Hydrostatic Pressure-Induced Deformation of Polycrystalline Zinc

S. H. Gelles

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]

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

S. H. GELLES, Member AIME, is Physical Metallurgist, Ledgemont Laboratory, Kennecott Copper Corp., Lexington, Mass.

Manuscript submitted December 13, 1965. IMD

where

σ_c = the shear stress required for dislocation movement,

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

μ = the shear modulus,

d = spacing between slip planes, and

b = Burgers vector.

For crystals having anisotropic linear compressibilities, the ratio d/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/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 ~43 at 1 atm[6,7] to ~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-μ 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 ~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 ~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 ~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.