

Hydrostatic Pressure-Induced Plastic Flow in Polycrystalline Metals

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The effects of hydrostatic pressures to 26 kbars on the microstructure of polycrystalline Cd, Zn, Bi, Sn, Zr, Mg, Cu, and Fe were examined. Pressure-induced microscopic plastic flow in the form of boundary migration, slip, multiple slip, or twinning has been observed either singly or in combination in cadmium, zinc, bismuth, and tin. No deformation was observed in zirconium, magnesium, copper, and iron. The occurrence of such deformation, in terms of its intensity and initiation pressure, relates directly to the degree of anisotropy in the linear compressibility. Pressure cycling does not significantly affect the residual tensile properties of a zinc alloy which exhibits pressure-induced deformation similar to that of pure zinc.

CLASSICAL elastic theory predicts that a superimposed hydrostatic pressure will not induce shear stresses in an ideal material. Thus, in the case of

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homogeneous and isotropic materials plastic flow will not occur as a result of pressure exposure, regardless of the magnitude of the pressure, unless some form of phase transformation or permanent density change takes place. However, real materials, such as metals, often vary considerably from the condition of homogeneity and isotropy. For this reason, Vu and Johannin¹ examined and observed hydrostatic pressure-induced microscopic deformation in polycrystalline cadmium and zinc, but not in aluminum, to pressures of 9 kbars and indicated that its occurrence is related to anisotropy in the linear compressibility. In addition, Davidson and Homan² have reported pressure-induced deformation in polycrystalline bismuth below the I-II transformation pressure. It becomes apparent then that under certain conditions hydrostatic pressure can induce localized internal shear stresses in some metals of sufficient magnitude to cause plastic flow.

In this work, eight pure single-phase metals of different lattice structures and degrees of elastic anisotropy were examined after pressure exposures of up to 26 kbars in order to establish the conditions under which such deformation occurs and the type of deformation as a function of pressure. The effects of pressure cycling on the mechanical proper-

ties of a metal in which pressure-induced deformation occurs are also examined.

THEORY

Linear compressibility is defined as the relative change in length of a line per unit increase in hydrostatic pressure. The crystallographic relationships for the linear compressibility of cubic and noncubic structures are:

$$s_{11} + 2s_{12} \quad [1]$$

and

$$s_{11} + s_{12} + s_{13} - \gamma^2(s_{11} + s_{12} - s_{13} - s_{33}) \quad [2]$$

respectively, where the s 's are the elastic compliances and γ the direction cosine with respect to the "c" axis. In the case of the cubic metals, the relationship does not contain the γ term; thus the linear compressibility is independent of crystallographic direction and one has isotropy with respect to this property. As is evident from the latter relationship, depending upon how much the coefficient of γ differs from zero, noncubic crystals may exhibit high degrees of anisotropy in the linear compressibility.

Since there must be continuity of strain across the grain boundaries of a polycrystalline anisotropic material, exposure to hydrostatic pressure will result in shear stresses at the boundary due to the difference in orientation of adjacent grains. If the superimposed pressure were great enough and/or the grain boundary angle large enough so that the induced shear stresses exceed the critical value for some form of slip, twinning, or boundary migration, then one will observe plastic deformation. In the isotropic case, no discontinuity in the magnitude of the linear compressibility across grain boundaries exists; therefore, no shear stresses nor associated plastic flow will result from a superimposed pressure.

MATERIALS AND PROCEDURE

The nine polycrystalline metals utilized in this investigation are listed in Table I.

The linear-compressibility ratio shown in Table I represents the ratio of the linear compressibility in the "c" crystallographic direction (k_c) to that in the "a" direction (k_a) and its deviation from unity is a measure of the degree of anisotropy in this property.

With the exception of zirconium which was in the crystal-bar form, all the above materials were in the hot-worked condition.

In addition to the above samples, single crystals of cadmium and zinc were utilized to detect the presence of any nonhydrostatic stresses during pressurization which would be evident from the deformation of these soft single crystals.

The specimens, approximately 1/8-in.-diam by 1/4-in.-long half cylinders, were metallographically prepared prior to pressurization. The pressure

treatments consisted of half-hour exposures to hydrostatic pressures at various pressure levels for each material, as shown in Table II. Since the pressure-transmitting medium was a 1:1 pentane-isopentane mixture which remained fluid at high pressures, the prepared surface could be directly examined after pressurization without further polishing. However, a very light repolishing and etching was used when questions existed as to whether the deformation observed at the grain boundaries was due to boundary migration.

The pressure system utilized in the work has been previously described.²

In order to determine the residual effects on the mechanical properties resulting from the pressure cycling of an anisotropic polycrystalline metal, six zinc-alloy tensile specimens were pressurized to 20 kbars and, along with an equal number of as-received specimens, were subsequently tested in an Instron testing machine at a strain rate of 0.05 in. per min. The zinc alloy was selected on the basis of its being a single-phase alloy having a high degree of elastic anisotropy and a recrystallization temperature above room temperature.

RESULTS AND DISCUSSION

The results of pressure cycling of the eight pure metals, including additional data for bismuth beyond that previously reported, are summarized in Table II. As can be observed, the severity of the localized deformation varies widely for the materials investigated, ranging from none at the peak pressures to very severe at pressures below 5 kbars. Up to 26 kbars, no deformation was observed in the zirconium, magnesium, copper, and iron polycrystalline samples, nor in the zinc and cadmium single crystals, the latter indicating that hydrostatic pressures were being achieved. Of the metals investigated, only zinc, cadmium, bismuth, and tin exhibited plastic flow. In the case of cadmium, the deforma-

Table I

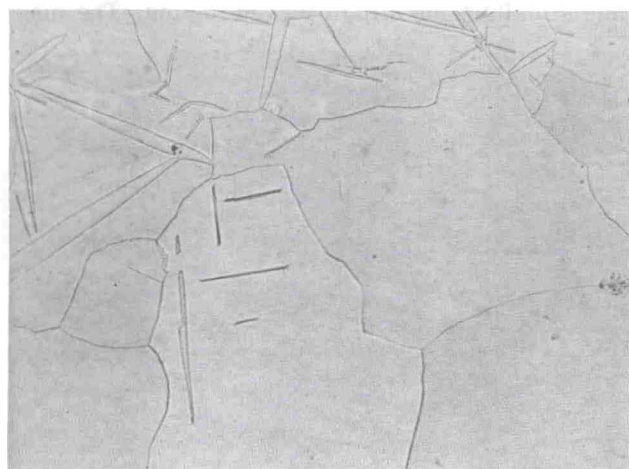
Material	Crystal Structure	Linear Compressibility Ratio, k_c/k_a^*	Purity, pct	Grain Size, mm per grain
Cadmium	Hcp	11.27	99.99	0.29
Zinc	Hcp	7.55	99.99	0.28
Zinc-0.005 Fe, 0.27 Cu	Hcp	7.55 (approx)	Alloy	0.06
Bismuth	Rhombohedral	2.43	99.99+	0.30
Tin	Tetrahedral	1.11	99.99	0.19
Zirconium	Hcp	0.86	99.9+	0.48
Magnesium	Hcp	1.04	99.8	0.12
Copper	Fcc	1.00	99.9	0.10
Iron	Bcc	1.00	99.8	0.04

*The elastic compliances used for computing k_c/k_a are given in Ref. 3 and 4.

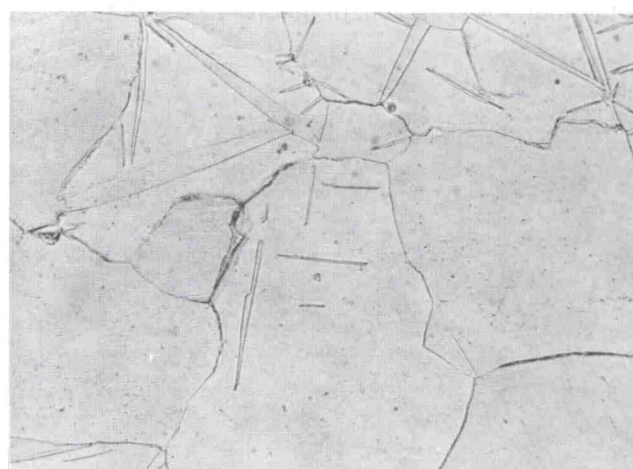
Table II. Pressure-Induced Deformations

Material	k_c/k_a	Pressure, kbars								
		1	2	3	4	5	10	15	20	26
Cadmium	11.27	GBM	GBM	GBM S T	GBM S T	GBM S T	GBM S T	GBM S T	GBM S T MS	GBM S T MS
Zinc	7.55	X	X	GBM S	GBM S	GBM S	GBM S	GBM S	GBM S T MS	—
Bismuth	2.43	X	X	GBM	GBM S	GBM S	GBM S MS	GBM S MS	GBM S MS	Transformation
Tin	1.11	—	—	—	—	X	X	X	X	GBM
Zirconium	0.86	—	—	—	—	—	—	—	X	X
Magnesium	1.04	—	—	—	—	—	—	—	X	X
Copper	1.00	—	—	—	—	—	—	—	X	X
Iron	1.00	—	—	—	—	—	—	—	X	X

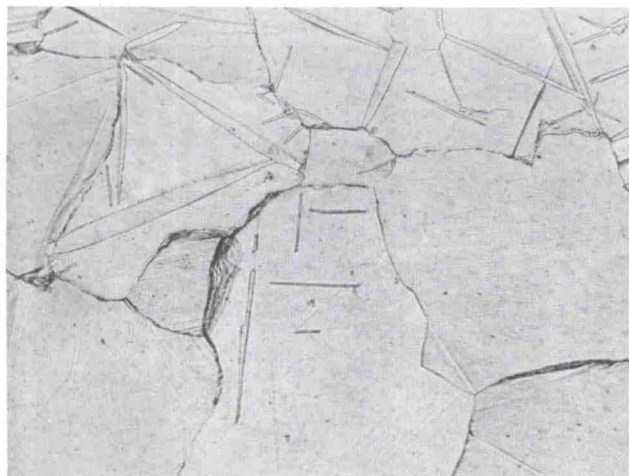
GBM = grain-boundary migration,
 S = slip,
 T = twinning,
 MS = multiple slip,
 X = no deformation.



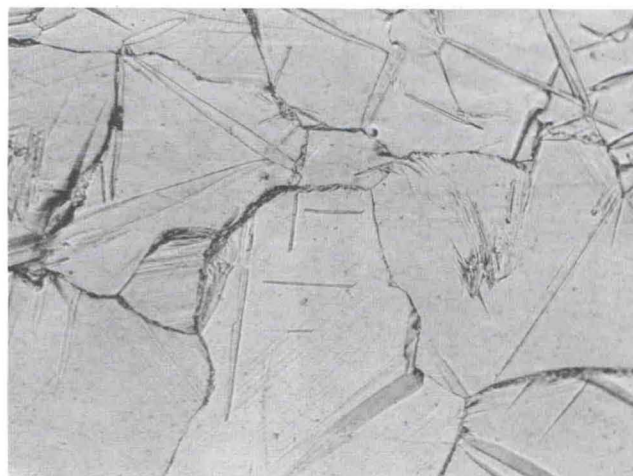
(a)



(b)



(c)



(d)

Fig. 1—Structural changes in polycrystalline cadmium induced by hydrostatic pressure. (a) Original structure; (b) after 4000 bars; (c) after 10,000 bars; (d) after 20,000 bars. X100. Reduced approximately 10 pct for reproduction.

tion, which was in the form of boundary migration, became detectable by optical microscopy after pressurizing to 1 kbar with a similar observation for zinc and bismuth at 3 kbars. In the case of tin, the first detectable flow, which was in the form of boundary migration, did not occur until pressures of 26 kbars.

It is apparent from Table II that the propensity to deform under a hydrostatic pressure is related directly to the degree of anisotropy in the linear compressibility. No deformation was observed in the case of copper and iron which are isotropic, nor in zirconium and magnesium which have quite low degrees of anisotropy. Tn, which required a pressure of 26 kbars to initiate plastic flow, is slightly anisotropic whereas bismuth, zinc, and cadmium, which exhibited severe deformation initiating at quite low pressures, are highly anisotropic. It is suspected that zirconium and magnesium would also exhibit pressure-induced plastic flow at some higher pressure than that possible in the equipment used in this experiment. However, due to the low degree of anisotropy and higher strength of magnesium and zirconium, the pressure to cause plastic flow would probably be quite high as compared to the four lower-strength metals in which flow was

observed. Vu⁵ found no deformation in aluminum, copper, and magnesium which had been pressurized to 9 kbars. In earlier work, Johannin and Vu⁶ did observe isolated regions of multiple slip in a single crystal of 24 pct Zn α brass. However, alloy segregation is suspected which could result in localized anisotropy of the linear compressibility. Thus, only unconstrained pure or chemically homogeneous single crystals and totally isotropic polycrystalline metals will not have internal shear stresses introduced by a superimposed hydrostatic pressure. In the case of even slightly anisotropic materials, internal shear stresses will occur and, if the pressure is high enough, microscopic plastic flow will result. This may be an important consideration when attempting to measure pressure effects on structure-sensitive properties of anisotropic polycrystalline materials.

The type of deformation observed in zinc, cadmium, tin, and, for comparison purposes from previous work, bismuth is shown in Figs. 1 to 4, respectively, and the approximate pressure at which each type was observed is summarized in Table II. In all cases, the initiation of deformation was principally in the form of boundary migration with some simultaneous slip observed in zinc. The mag-

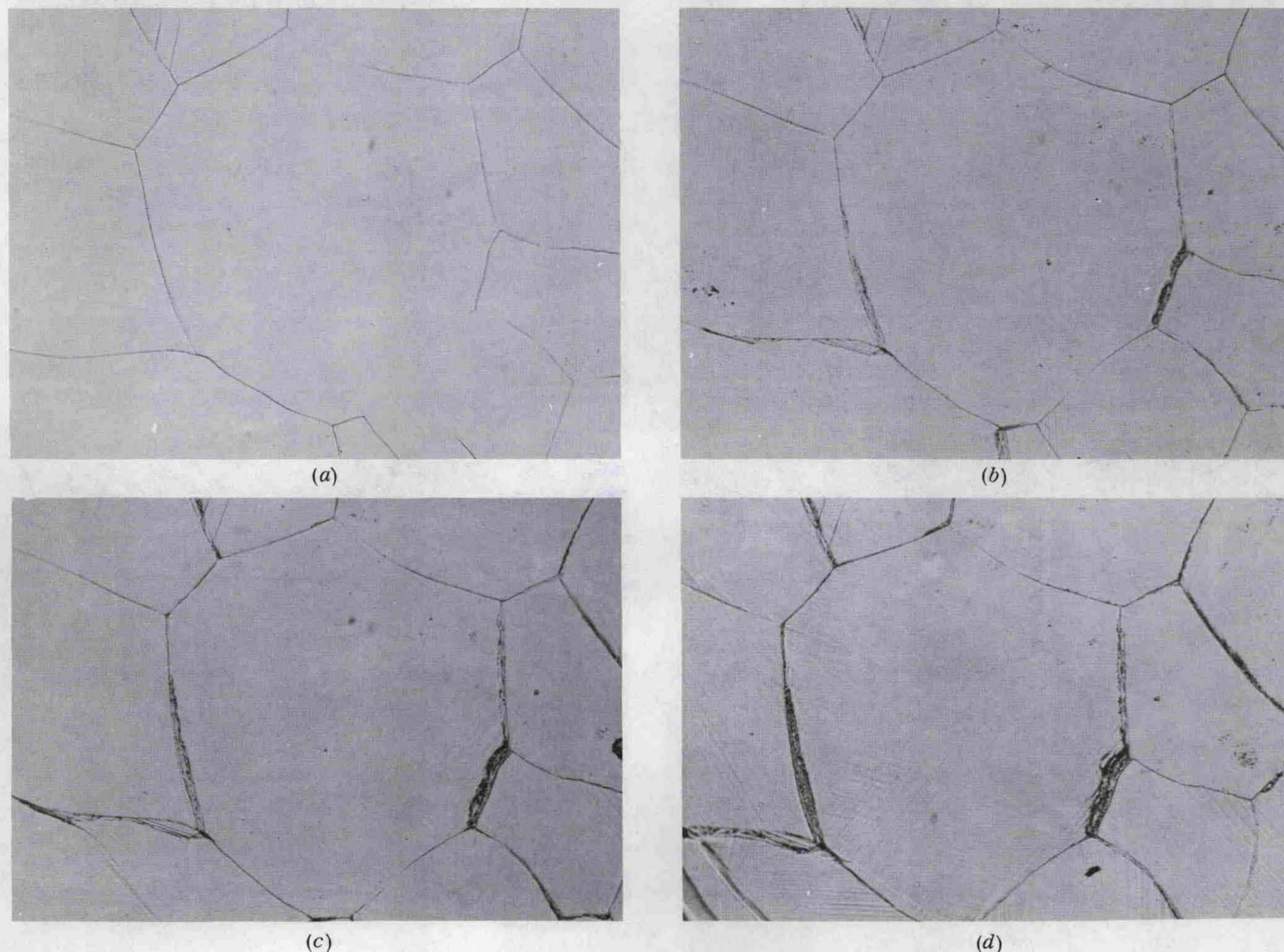
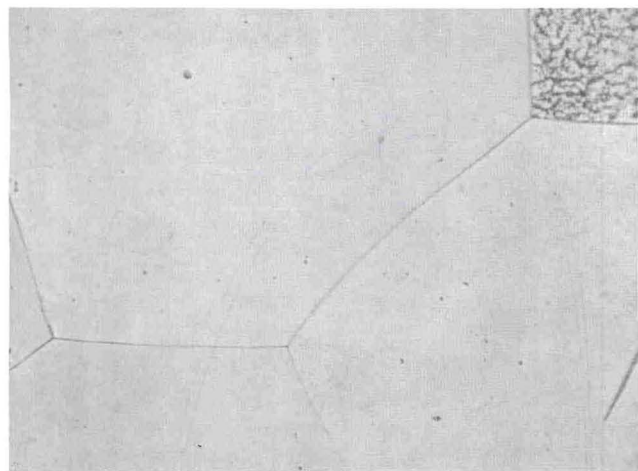
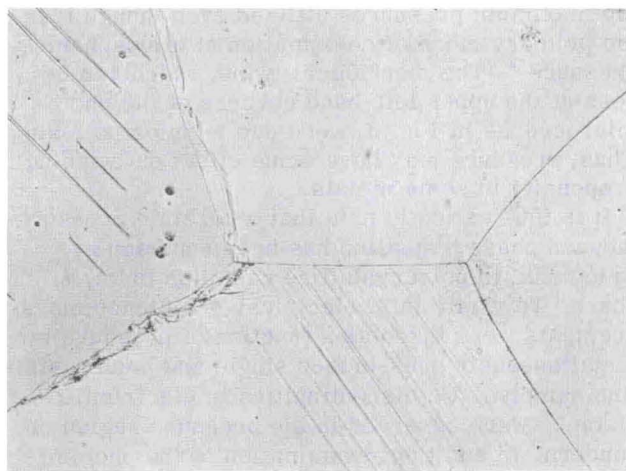


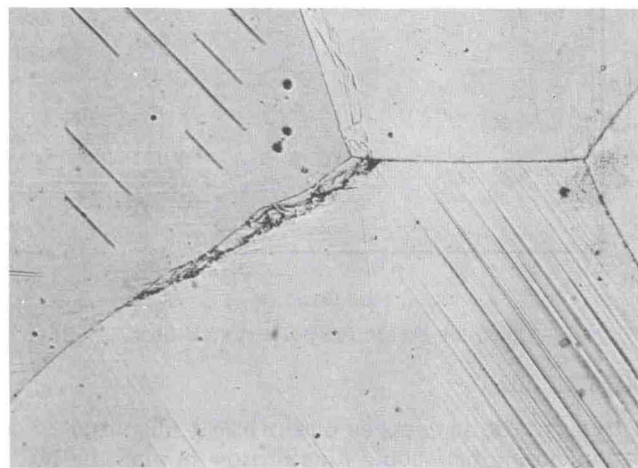
Fig. 2—Structural changes in polycrystalline zinc induced by hydrostatic pressure. (a) Original structure; (b) after 5000 bars; (c) after 10,000 bars; (d) after 20,000 bars. X100. Reduced approximately 10 pct for reproduction.



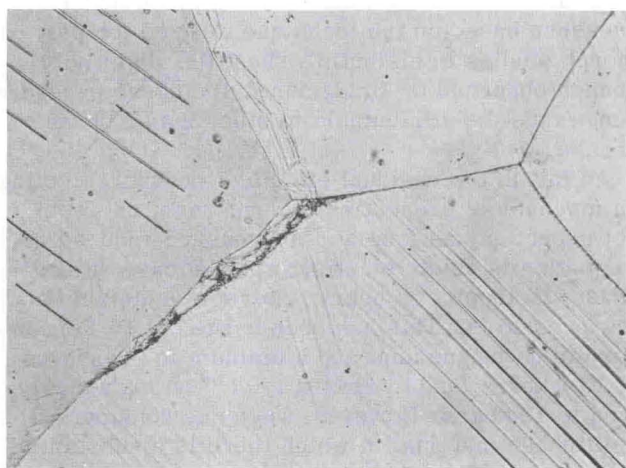
(a)



(b)

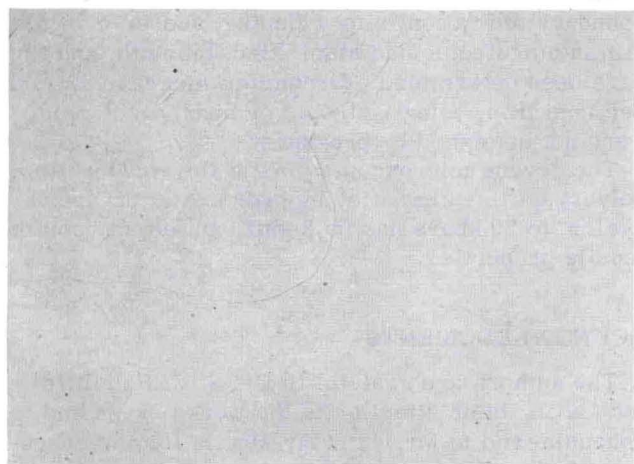


(c)

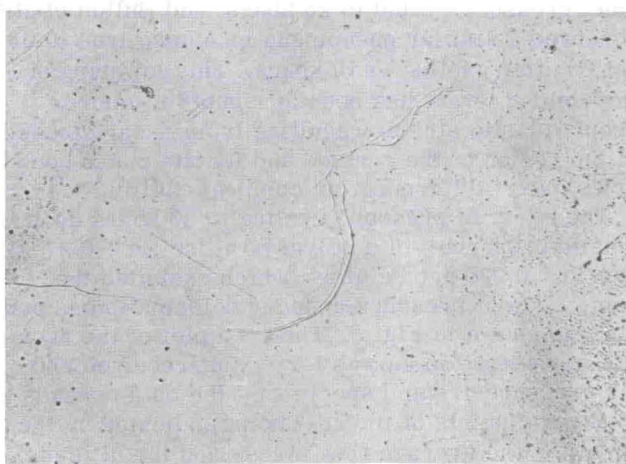


(d)

Fig. 3—Structural changes in polycrystalline bismuth induced by hydrostatic pressure. (a) Original structure; (b) after 10,000 bars; (c) after 15,000 bars; (d) after 20,000 bars. X100. Reduced approximately 10 pct for reproduction.



(a)



(b)

Fig. 4—Structural changes in polycrystalline tin induced by hydrostatic pressure. (a) Original structure; (b) after 26,000 bars. X100. Reduced approximately 10 pct for reproduction.

nitude of the boundary migration increased with increasing pressure in zinc, cadmium, and bismuth. In tin, deformation only initiated at 26 kbars and was also in the form of boundary migration. At increas-

ing pressure, slip, multiple slip, and twinning became quite evident in the case of zinc and cadmium. In contrast, it is interesting to note that in bismuth no mechanical twinning was observed even at

the maximum pressures utilized even though it is the primary mode of deformation at atmospheric pressure.² (The mechanical twins, which can be seen at the upper left-hand corners of the photomicrographs in Fig. 3, were due to prior polishing.) Thus, pressure may have some effect on twinning propensity in some metals.

It is interesting to note that a possible pressure-induced phase transition has been reported by Bridgman⁷ in polycrystalline cadmium below 7 kbars. To verify this, electrical-resistance measurements were taken as a function of pressure for simultaneously pressurized single and polycrystalline samples. No discontinuities in electrical resistance were observed in the pressure region of concern. In addition, examination of the photomicrographs for cadmium do not show any images indicative of a phase change taking place under pressure based on the technique utilized for polymorph studies in bismuth.⁸ Thus, the resistivity change observed by Bridgman in the above pressure range must be attributable to other than a phase change.

As far as the residual effects of pressure cycling on mechanical properties is concerned, one would not expect any as long as the pressure-induced deformation is solely elastic, as in the case of single phase, isotropic, or nearly isotropic materials at low pressures. This has been borne out by Ferron's⁹ results on magnesium and aluminum to pressures of 13.8 kbars, and by Bullen *et al.*¹⁰ on high-purity iron to 10 kbars. However, when one considers a multiphase material in which there is a substantial difference in the elastic properties of the various phases, or a polycrystalline anisotropic material, pressure cycling may have an effect. Radcliffe¹¹ found a loss in the sharp yield point and a substantial lowering of the yield strength in annealed mild steel pressure cycled to 25 kbars, and Bullen *et al.*¹⁰ observed a similar phenomena in Armco iron (0.03 pct C) after cycling to 10 kbars. This lowering of the yield strength has been attributed to microscopic plastic strains resulting from shear stresses in the region of the carbide and ferrite phase boundaries due to differences in compressibility.

The effect of pressure cycling to 20 kbars upon the tensile properties of a polycrystalline Zn + 0.27 pct Cu + 0.005 pct Fe alloy, which exhibited the same form of pressure-induced deformation as pure zinc, is shown in Fig. 5. This is a plot of the average stress-strain curves for six as-received and six pressure-treated specimens; the data spreads at several points of interest being indicated by the arrows. The average flow stress and the ultimate tensile stress have been slightly increased by pressure cycling. However, the data spreads overlap; thus the effects of pressure cycling upon tensile properties are considered insignificant. The data spread in the total elongation is much greater for the pressure-cycled specimens.

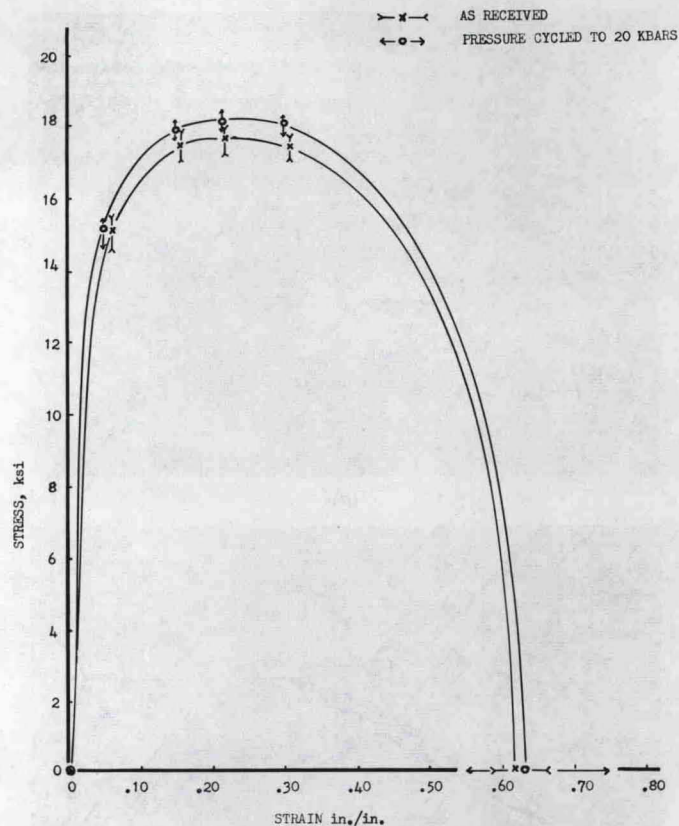


Fig. 5—Stress vs strain for polycrystalline zinc alloy.

CONCLUSIONS

Hydrostatic pressures of sufficient magnitude can induce microscopic plastic flow in elastically anisotropic polycrystalline metals but not in homogeneous single crystals or elastically isotropic polycrystals. The propensity to deform is related directly to the linear-compressibility ratio.

The approximate pressures for the onset of grain boundary migration, slip, twinning, and/or multiple slip in anisotropic cadmium, zinc, bismuth, and tin have been determined. Zirconium, magnesium, copper, and iron, being isotropic or nearly isotropic, were not deformed by pressure.

The severe microscopic plastic flow induced in a polycrystalline zinc alloy by hydrostatic pressure cycling to 20 kbars has no significant effect upon the tensile properties.

ACKNOWLEDGMENTS

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