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by

W. E. Elkington,* G. Thomas** and J. Washburn**

ABSTRACT

The dislocation substructures in single crystals of MgO formed in four-point bending at temperatures from -196°C to 1300°C have been observed by transmission electron microscopy. Elongated edge dislocation pairs were found at all deformation temperatures. The majority of pairs originated where screw dislocations intersected grown-in dislocations. Grown-in dislocations always contained impurity precipitates along their length and often did not lie exactly in a slip plane. Because of either or both of these factors, grown-in dislocations remained immobile during deformation.

The stability of dislocation pairs depended upon the separation of the two dislocations and the deformation temperature. Narrow pairs broke up into small prismatic loops at 750°C and above. The width of the largest observed pairs approached but never exceeded the calculated value;

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

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INTRODUCTION

The mechanism of plastic deformation in magnesium oxide and other crystals exhibiting the NaCl structure has been extensively studied because: (a) Simple and reliable etch pit techniques which reveal the positions of dislocation lines have been developed.[1] (b) Direct observations of dislocation lines by transmission electron microscopy can be conveniently made in MgO.[2] (c) Specimens of high structural perfection have been available. (d) In some respects the crystallography of slip is simpler in the NaCl structure than in any metallic structure.[3] Consequently, it has been possible for these materials (to a greater extent than for metals) to relate plastic properties directly to the behavior of individual dislocations.[3, 4, 5, 6]

In MgO it has been possible to study in detail, by transmission electron microscopy, the damage left by moving dislocations.[2, 7] Previous work has shown that the most important defects within a slip band are elongated edge dislocation loops (dipoles). It is this kind of damage that causes slip to stop on an active plane and to spread to nearby parallel planes; it also results in strain hardening whenever further plastic deformation requires intersection or overlapping of slip bands.[5]

The purpose of the present experiments was to further study the origin of the dislocation pairs and prismatic loops and also to try to clarify the mechanism of dislocation multiplication that operates during the widening of slip bands. The interactions between moving dislocations and immobile dislocations and precipitates were also investigated over the temperature range -196°C to 1300°C.

EXPERIMENTAL PROCEDURES

Test Sample Preparation

Bulk MgO single crystals obtained from the Norton Company were used in this study. To prepare test samples, thin sheets approximately 0.02 x 0.3 x 1 in. with {100} faces were cleaved from the bulk crystals. After being cleaved, the sheets were mechanically polished to remove cleavage steps and annealed for two hours at 1250°C to remove the surface damage which occurred during polishing. The sheets were then chemically polished to obtain smooth surfaces and to remove any remaining surface damage. The final sheet thickness varied from 0.009 to 0.018 in.

Test Procedure

In the temperature range -196°C to 750°C, four-point bend tests were performed using a stainless steel testing frame mounted on an Instron testing machine. The span length of the frame was 3/4 in. with the inner knife edges 1/4 in. apart. The two center knife edges could be moved about two perpendicular axes to provide uniform loading. The outer knife edges were fixed but were parallel to within 0.001 in. in 1/2 in. All samples were deformed with the testing machine operating at a constant cross-head speed of 0.05 cm/min. The deflection of the samples was taken as the cross-head displacement and so was a measure of the net displacement between the center knife edges with respect to the outer knife edges.

The samples were heated by a small resistance furnace which fitted around the testing frame. Temperature measurements were made using a thermocouple which was placed about 1/8 in. below and under the test samples. After being tested, the samples were immediately removed from the furnace and allowed to air cool.

All samples were deformed by an amount corresponding to an outer fiber strain of approximately 1%. The outer fiber stress was calculated using elastic theory throughout the load deflection curve.

Samples deformed at 1000°C and higher were bent by manually pressing an alumina rod against the back face of the sample while it was in the furnace. After being deformed, the sample was removed from the furnace and allowed to air cool.

Transmission Sample Preparation

The deformed specimens were next etched for a few seconds in hot nitric acid. The general area selected for examination in the electron microscope was thinned by translating the sample slowly along a circular path above a small jet of hot orthophosphoric acid (110°C). The bend samples were polished from the compression side so that the thin sections obtained were from the tensile surface. Polishing was continued until a hole appeared at some point. The sample was then immediately immersed in distilled water and rinsed in alcohol and anhydrous ether. A diagram of the polishing apparatus is shown in Fig. 1.

The hole produced was usually less than 1 mm in diameter. Areas sufficiently thin to be transparent to the electron beam of the microscope extended in a few hundred microns from the periphery of the hole and could be detected by bright interference fringes when observed in vertical illumination.

The next step in preparing the transmission samples was that of removing the thin section from around the hole. The following procedure was finally adopted. A small drop of Lacomit masking lacquer on the end of a fine paint brush was lightly touched to the sample in the area of the hole. The Lacomit wet the surface around the hole forming a circle about 2 mm in diameter which, when dried, protected the thin section. The sample was again polished over the hot acid jet and new holes were allowed to form around the mask. Just before these secondary holes started to join, polishing was stopped by immersing the sample in distilled water. The mask was then removed by disolving it in acetone and the sample was rinsed in distilled water, alcohol, and anhydrous ether. At this point the thin section around the periphery of the hole was almost completely surrounded with secondary holes, making it a rather simple matter to chip the material between the secondary holes and allow the thin section to fall into an electron microscope holder. Figure 2 shows a specimen which has been prepared and placed on an electron microscope grid in this manner.

Observations were made using both a Hitachi HU-10 and a Siemens Elmiskop I electron microscope operating at 100kV.

RESULTS AND DISCUSSION

Mechanical Test Results

When an MgO sample is deformed in four-point bending, there is an equal shear stress on four of the six possible $\langle 110 \rangle \{110\}$ slip systems. Two of the active systems are on planes which intersect the tension surface (a $\{100\}$ plane) at 45° and two intersect it at 90°. Hereafter, these planes will be referred to as 45° and 90° planes, respectively. In deforming as-polished samples, despite the equal shear stress on the slip planes, deformation occurred predominantly on 45° planes concentrated under the center knife edges. The stress deflection curve for such samples invariably had a high yield stress followed by a sharp drop and jerky flow (Fig. 3a). The high yield stress followed by a sharp drop in stress appears to be a result of the higher stress necessary to nucleate dislocations in MgO than to move them.[8]

Since in this study it was desired to observe dislocations on both types of slip planes, it was necessary to force the 90° planes to operate. To do this, the tension surface of all remaining samples was lightly sprinkled with alumina particles to introduce fresh dislocations on all slip planes. The effect this had on the stress deflection curve can be seen in Figs. 3b and 3c. The yield stress was lowered and flow was smooth. Microexamination revealed that deformation had occurred on both the 45° and 90° slip planes and was uniformly distributed between the center knife edges.

Sprinkling was effective in activating both types of slip systems and in giving uniform deformation at test temperatures to 550°C. At 750°C, however, samples again showed a high yield stress, a sharp drop in stress, and jerky flow (Figs. 3h and 3i). Microexamination revealed that, as in the case of the as-polished samples, deformation had occurred mainly on 45° planes concentrated under the center knife edges. Further examination showed that the dislocations introduced by sprinkling had not moved during deformation. Figure 4 shows a "rosette" of such "pinned" dislocations in a sample deformed at 750°C. In contrast to this behavior, Fig. 5 shows how fresh dislocations have formed slip bands in a sample deformed at room temperature.

It is apparent that some impurity diffuses to dislocations at temperatures above 500°C causing them to become pinned. This "pinning" or "aging" phenomenon is also observed in LiF.[1] Although aging at 500°C and 750°C for short periods did lock dislocations, it did not produce any precipitates along the dislocations large enough to be observed directly in the microscope or to cause any noticeable change in the electron diffraction contrast of the pinned dislocation lines.

Observations

When transmission samples were viewed in the electron microscope, a {100} plane was always very nearly perpendicular to the electron beam. Therefore, dislocation images were always a projection onto a {100} plane. In the NaCl structure there is only one <110> slip direction for a given {110} slip plane so that all glide dislocations in a given plane will have the same Burgers vector. In a 45° plane, therefore, a pure screw dislocation appears as a straight line lying along a <100> direction. Its length is determined by the thickness of the foil because it terminates at the top and bottom surfaces of the crystal. A pure edge dislocation on this inclined plane appears as a straight line parallel to the trace of the glide plane on the plane of the foil. A mixed dislocation may have any shape and direction. In a 90° band a pure screw dislocation, a mixed dislocation, or even curved lines appear in projection as straight lines lying parallel to the trace of the glide plane which is a <110> direction. A pure edge dislocation is seen end on.

The Effect of Temperature on Dislocation Substructure

The character of the substructure within slip bands depended markedly on the deformation temperature. On 45° planes of samples deformed at -196°C the substructure was very dense, being composed of lines which were irregular in shape (Fig. 6). Only a few resolvable dislocation pairs were observed in these samples but many of the lines appeared to be wider and darker in contrast than others. These were probably pairs with a spacing below the limit of resolution. At ambient temperatures the substructure was less dense and dislocations tended to be straighter. Many dislocation pairs were readily resolved in these samples. Further increase in deformation temperature resulted in a continuous decrease in dislocation density. Figure 7 shows a typical substructure in a sample deformed at 750°C. Increasing the temperature to 1000°C and 1200°C further decreased the dislocation density but not to the degree observed in the lower temperature range. The density of dislocation pairs also decreased with increasing temperature but the average pair size increased. Figures 8 and 9 show the largest pair spacing observed in samples deformed at room temperature and 500°C respectively.

In samples deformed at -196°C dislocations and pairs were always accurately on the {110} glide planes. (Fig. 10). Therefore, when observed on the 90° planes, the projections were straight lines parallel to a {110} direction. For samples deformed at room temperature, the projections, while being straight lines, often deviated by a few degrees from a $\langle 110 \rangle$ direction (Fig. 11). This misalignment indicates that the dislocations must contain many small uniformly spaced jogs along their length. In a sample deformed at 1300°C dislocations on 90° planes often contained jogs large enough to be resolved. Figure 12 shows such jogs in an area where two 90° slip bands have intersected; large jogs were, however, not uniquely associated with intersections since some were also observed in areas away from slip band intersections.

In samples deformed at 750°C and above, narrow dislocation pairs broke up into rows of small prismatic loops. Figure 13 shows a slip band within which this had occurred in a sample deformed at 1200°C. This phenomenon has been observed previously by Washburn et al. in MgO[7] and by Price in Zn[9] and Cd.[10] The driving force for this change in configuration has been shown to be the associated decrease in elastic energy.[7, 9] Since the transformation occurred at temperatures where thermally generated vacancies were present in small concentration, the mechanism appears to be one of local diffusion along dislocations. The sharp curvature at the end of the dislocation pairs provides an additional climb force and it is here that the pinching off of loops occurred.

An increase in deformation temperature can affect the dislocation substructure by: (a) increasing the ease of slip on secondary slip planes, (b) increasing the rate of climb for a given climb force, (c) increasing the equilibrium concentration of jogs on a dislocation, and (d) by decreasing the stress necessary to move dislocations.

Figure 3 shows that the yield stress, which can be defined as the stress necessary for the growth of wide slip bands, drops rapidly with increasing temperature in the range -196°C to 250°C but does not change much on going to higher temperatures. It has also been shown by Hulse et al.[11] that the stress necessary to move dislocations on the {100} cross-slip planes changes even more rapidly with temperature. At room temperature the stress required to move a dislocation on the {100} plane in MgO is probably at least 50 times higher than that required for {110} slip band growth. However, at 1000°C the ratio of these two stresses appears to be only 2 or 3 instead of 50. Therefore, at the higher deformation temperatures it becomes increasingly possible for dislocations of like sign to avoid each other by cross-slip and for dislocations of opposite sign to glide together and annihilate. Cross-slip of dislocations in order to by-pass others of like sign has been directly observed in thin foils.[2] In these observations the temperature during the motion was not known exactly but it was probably in excess of 1000°C due to heating by the electron beam. Since jogs must glide on {100} in the sodium chloride structure, the critical stress to move dislocations on {100} is directly related to jog mobility.

The fact that the decrease in density of pairs within the slip bands is paralleled within the same temperature range by the decrease in flow stress, is consistent with the hypothesis that widening of a slip band depends on the formation of a widely spaced pair.

The mechanism of pair formation in MgO has been described previously.[7, 3] Briefly, a dislocation pair will be formed when a moving screw dislocation develops a jog large enough to pin it at a point. When a screw dislocation becomes pinned in this manner a plus-minus edge dislocation pair will be formed when the unpinned arms on either side of the jog move forward. If the unpinned arms are on slip planes far enough apart, they will pass over each other and act as a dislocation mill. If they are on slip planes close together they cannot pass and a relatively stable pair will be formed. The critical separation, h, of the slip planes is given by:

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

where G is the shear modulus, b is the Burgers vector, ν is Poisson's ratio, and τ is the applied shear stress. It can be seen from this relation that for each stress there is a maximum stable pair size. As the applied shear stress necessary to cause flow decreases with increasing temperature, the maximum stable pair size increases. The maximum separation that was observed approached, but never exceeded, this calculated value. For the pairs in Fig. 8 and Fig. 9 the observed separations were approximately 30% smaller than the calculated maximum in each case.

Nucleation of Pairs at Grown-in Dislocations

The grown-in substructure in NaCl type single crystals has been shown to consist of planar arrays of dislocations which make up subgrain boundaries and of random three-dimensional networks of individual dislocations. Microexamination of etched MgO crystals used in these experiments showed that the crystals had a subgrain size which varied from 1mm to 10mm in diameter. Within the subgrains there was an etch pit density of approximately $5 \times 10^4/cm^2$. Most of these pits had conical bottoms and marked the point of emergence of a grown-in dislocation at the crystal surface. A few of the pits were flat bottomed. It is believed that these flat pits occurred at impurity precipitates which may or may not have been associated with a dislocation.

It was found that the grown-in dislocations always contained precipitates along their length and often did not lie exactly in a slip plane (Fig. 14). Because of either one or both of these factors, the grown-in dislocations remained immobile during deformation.

The size and spacing of the precipitates varied from one sample to another and in some cases varied from one dislocation to another within the same sample. Large precipitates were generally present at dislocation nodes. The spacing of visible precipitates was not always small enough to have pinned the dislocation at the maximum stress level reached in the test. In Fig. 14 the dislocation should have been able to escape from the line of precipitates during the test because the yield stress of the crystal was greater than $\frac{Gb}{\ell}$ where ℓ is the largest distance between precipitates. This suggests that the large precipitates that are observed do not play an important role in the locking of grown-in dislocations. They are immobile either because of invisible impurity atmospheres or because they contain a high jog concentration.

Although the grown-in dislocations do not move during deformation, they probably play a very important role both in the mechanism of multiplication and in the formation of pairs. Figure 15 shows a high density of pairs that start at a grown-in dislocation which cuts through the glide plane. Many areas of this type were observed and a large fraction of the pairs observed in a given crystal started at either a grown-in dislocation or at an impurity precipitate.

The nucleation of pairs at grown-in dislocations suggests that many of the glide dislocations moving through a crystal already contain jogs. A jog on a dislocation is a short segment of edge dislocation which is at right angles to the primary glide plane and can glide only along the direction of the Burgers vector. If the jogs are forced to move in any other direction, they must climb. This requires emission of vacant lattice sites or interstitial atoms. When a jogged dislocation intersects a grown-in dislocation, it is quite likely that a few jogs may glide together at the cusp that is created to form an immobile multiple jog. In this case, a dislocation pair will be formed when the unpinned portions of the dislocation on either side continue to move forward.

This mechanism of pair formation can explain the origin of most of the pairs observed in the deformed MgO samples. Sometimes, however, closed pairs were found in the slip bands in regions away from grown-in dislocations which suggests that other nucleation mechanisms may operate.

Acknowledgment

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Key to Figures

Fig. 1 Polishing Apparatus.

Fig. 2 Transmission sample on an electron microscope grid.

Fig. 3 Effect of temperature and surface condition on the stress deflection curve.

Fig. 4 Rosette of "aged dislocations" in sample deformed at 750°C.

Fig. 5 Rosette of "fresh" dislocations which has initiated slip bands during deformation.

Fig. 6 Dense dislocation substructure on 45° planes in a sample deformed at -196°C.

Fig. 7 Dislocations on 45° slip planes in a sample deformed at 750°C.

Fig. 8 Largest pair observed in a sample deformed at room temperature.

Fig. 9 Largest pair observed in a sample deformed at 500°C.

Fig. 10 90° slip band in a sample deformed at -196°C.

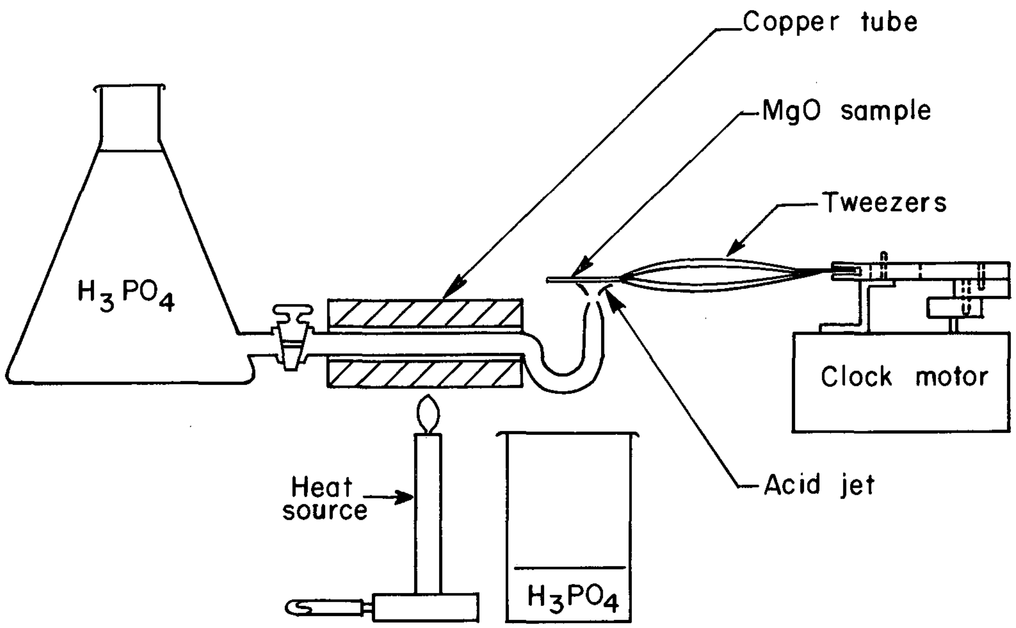
Fig. 11 90° slip band in a sample deformed at room temperature. Arrows locate dislocations which deviate from <110> alignment.

Fig. 12 Intersection of two 90° slip bands in a sample deformed at 1300°C. Arrows locate large jogs.

Fig. 13 Dislocation pairs formed at grown-in dislocations and at impurity precipitates. Smaller pairs have broken up into prismatic loops.

Fig. 14 Grown-in dislocation which does not lie on a slip plane. Dotted lines show approximately the projection of a {110}.

Fig. 15 Dislocation pairs formed at a grown-in dislocation.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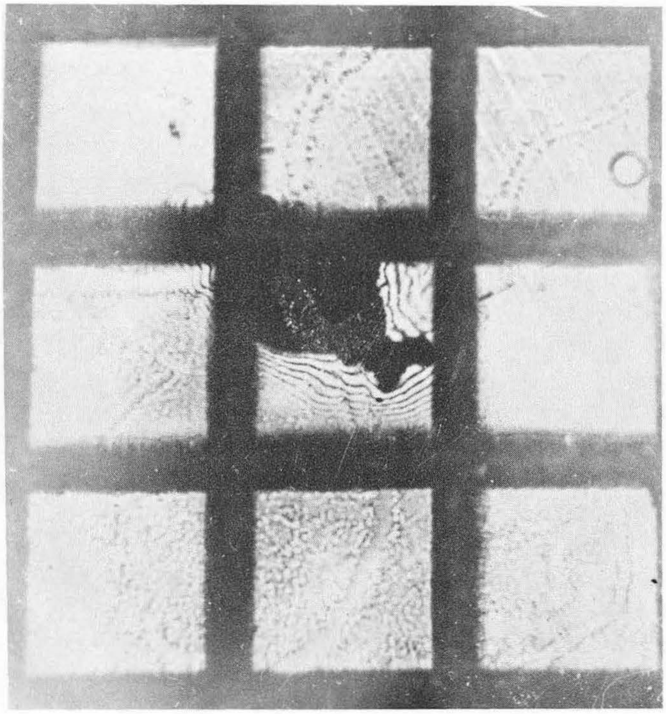
MgO sample
Tweezers
H_3PO_4
Clock motor
Acid jet
Heat source
H_3PO_4

MU-27554



100 μ

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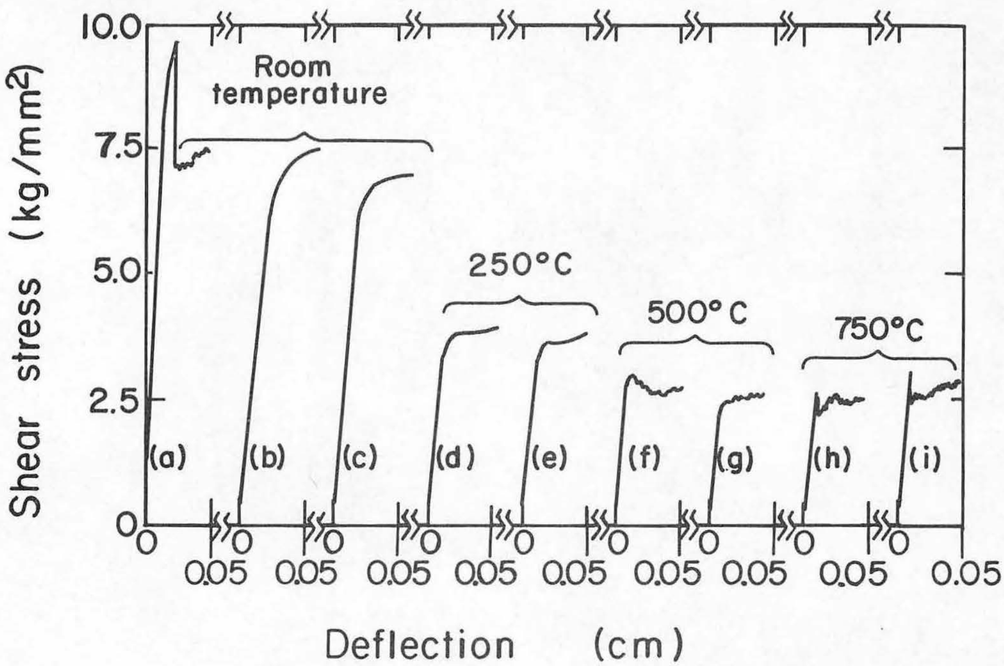
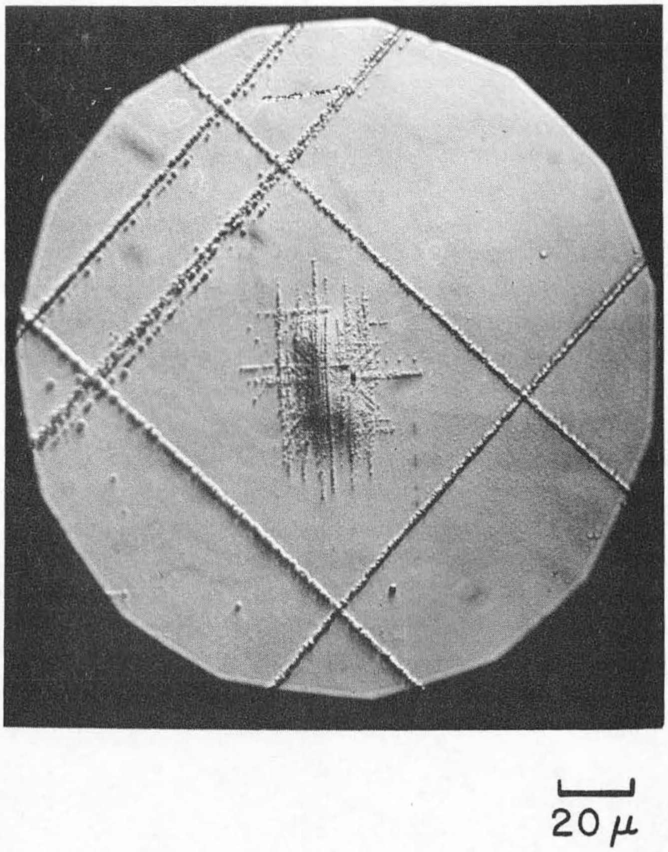


10.0
7.5
5.0
2.5
0
Shear stress (kg/mm²)
Room temperature
250°C
500° C
750°C
(a)
(b)
(c)
(d)
(e)
(f)
(g)
(h)
(i)
0
0.05
Deflection (cm)

MU-27555



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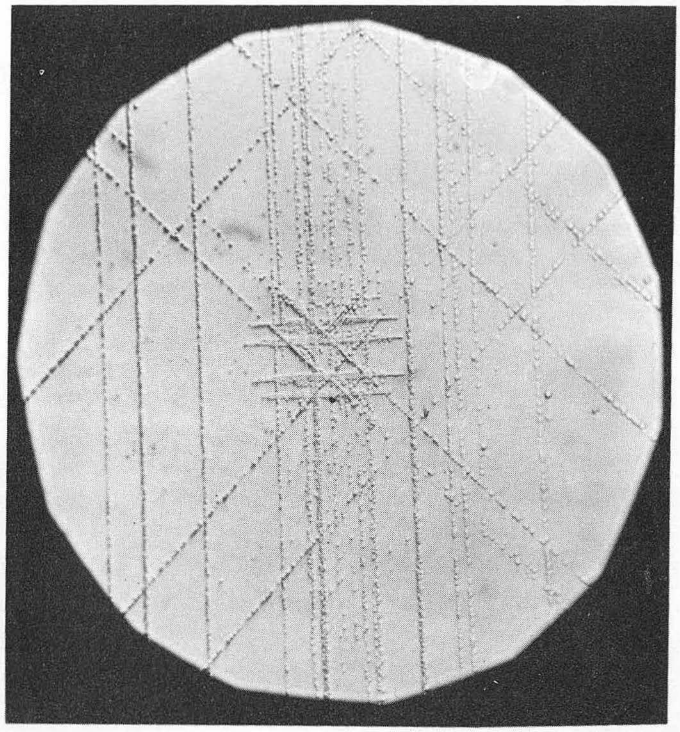

100μ

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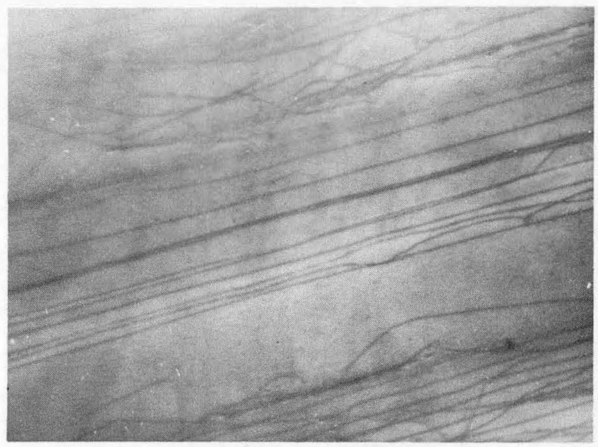

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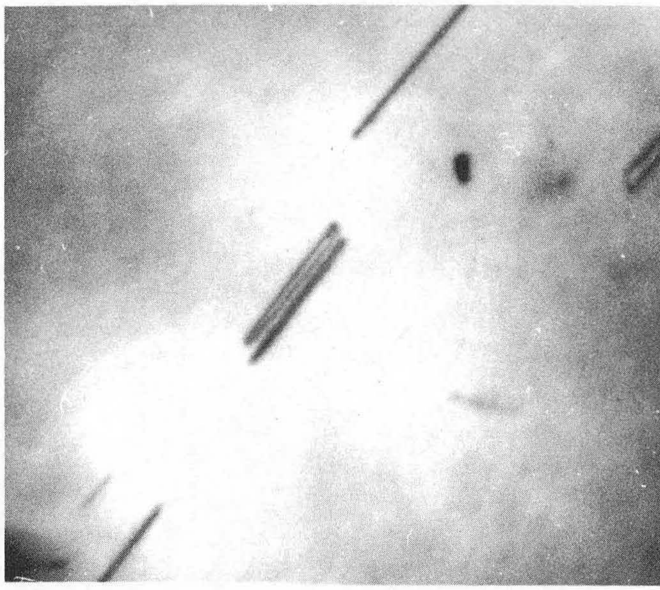

(a)

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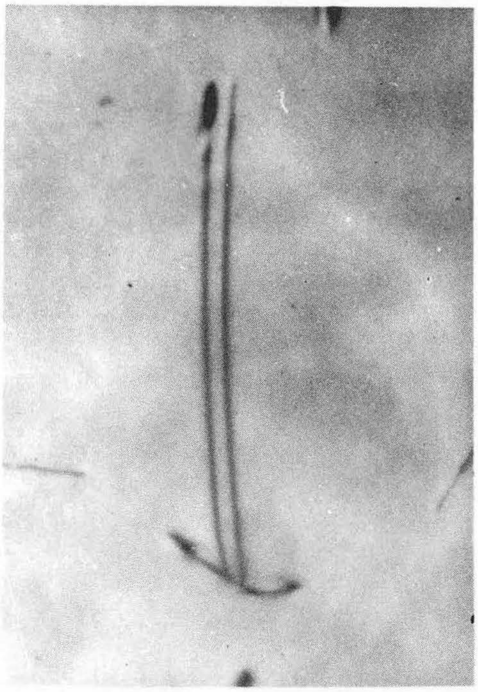
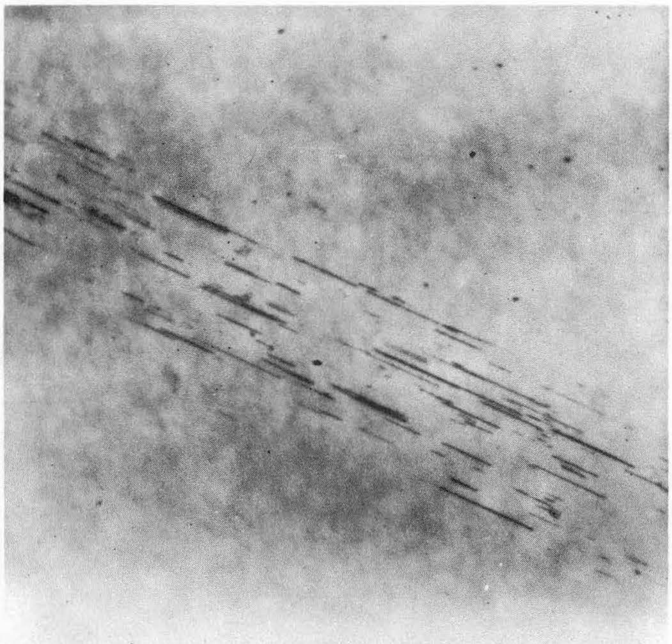
ZN-3208



(b)

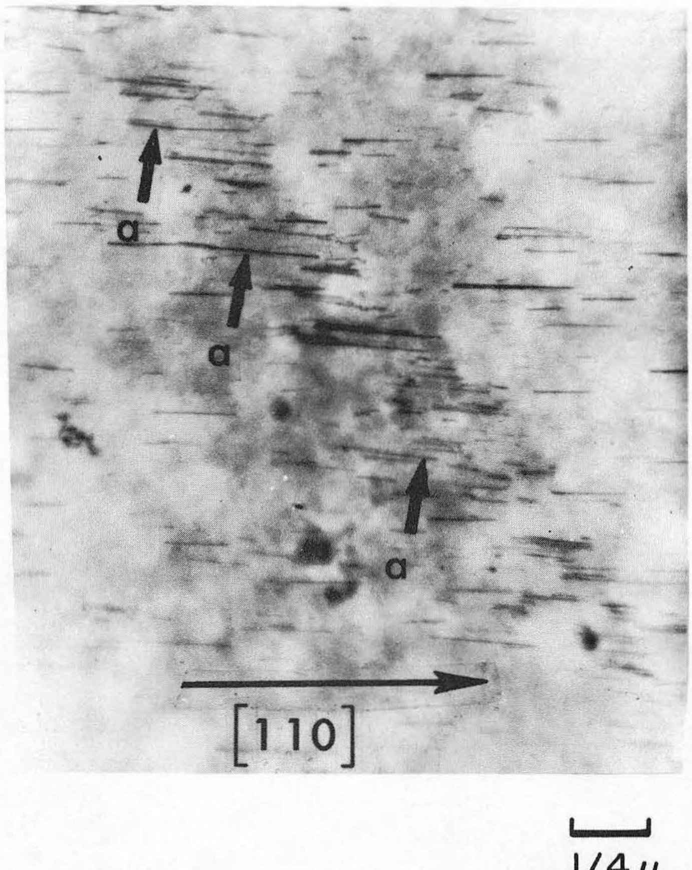
1/4 μ

ZN-3206



1/4 μ

ZN-3167



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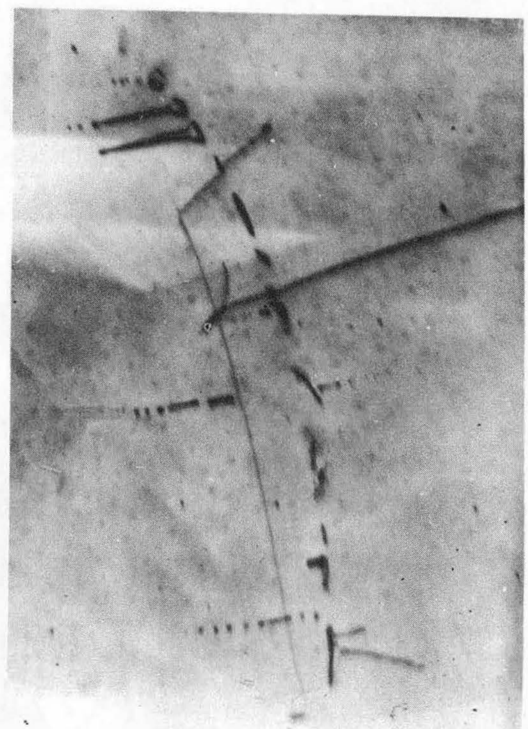

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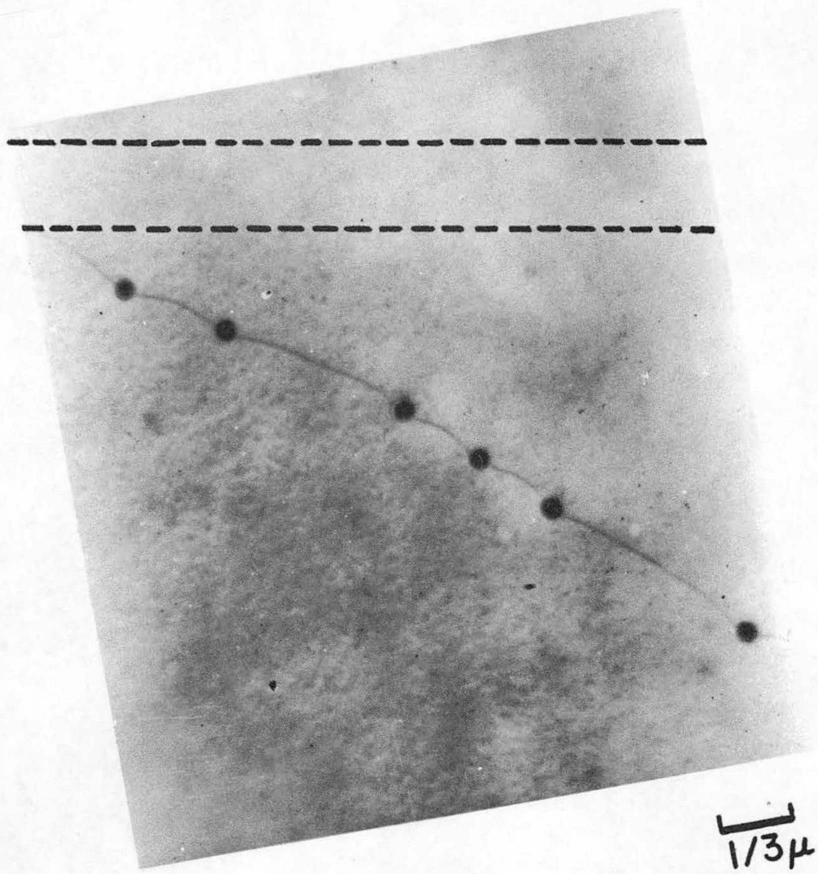

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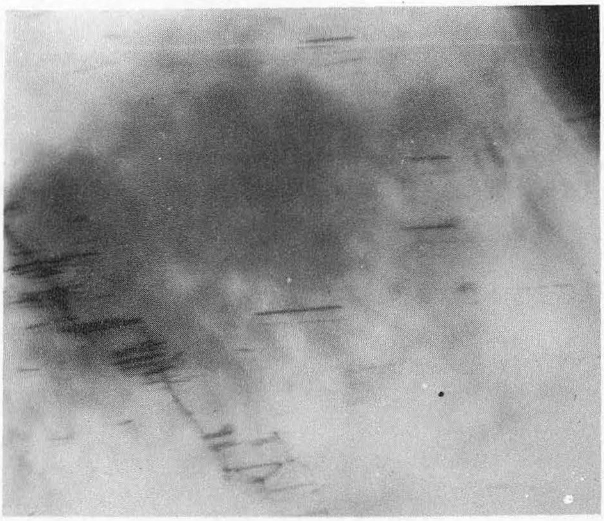

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