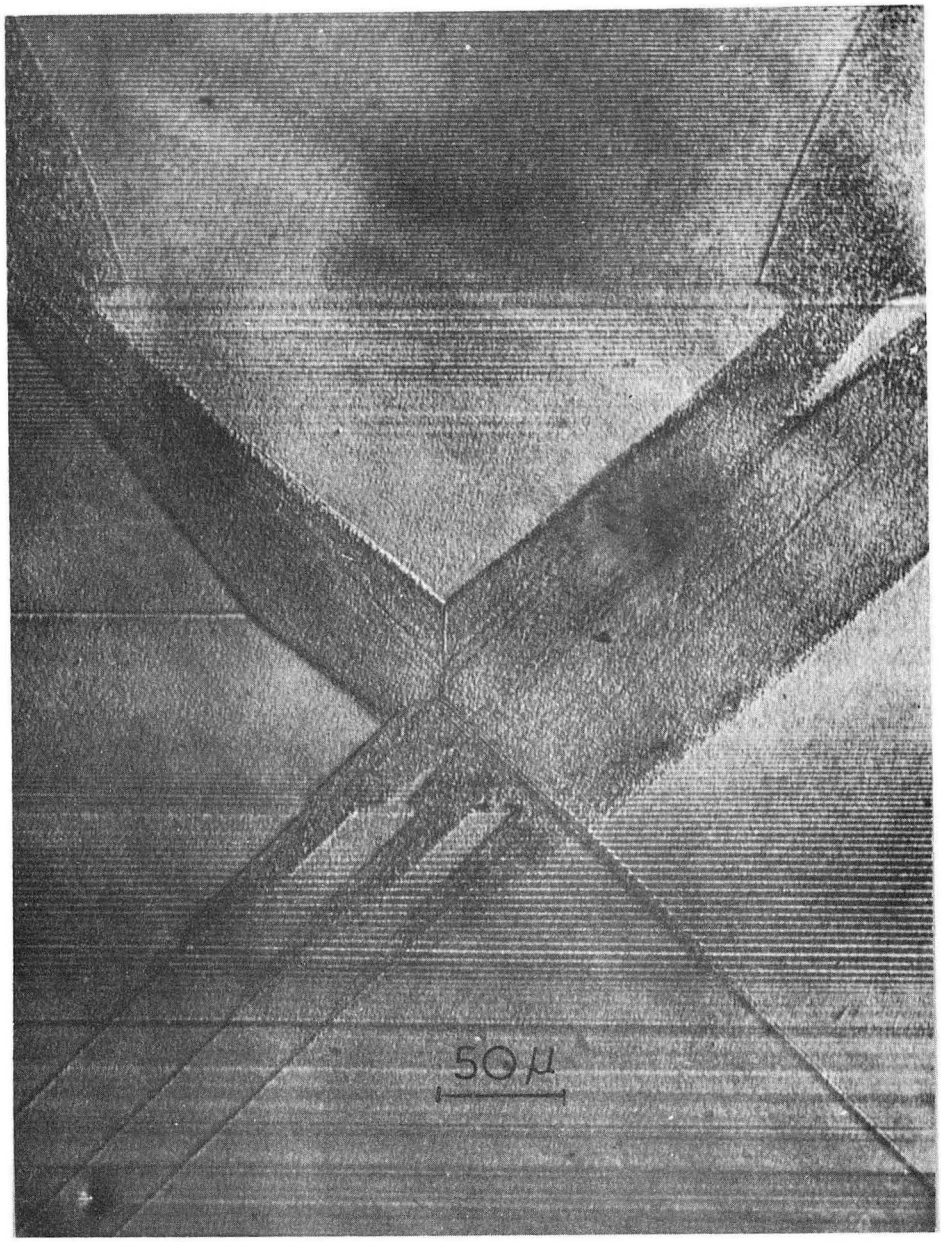

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Fig. 8

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