# Hydrostatic Pressure-Induced Deformation of Polycrystalline Zinc

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*Samples of polycrystalline zinc of 99.999+ pct purity were observed metallographically after having been exposed to hydrostatic pressures of up to 27 kbars. The deformation produced by this treatment was analyzed using X-ray and metallographic techniques. Basal-plane slip occurred universally in all grains examined at the lowest pressure used (8 kbars). At pressures of >23 kbars, $\{10\bar{1}2\}$ twin nucleation and growth were noted as well as $\{11\bar{2}2\}$ slip. Formation of bend planes, complex kink bands, and recrystallization at the grain boundaries were also observed in this pressure range. These observations are discussed in reference to the expected deformation behavior of zinc under high hydrostatic pressure.*

It has been shown over the past few years[1-3] that anisotropic, polycrystalline metals can be made to deform plastically by the application of a hydrostatic pressure. The effect is greater 1) the larger the anisotropy of linear compressibility and 2) the smaller the stress level needed to produce plastic deformation. Zinc having both very anisotropic linear compressibilities and a very low critical resolved shear stress (CRSS) for basal-plane slip has been shown to exhibit large amounts of plastic deformation when subjected to a hydrostatic pressure.[2,3] At pressures up to 15 kbars, grain boundary migration and slip on one set of glide planes have been reported.[3] After exposing samples to 20 kbars pressure, grain boundary migration, single slip, multiple slip, and twinning have been noted.[3] The identification of the deformation modes operative under these conditions has not been reported.

It was the purpose of this research to identify the deformation modes induced in polycrystalline zinc by the action of a hydrostatic stress and to study the strain accommodation at grain boundaries.

Hydrostatic pressure could affect the operational modes of deformation by altering the Peierls-Nabarro stress. This may be roughly approximated by the relationship[4]

$$\sigma_c \simeq \frac{2\mu}{K} \exp[-2\pi d/K\mathbf{b}] \qquad [1]$$

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where

$\sigma_c$ = the shear stress required for dislocation movement,

$K$ = 1 or $1 - \nu$ for edge and screw dislocations, respectively,

$\mu$ = the shear modulus,

$d$ = spacing between slip planes, and

b = Burgers vector.

For crystals having anisotropic linear compressibilities, the ratio $d/\mathrm{b}$ will vary with hydrostatic pressure. For zinc undergoing a pressure change from 1 bar to 30 kbars, a change in axial ratio, $\Delta(c/a) \simeq -0.056$,[5] can be expected. This leads to $\Delta(d/\mathrm{b})$ for basal plane slip of approximately $-0.028$. The latter can cause an increase of as much as 25 pct in the shear stress to move screw dislocations on the (0002) planes (not including that due to change in elastic modulus with pressure). The change in shear modulus with pressure has not been reported.

The ratio, $d/\mathrm{b}$, of other potential slip systems could increase with hydrostatic pressure. For instance, the $\{11\bar{2}2\}[\bar{1}\bar{1}23]$ slip system[6] which has been found to be operative in zinc under special stress conditions would show an increase, $\Delta(d/\mathrm{b}) \simeq +0.003$, which would lead to a 2.7 pct decrease in stress level for screw dislocation motion if a Peierls mechanism were operative for this system. The ratio, $\tau_{\mathrm{CRSS}\{11\bar{2}2\}}/\tau_{\mathrm{CRSS}\{0002\}}$, would then decrease from about $\frac{1300}{30}$ or $\sim 43$ at 1 atm[6,7] to $\sim 30$ at 30 kbars. Such a decrease might allow $\{11\bar{2}2\}[\bar{1}\bar{1}23]$ slip to play a more important role in the deformation of zinc and cadmium.

Hydrostatic pressure may also increase the stress levels for dislocation movement as a result of processes which lead to creation of volume because of additional work required for them to take place in a hydrostatic pressure environment or it may change the deformation process to one energetically more feasible.[8]

The application of hydrostatic pressure appears to prevent the nucleation and/or growth of cracks and in this manner provides a means by which large amounts of deformation can be sustained without fracture. Thus, in addition to effects associated with the high hydrostatic pressure, it provides an opportunity to study grain boundary accommodation in large-grained metals which are usually brittle at atmospheric pressure. The large grain size aids in the ease at which individual grains can be oriented. The disadvantage of the technique is the difficulty of distinguishing between the effects due to the high pressure and those due to large amounts of deformation.

## EXPERIMENTAL METHODS

Sample Preparation. Samples were prepared from Cominco Products grade 69 zinc, 99.999+ pct pure, containing 3 ppm Fe, <1 ppm Pb, 0.1 ppm Cd, <0.1 ppm Cu, 0.1 ppm Mg, and 0.1 ppm Si. Sections of the as-received zinc were machined into cylinders approximately $\frac{3}{8}$ to $\frac{1}{2}$ in. in diam and swaged with a 10:1 reduction in area; diameters of the swaged rod ranged from 0.122 to 0.162 in. The samples were then formed by filing into rods having an approximately square cross section 0.1 in. square and lengths ranging from approximately $\frac{5}{8}$ to $1\frac{1}{8}$ in. They were polished on two adjacent longitudinal surfaces through 600-grit paper and diamond-polished with a 6-$\mu$ paste. Final polishing was carried out on gammal cloth with aluminum oxide. Initially, the final polishing step was performed after a 400°C, 2-hr anneal which was intended to produce grains large enough for X-ray orientation. However, to insure that the samples would be as free of damage as possible, this procedure was altered so that the annealing procedure would be the last step. It was found, however, that even these samples had to be slightly repolished after annealing. Etching to reveal the grain structure was done in a 2 pct $HNO_3$-98 pct lactic acid (89.7 pct) solution for $\sim 15$ min.

The structure produced by this treatment consisted of large grains which usually occupied the entire cross section of the sample rods and which varied in length from $\sim 1$ to 3 mm. The grains were free of deformation markings except for the occasional occurrence of very light basal-slip traces or twins.

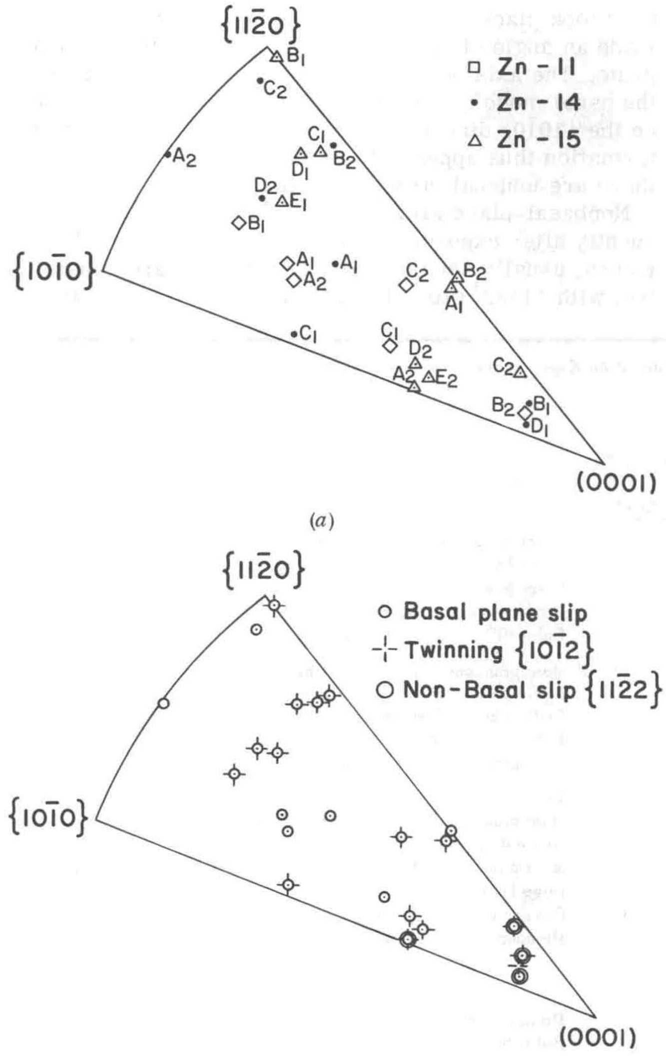
Pressurization was carried out in a 30-kbar apparatus manufactured by Harwood Engineering Co. and is similar to that previously described.[9] Pressure was measured by monitoring the electrical-resistance change of a manganin coil with a Foxboro Dynalogue recorder.

The calibration of the coil was checked in a device similar to that described by Bridgman[10] at the solidification pressure of mercury at 25.2° and 26.2°C (12.3 and 12.5 kbars)[11] and at the Bi I-Bi II transition pressure at 25°C (25.2 to 25.4 kbars)[12,13] by noting discontinuities in electrical resistance. The transformations were found to occur at pressures within $\sim 4$ pct of the accepted transition points.

After careful metallographic examination and photographic recording of the two polished samples, the orientation of individual grains was determined by the back-reflection Laue technique. The samples were then subjected to a series of increasing pressures. After each pressure step the pressure was released and the samples examined metallographically for evidence of deformation. Any changes were noted and photographs of the changed areas were usually taken. Pressurization rates ranged from approximately 200 to 500 bars a min; whereas, depressurization rates varied from approximately 500 to 1400 bars per min. Identification of the deformation mode was accomplished mainly by a two-surface stereographic technique or in the cases where traces were observed only on one surface by a one-surface technique. The Laue back-reflection technique was applied to the deformed samples, as well, to deduce lattice rotations from Laue asterism, to determine lattice misorientations across bend planes, and to confirm the identify of twin planes by a check of twin-matrix crystallographic relationships.

## RESULTS

Twelve grains in a total of three samples were analyzed. The grain orientations with respect to the surface normals and grain identification are summarized in the stereographic triangles of Fig. 1. It may be seen that a large spread of orientations is covered by this work.



(a)

(b)

Fig. 1—(*a*) Orientation and grain designation; (*b*) orientation dependence of deformation behavior of individual grains in polycrystalline zinc.

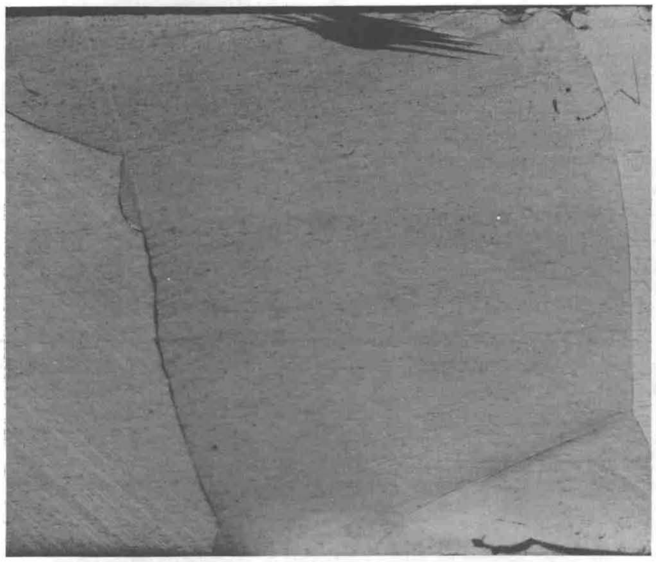


Fig. 2—Grain $C_1$ sample Zn-15 before pressurization. Polarized light; X48. Reduced approximately 35 pct for reproduction.

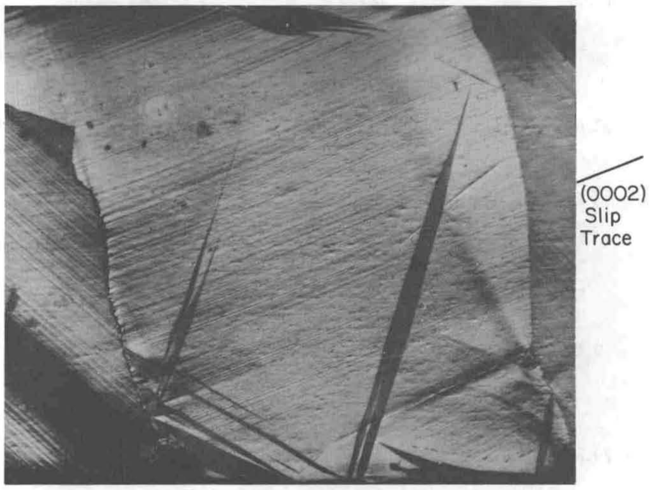


Fig. 3—Grain $C_1$ sample Zn-15 after exposure to 26.91 kbars hydrostatic pressure. Polarized light; X48. Reduced approximately 43 pct for reproduction.

The results of surface observations are summarized in Table I. In general, up to >12 kbars, deformation is restricted to glide on a single set of slip planes and is generally concentrated near the grain boundaries; this is in general agreement with the work of Davidson *et al.*[3] This set of slip planes has been identified as the (0002).

As the pressure increases, basal-plane slip becomes more pronounced, and the nucleation of a large number of twins is also observed. These have been identified as {10$\bar{1}$2} by two-surface trace analyses and the twin-matrix relationships confirmed by analysis of back-reflection Laue X-ray pictures of regions embracing a twin and matrix. In addition, the deformation becomes more inhomogeneous with the formation of bend planes and in one case a pronounced kink band. Evidence of recrystallization is also noted in the neighborhood of some of the grain boundaries. These effects are illustrated in Figs. 2 through 6. Fig. 2 shows grain $C_1$ of sample Zn-15 before being subjected to the hydrostatic pressure treatment. Minor amounts of basal-plane slip are seen, as well as a small twinned region. After subjecting the sample to a pressure of approximately 27 kbars, various types of deformation are encountered, Fig. 3. One notes four sets of {10$\bar{1}$2} twins, large amounts of basal-plane slip showing some curvature, and shaded regions illustrative of bend-plane formation. One may also note that the grain boundary on the left in Fig. 3 has changed somewhat from that shown in Fig. 2. This will be discussed below. Figs. 4 and 5 show grain $E_2$ of sample Zn-15 after exposure to a hydrostatic pressure of approximately 12 kbars and after being exposed to 27 kbars, respectively. It will be noted that the intensity of basal-plane slip has increased markedly with increasing pressure and that a sharp bend plane (denoted by a discontinuous change in contrast) has formed near the left center of the grain. In addition, other less intense bend planes have formed. A grain boundary to the right of Fig. 5 has become much coarser and on examination at higher magnification, Fig. 6 appears to be made up of small grains. Other grain boundaries in this sample have similar appearance after being exposed to 27-kbar pressure.

Brinson and Hargreaves[14] have also noted recrystallization in zinc at room temperature beneath hardness indentations.

The degree of misorientation across various bend planes was determined by a back-reflection Laue technique in which the beam was made to encompass the two misoriented grain portions. The misorientation was found to range from 1 to 2.5 deg in the small number of bend planes investigated. The misorientation across the sharp bend plane in Fig. 5, for instance, was 2.5 deg. The axis around which the bend took place could not be determined precisely but made an angle of between 20 and 30 deg with the basal plane. The axis of rotation that would be deduced from the usual models of bend-plane formation in zinc would be the $\langle 10\bar{1}0\rangle$ direction. The mechanism of bend-plane formation thus appears to be more complicated when there are unusual stress conditions.

Nonbasal-plane slip was observed relatively frequently after exposure to the higher pressures. The traces, usually seen only on one surface, are consistent with $\{11\bar{2}2\}$ slip. The appearance of these traces,

**Table I. Summary of Pressurization Experiments**

| Pressure, kbar | Rate of Pressure Increase, bars per min | Rate of Pressure Decrease, bars per min | Grain | Observations |
|---|---|---|---|---|
| | | | Sample no. Zn-11 | |
| 7.89 | 523 | 525 | $A$ | Faint basal-plane slip lines in $A_1^\dagger$ and $A_2$ |
| | | | $B$ | Faint basal-plane slip lines in $B_1$ |
| | | | $C$ | Faint basal-plane slip lines in $C_2$, more pronounced in $C_1$ |
| | | | $D_2^*$ | Pronounced slip markings |
| | | | $E^*$ | Faint slip lines in $E_1$, none in $E_2$ |
| 11.86 | 282 | 564 | $A$ | More pronounced basal slip lines in $A_1$ and $A_2$ |
| | | | $B$ | Little change from observations at lower pressure |
| | | | $C$ | Little change from observations at lower pressure |
| | | | $D_2^*$ | Pronounced slip markings |
| | | | $E^*$ | More pronounced slip markings in $E_1$ |
| 23.69 | 358 | 1390 | $A$ | Very pronounced basal slip lines |
| | | | $B$ | More pronounced basal slip lines; growth of existing $\{10\bar{1}2\}$ twin<br>Nucleation of new twins near grain boundary |
| | | | $C$ | More pronounced basal slip lines in $C_1$ and $C_2$; nucleation of large $\{10\bar{1}2\}$ twin in $C_2$ |
| | | | $D_2^*$ | Pronounced slip markings |
| | | | $E^*$ | Pronounced slip markings in $E_1$; twin nucleation in $E_2$ |
| | | | Sample no. Zn-14 | |
| 9.87 | 235 | 658 | $A$ | Pronounced basal slip traces in $A_1$ and $A_2$ |
| | | | $B$ | Faint basal-plane slip traces in $B_1$; bend-plane activity in $B_1$ |
| | | | $C$ | Basal-plane slip traces in $C_1$ and $C_2$ |
| | | | $D$ | Faint basal-plane slip traces in $D_1$, none in $D_2$ |
| 24.78 | 263 | – | $A$ | Very pronounced basal-plane slip traces in $A_1$ and $A_2$ |
| | | | $B$ | Pronounced basal-plane slip in $B_1$ and $B_2$; large amount of $\{10\bar{1}2\}$ twinning in $B_1$; nonbasal slip traces in $B_1$, probably $\{11\bar{2}2\}$ |
| | | | $C$ | More pronounced basal-plane slip in $C_1$ and $C_2$; nucleation of $\{10\bar{1}2\}$ twin in $C_1$; growth of existing twin in $C_2$ |
| | | | $D$ | Pronounced basal-plane slip lines in $D_1$ and $D_2$; bend-plane activity in $D_1$; evidence of duplex slip consistent with $\{11\bar{2}2\}$ |
| | | | Sample no. Zn-15 | |
| 11.86 | 348 | 592 | $A$ | Fairly pronounced basal-plane slip in $A_1$ and $A_2$ |
| | | | $B$ | Fairly pronounced basal-plane slip in $B_1$ and $B_2$ |
| | | | $C$ | Small amount of basal slip lines in $C_1$ and $C_2$ |
| | | | $D$ | Faint basal-plane slip traces in $D_1$ and $D_2$ |
| | | | $E$ | Faint basal-plane slip traces in $E_1$ and $E_2$ near grain boundary |
| 26.91 | 532 | 666 | $A$ | Extensive amount of basal-plane slip; large amount of twinning and bend-plane fomation; presence of nonbasal slip in $A_2$, probably $\{11\bar{2}2\}$ |
| | | | $B$ | Large amount of basal-plane slip in $B_1$ and $B_2$; large kink formation accompanied by necking; rotations of the order of 15 deg involved |
| | | | $C$ | Pronounced basal slip traces in $C_1$, less pronounced in $C_2$; large amount of $\{10\bar{1}2\}$ twinning and very pronounced bend-plane activity |
| | | | $D$ | Very pronounced basal-plane slip lines in $D_1$ and $D_2$; small amount of $\{10\bar{1}2\}$ twinning in $D_1$ and $D_2$; bend-plane fomation in $D_1$ and $D_2$ |
| | | | $E$ | Very pronounced basal slip traces in both $E_1$ and $E_2$; extensive bend-plane activity and $\{10\bar{1}2\}$ twin formation |

*Orientation of grain not determined.

†Subscripts denote surfaces observed.

Fig. 4—Grain $E_2$ sample Zn-15 after exposure to 11.86 kbars. Polarized light; X48. Reduced approximately 47 pct for reproduction.

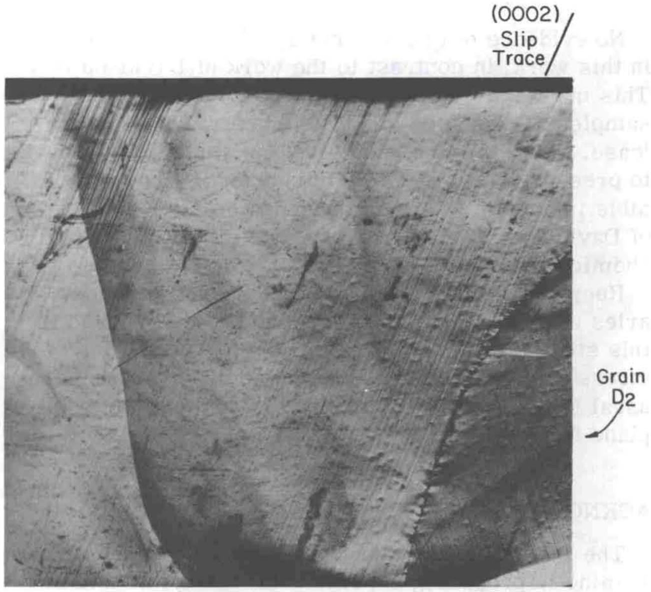


Fig. 5—Grain $E_2$ sample Zn-15 after exposure to 26.91 kbars. Polarized light; X48. Reduced approximately 43 pct for reproduction.

as has previously been noted,[15] is rather indistinct, Fig. 7, but they are more readily seen with the aid of phase-contrast metallography as shown in Fig. 8. {11$\bar{2}$2} slip was seen in almost all grains whose surface normals were close to the ⟨0001⟩ direction as is illustrated in Fig. 1. In these orientations the intensity of basal-slip lines is relatively small and so would not be expected to interfere with the viewing of other slip lines. It is more than likely that {11$\bar{2}$2} slip has taken place in many more of the grains, but interference by basal-plane slip traces has prevented general observation of the pyramidal-plane slip traces.

Other more complex deformation behavior has been observed in these experiments. Fig. 9 illustrates one such region, grain $B_1$ of sample Zn-15, after exposure to ~27 kbars. The central region located approximately halfway between the grain boundaries (not shown) has been reduced in dimension substantially, whereas the dimensions of the region near the left and right of the photograph, close to the grain boundaries, have remained unchanged. The basal planes whose traces form a kinklike pattern are nearly perpendicular to the plane of view. To ascertain the orientation shifts involved in the different regions of the grain, Laue back-reflection X-ray patterns were taken in the various numbered regions and compared to the original orientation. In Region 1 the orientation change involves a 15-deg rotation about a ⟨10$\bar{1}$0⟩ direction lying in the plane of view and parallel to the basal slip trace. In Region 2 the major orientation change can be described by a rotation of 15 deg around a normal to the

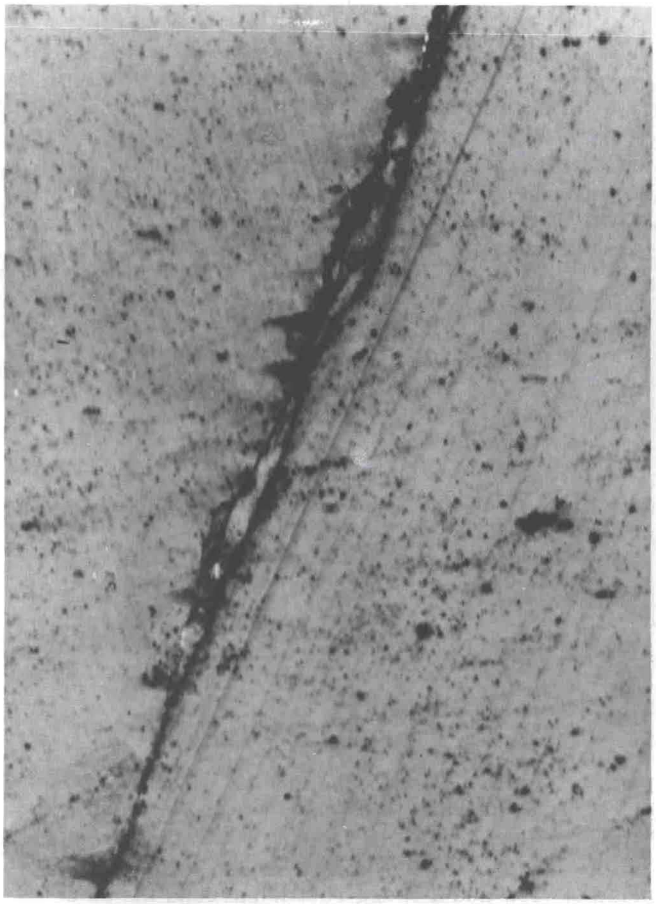


Fig. 6—Grain boundary between grains $D_2$ and $E_2$ sample Zn-15 after exposure to 26.91 kbars. Polarized light; X600. Reduced approximately 18 pct for reproduction.

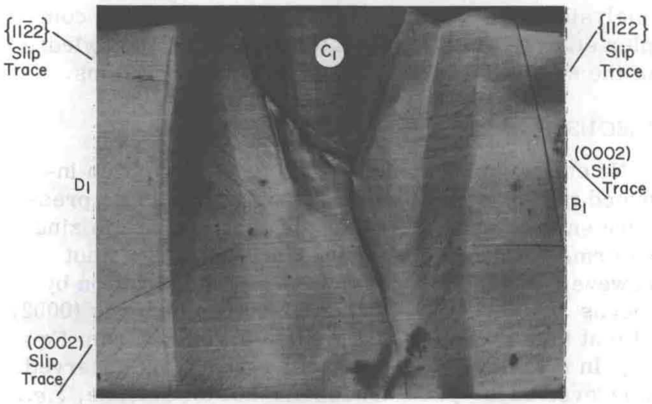


Fig. 7—Grains $B_1$, $C_1$, and $D_1$ sample Zn-14 after exposure to 24.78 kbars. Polarized light; X48. Reduced approximately 53 pct for reproduction.

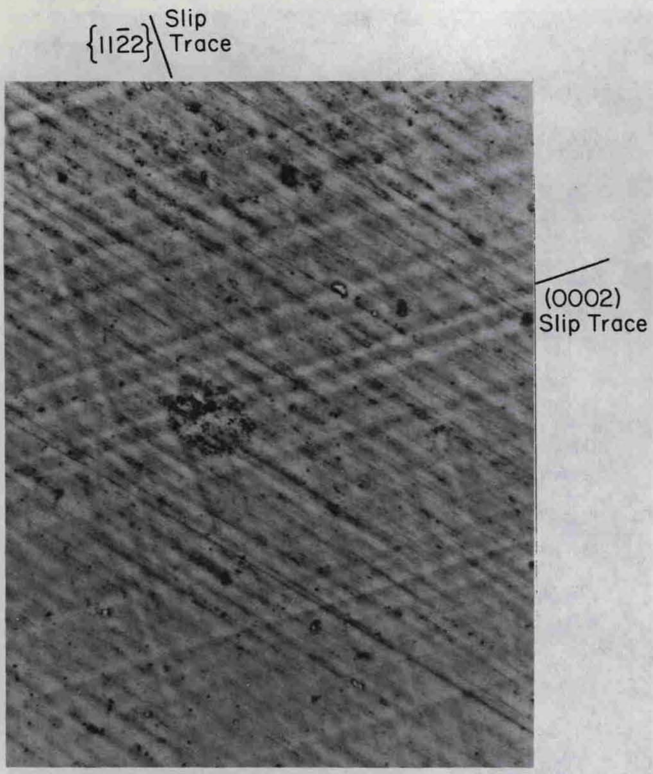


Fig. 8—Grain $D_1$ sample Zn-14 after exposure to 24.78 kbars. Phase contrast; X937. Other traces are polishing scratches. Reduced approximately 34 pct for reproduction.

plane of view and a 5-deg rotation around the same axis as in Region 1. The orientation of Region 3 is essentially unchanged from its original one; however, the Laue pattern shows a large amount of asterism demonstrating the inhomogeneous deformation. The analysis of the mechanism of formation of such complex structures is complicated by a lack of knowledge of the stresses induced by the neighboring grains.

## DISCUSSION

No entirely new deformation modes have been induced in zinc by exposure to a high hydrostatic pressure environment. All have been seen before in zinc deformed at atmospheric pressure. This does not however rule out the possibility that deformation by means of $\{11\bar{2}2\}$ slip is made easier relative to (0002) slip at high pressure as would be predicted from Eq. [1]. In fact, the evidence that $\{11\bar{2}2\}$ slip is observed whenever basal-plane traces are not too intense, *i.e.*, when the normal to the observed surface is close to $\langle 0001 \rangle$, gives some support to this hypothesis, see Fig. 1(*b*).

The observations are not at variance with Pugh's idea that the enhanced ductility of zinc under high pressure is based on a critical tensile stress criterion for fracture.[16] The application of a hydrostatic pressure could thus prevent the nucleation and/or growth of cracks and allow the shear-stress level to become higher. This in turn might allow more systems to enter into the deformation process. Measurements on single crystals of the stress levels to activate the different deformation processes should provide an answer as to which of the aforementioned processes is more important.

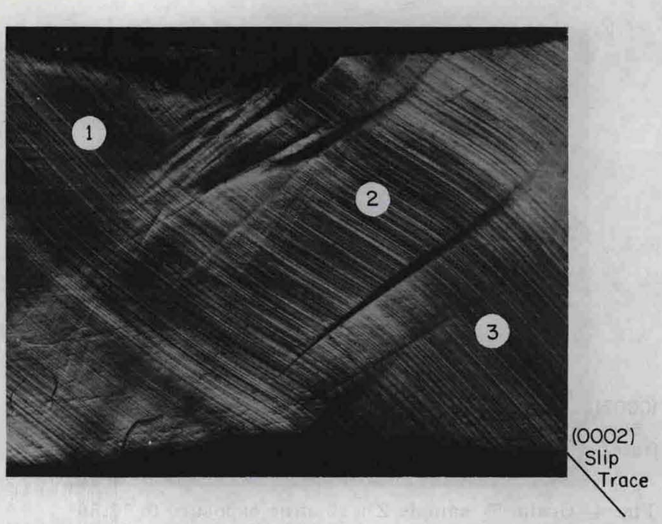


Fig. 9—Grain $B_1$ sample Zn-15 after exposure to 26.91 kbars. Polarized light; X48. Reduced approximately 45 pct for reproduction.

No evidence of grain boundary sliding was detected in this work, in contrast to the work of Davidson *et al.*[3] This may be due to the fact that Davidson held his samples at pressure for 30 min before pressure release, whereas the samples in this study were brought to pressure and immediately released. Another possible important factor is the much smaller grain size of Davidson's samples or possible differences in chemical composition.

Recrystallization near some of the grain boundaries after exposure to the highest pressure used in this study (27 kbars) offers means for relaxation of stress in polycrystalline zinc in addition to the more usual (0002), $\{11\bar{2}2\}$ slip, $\{10\bar{1}2\}$ twinning, and bend-plane formation.

## ACKNOWLEDGMENT

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