Some Observations on the Effects of Hydrostatic Pressures to 20,000 Atm on the Structure of Polycrystalline Bismuth

by
T.E. Davidson
C.G. Homan

REPRINTED FROM

TRANSACTIONS

OF THE METALLURGICAL SOCIETY OF AIME
Vol. 227, No. 1, pp. 167-176 Feb. 1963

Some Observations on the Effects of Hydrostatic Pressures to 20,000 Atm on the Structure of Polycrystalline Bismuth

T. E. Davidson and C. G. Homan

This report deals with a study of the effects of extreme hydrostatic pressure on a polycrystalline material which exhibits a high degree of elastic anisotropy. Metallographically prepared polycrystalline bismuth samples were subjected to pressure levels up to 20,000 atm in a piston type gas-liquid system. The observed structural changes consisted of deformation localized along the grain boundaries, which appears to be a boundary migration phenomena, and widespread slip and cross slip. Subsequent deformation is progressive with increased pressure, and is substantially different in type from the deformation characteristic of uniaxial compression.

A large amount of interest has been shown in recent years in the effects of extreme pressure on metals. In this connection, many investigations into the effects of pressure on physical and mechanical properties and reactions have been undertaken. Due primarily to the experimental difficulties involved, there has not been a great deal of study of the structural changes resulting from exposure of metals to extreme pressure. It has been difficult, for instance, to produce a true hydrostatic stress state in many of the extreme pressure devices used, and also to directly examine specimens metallographically before and after pressurization. The results of a preliminary investigation into one of the effects of extreme pressure, and its associated true hydrostatic stress state, on the structure of metals are discussed herein.

Metals exhibit varied degrees of anisotropy in their physical and mechanical properties. Significant among the anisotropic properties is the modulus of elasticity, which varies by a factor of as much as three in some of the fcc metals, and by much larger factors in some of the less symmetrical elements. As a result of the variation of the modulus of elasticity with crystallographic direction, shear stresses will be induced in polycrystalline samples under true hydrostatic compression. Depending on several factors, these shear stresses could become of sufficient magnitude to cause localized plastic deformation.

Presented herein are some of the structural changes observed in polycrystalline bismuth as a result of exposure to hydrostatic pressures of up to 20,000 atm. Although several other materials in both bicrystal and polycrystalline form are also currently under investigation, bismuth was chosen initially, due to its low symmetry and moderately high degree of anisotropy in the elastic properties.

The fact that extreme hydrostatic pressure can induce structural changes in some elements in the polycrystalline state is of importance in itself. However, it may also be a factor in many of the other extreme pressure effects. For instance, this localized plastic deformation could substantially lower the recrystallization temperature, thus altering the effects of extreme pressure on this phenomenon. Also, as commented on by Bridgman,[1] the hysteresis in the polymorphic transition of polycrystalline bismuth under extreme pressures could possibly be attributed to structural changes brought about by unequal compressibility. Therefore, to accurately determine the true effects of extreme pressure on metallic substances, one first must determine if any structural changes due to hydrostatic compression are also simultaneously occurring.

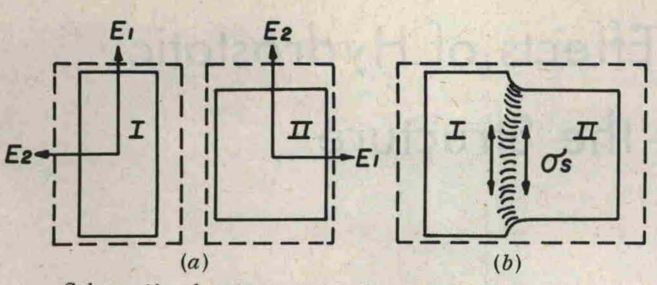
The purpose of this work has been to determine whether the shear stresses induced by hydrostatic pressure in an anisotropic polycrystalline material, such as bismuth, could reach a magnitude great enough to cause plastic deformation and to study the resultant structural changes and effects.

THEORY

In a metallic crystal, the elastic properties are dependent upon the crystallographic direction and plane. Depending upon the degree of anisotropy of the elastic properties and other factors, internal shear stresses of sufficient magnitude to cause localized plastic deformation may be induced in polycrystalline material by true hydrostatic compression. The following highly simplified two-dimensional model of two adjacent grains in a polycrystalline aggregate under hydrostatic pressure serves to schematically demonstrate how these shear stresses arise in an anisotropic material.

T. E. DAVIDSON and C. G. HOMAN are with the Research Div., Watervliet Arsenal, Watervliet, N. Y. This work was supported by the Ordnance Materials Research Office.

Manuscript submitted May 7, 1962. IMD



Schematic showing origin of internal shear stresses.

In (*a*), the approximate elastic dimensional changes of Grain I and II under hydrostatic compression are shown separately. For the purposes of the diagram, the maximum and minimum modulus of elasticity are shown to be normal, which, of course, is usually not the case, and E_1 is assumed to be much larger than E_2. (*b*) shows Grain I and II with a common boundary. Assuming the continuity of material across the boundary, the source of the shear stresses at the boundary due to accommodation is obvious.

The model shown is highly simplified as compared to the three dimensional case of a polycrystalline aggregate. From the two dimensional model, however, one can readily see how shear stresses can arise in the polycrystalline aggregate under hydrostatic pressure.

The magnitude of the induced shear stresses will depend on several factors. The stresses will be proportional to the applied hydrostatic pressure up to the point where plastic deformation occurs, and will be related to the degree of anisotropy of the material, the magnitude of the elastic modulus, and to the relative orientation between the adjoining grains of primary interest. As an example, for most cubic materials the maximum and minimum modulus of elasticity lie along the (111) and (100) directions respectively. Considering only two grains then, the maximum shear stress would occur when the (111) direction in one grain is parallel to the (100) direction in the adjoining grain.

Whether the induced shear stresses are of sufficient magnitude to cause localized plastic deformation will, of course, depend upon the lattice symmetry of the material and whether the critical resolved shear stress for slip or twinning has been exceeded. How all of these variables affect the magnitude of the shear stress and the amount of plastic deformation are subjects of other current investigations.

Based on the above considerations, hydrostatic pressure induced plastic deformation has a substantial probability of occurring in a material such as bismuth since it has low lattice symmetry (rhombohedral), a rather low modulus of elasticity and a reasonably high degree of anisotropy which is estimated to be a factor of 2.9 between the maximum and minimum elastic moduli based on the work of Wachtman[2] for rhombohedral corundum.

PROCEDURE

Specimen. The bismuth specimens used in this investigation were of 99.95 pct pure material which was obtained commercially in the as-cast lump form. For the actual specimens, this as-cast material was cast into 3/4-in.-diam, 1-in.-long cylinders, which were then extruded into 0.155-in.-diam rod. To enhance the actual extrusion process and soundness of the resultant material, the extrusion was performed at 220°C. The final specimens, which were 0.125 in. in diam and 0.200 in. long, were machined from the extruded rod.

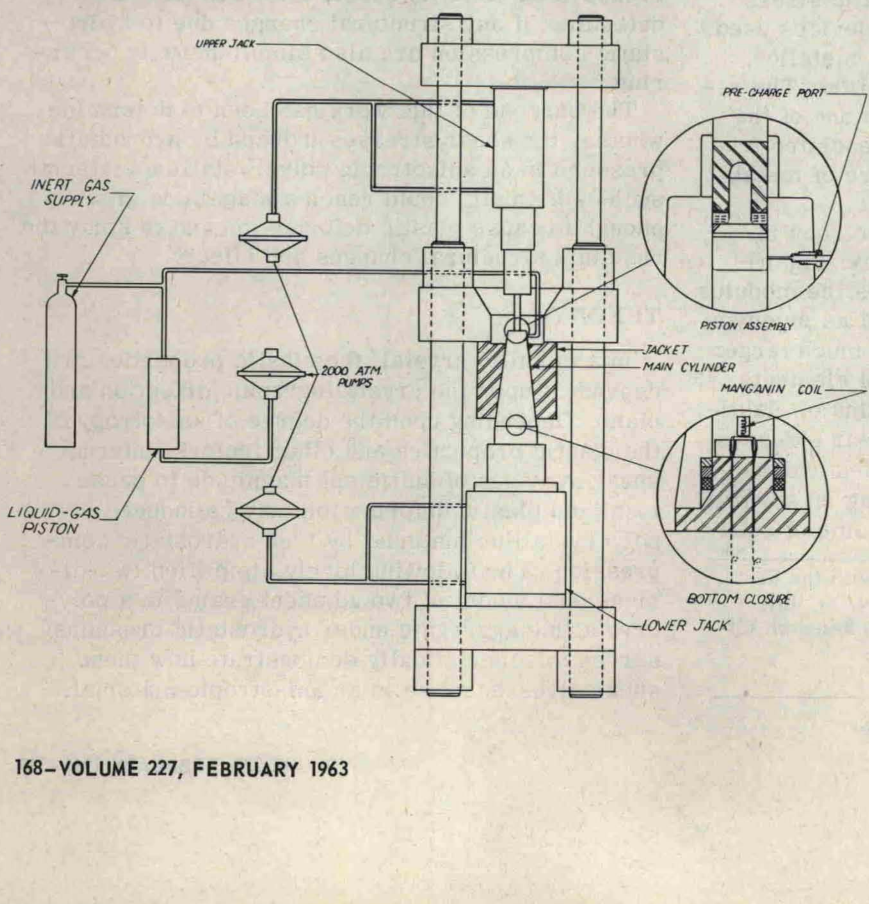
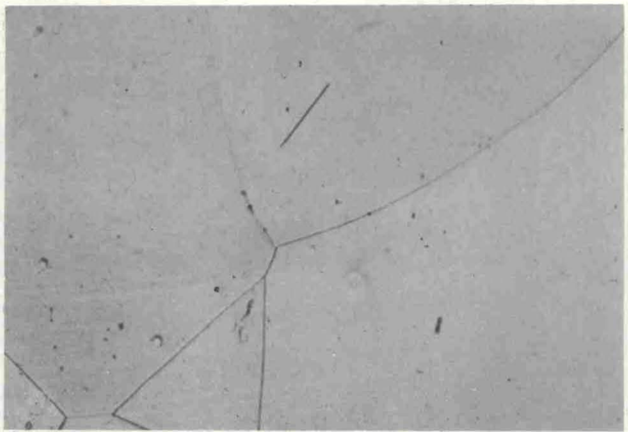
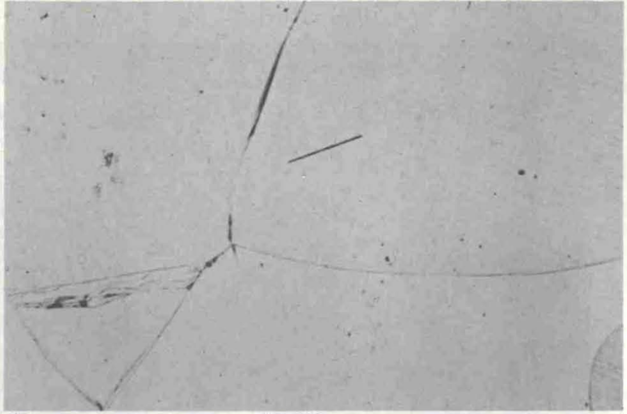


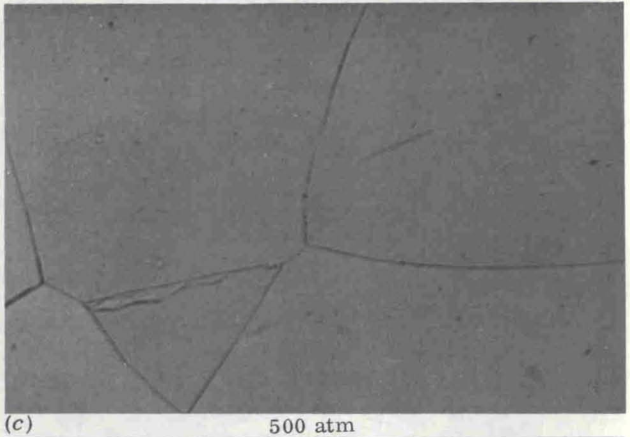
Fig. 1—Schematic of 30,000 atm pressure system.



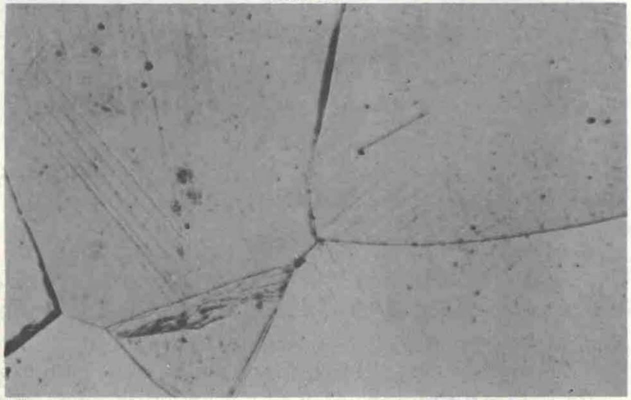
(a) 0 pressure



(b) 15,000 atm

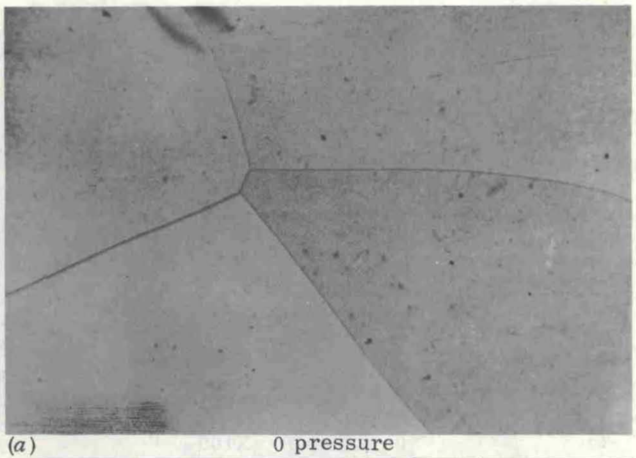


(c) 500 atm

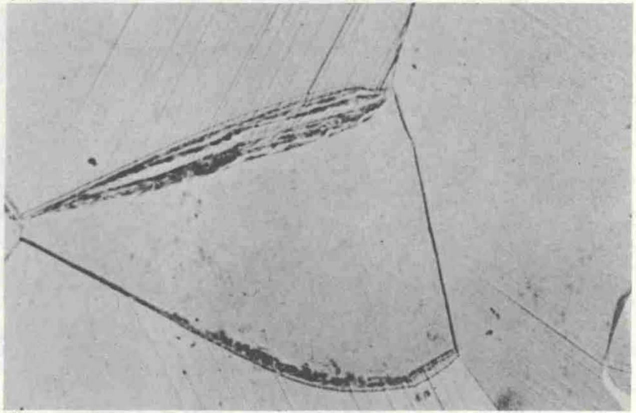


(d) 20,000 atm

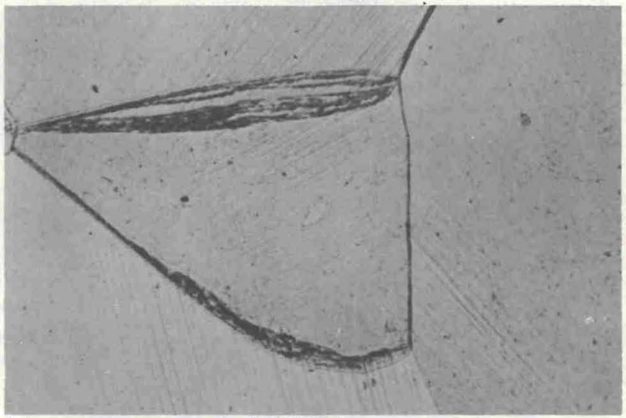
Fig. 2—Typical structural changes along one side of original grain boundary. X100. Enlarged approximately 2 pct for reproduction



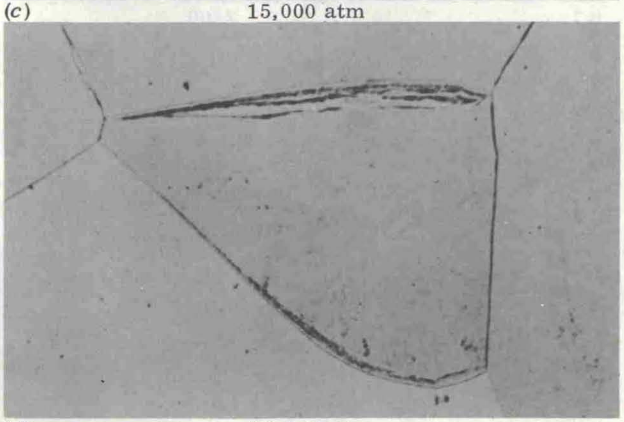
(a) 0 pressure



(b) 10,000 atm

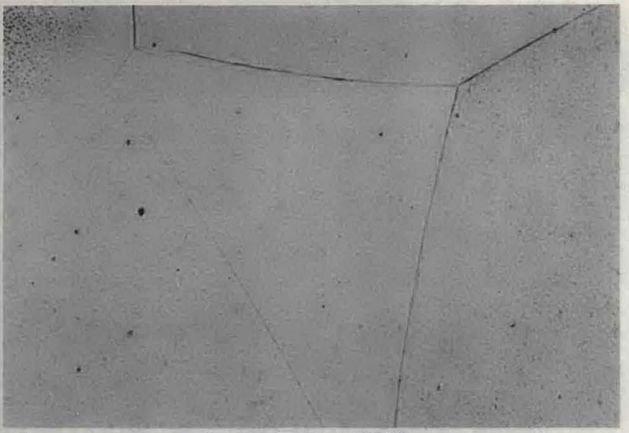


(c) 15,000 atm

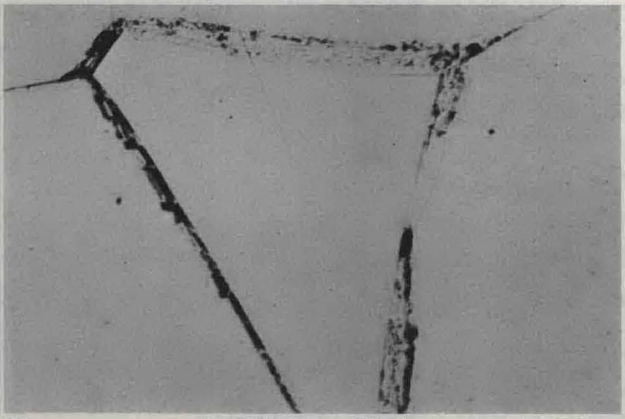


(d) 20,000 atm

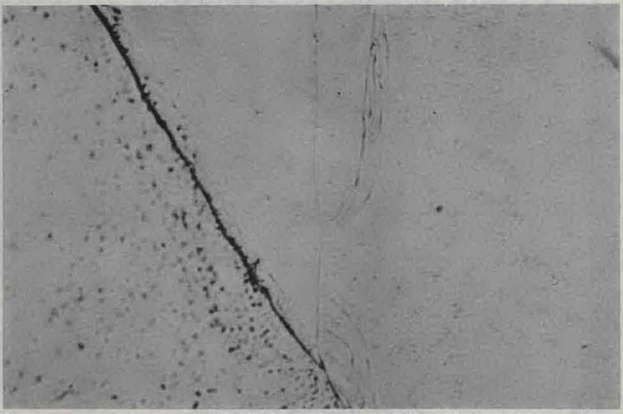
Fig. 3—Localized structural change adjacent to boundaries with slip continuing across original boundaries. X100. Enlarged approximately 9 pct for reproduction.



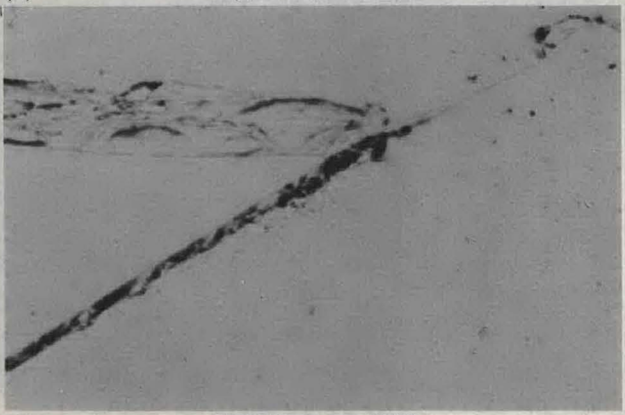
(*a*) 0 pressure X100



(*b*) 20,000 atm X100

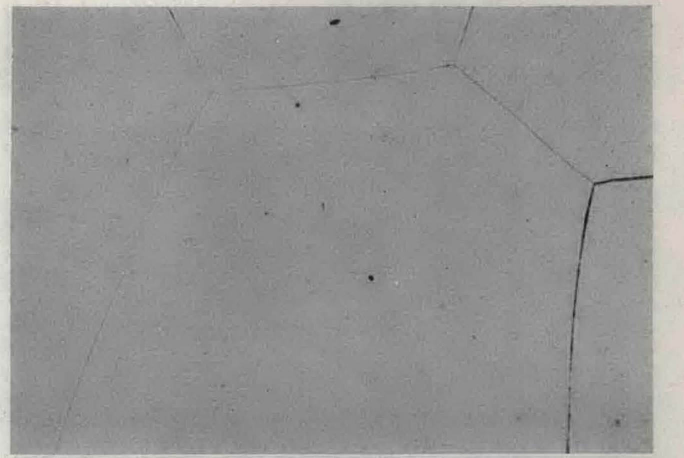


(*c*) 15,000 atm X400

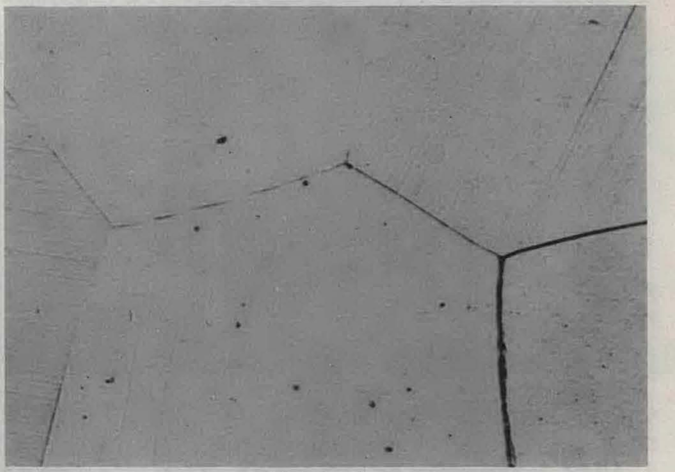


(*d*) 20,000 atm X400

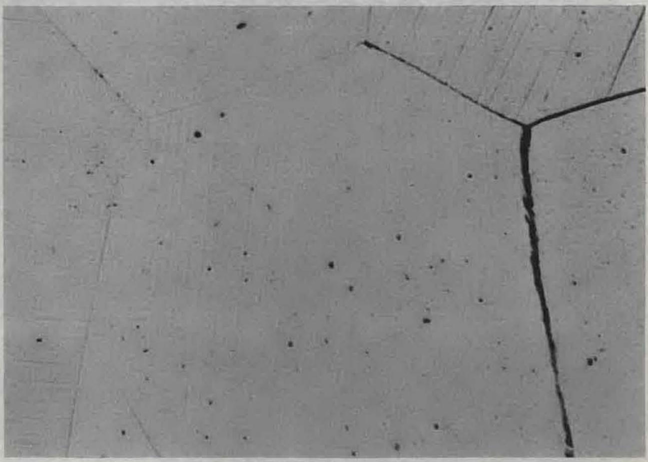
Fig. 4—Structural change along grain boundary surrounding entire grain. Enlarged approximately 2 pct for reproduction.



(*a*) 0 pressure



(*b*) 10,000 atm



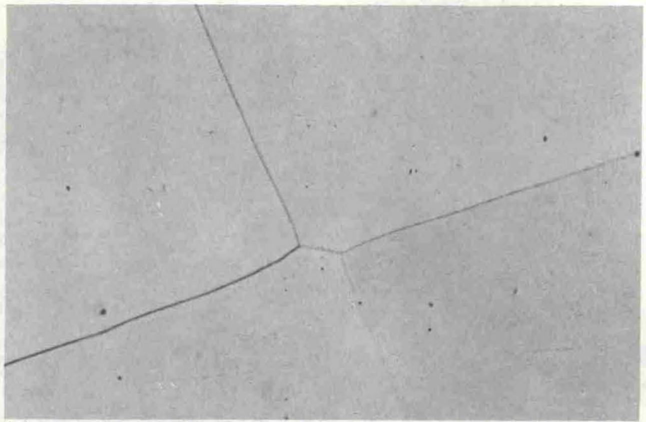
(*c*) 20,000 atm

Fig. 5—Generalized slip and multiple glide. X100. Enlarged approximately 4 pct for reproduction.

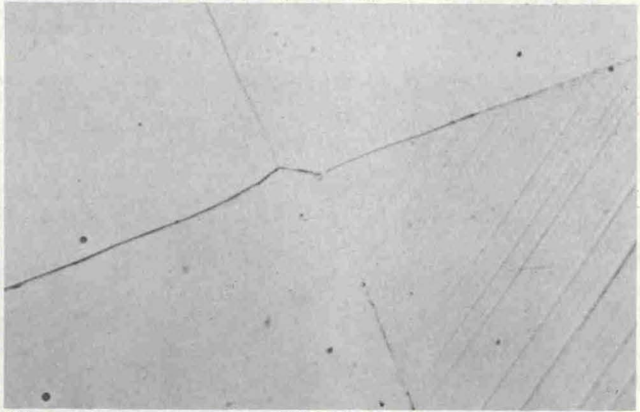
In order to obtain the large grain size, deemed desirable for this investigation, and also to remove any strains induced by machining and handling, the specimens were annealed at 260°C for 48 hr.

The single crystal specimens which were machined from 0.125-diameter, 4-in.-long crystals were of the same configuration as the polycrystalline samples.

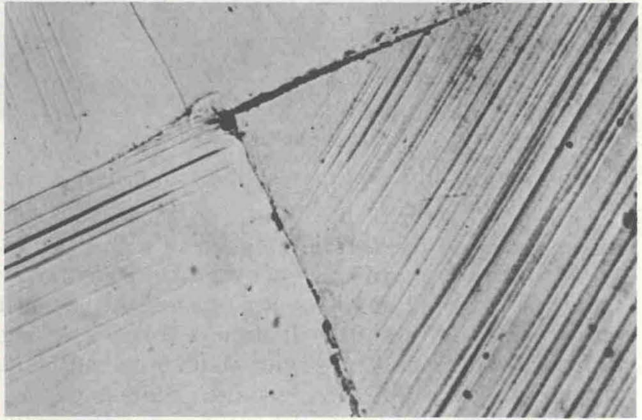
A plane parallel to the longitudinal axis of the specimen was polished and etched prior to pressuri-



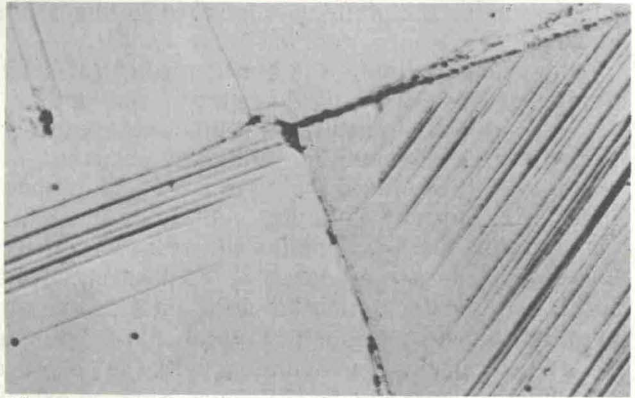
(a) 0 pressure



(b) 5000 atm

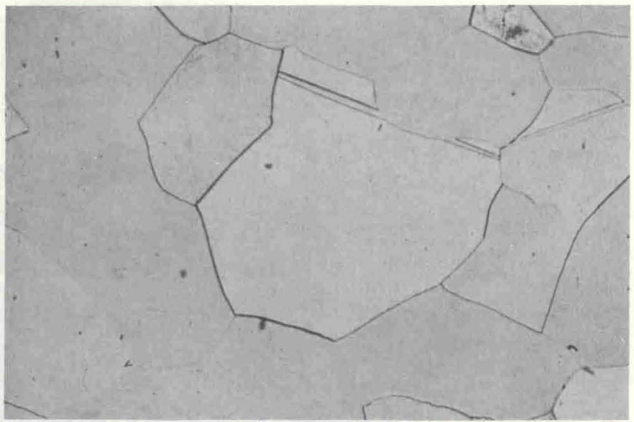


(c) 15,000 atm

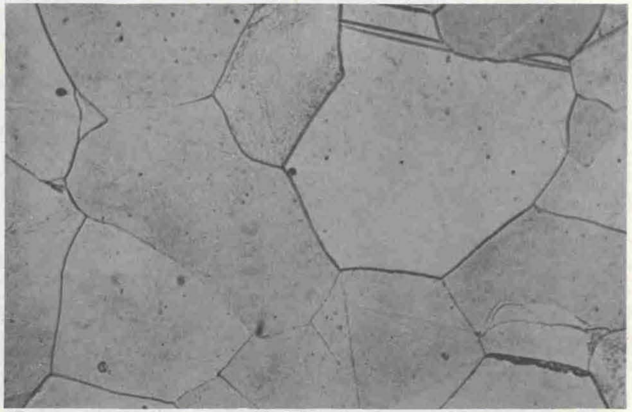


(d) 20,000 atm

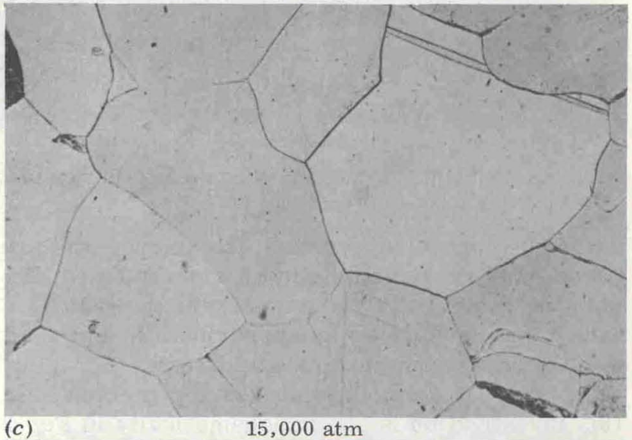
Fig. 6—Highly concentrated slip bands in conjunction with localized deformation along boundaries. X100. Enlarged approximately 4 pct for reproduction.



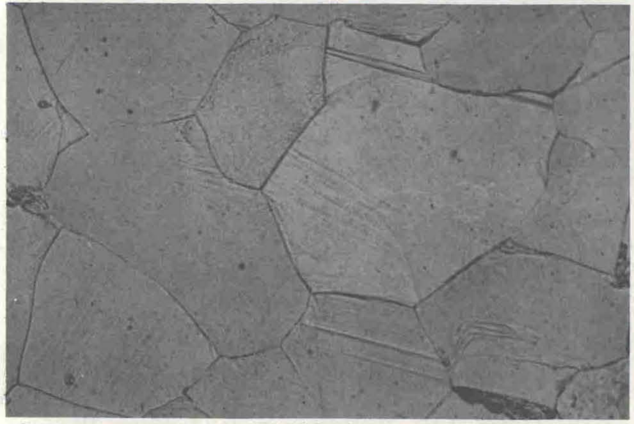
(a) 0 pressure



(b) 10,000 atm

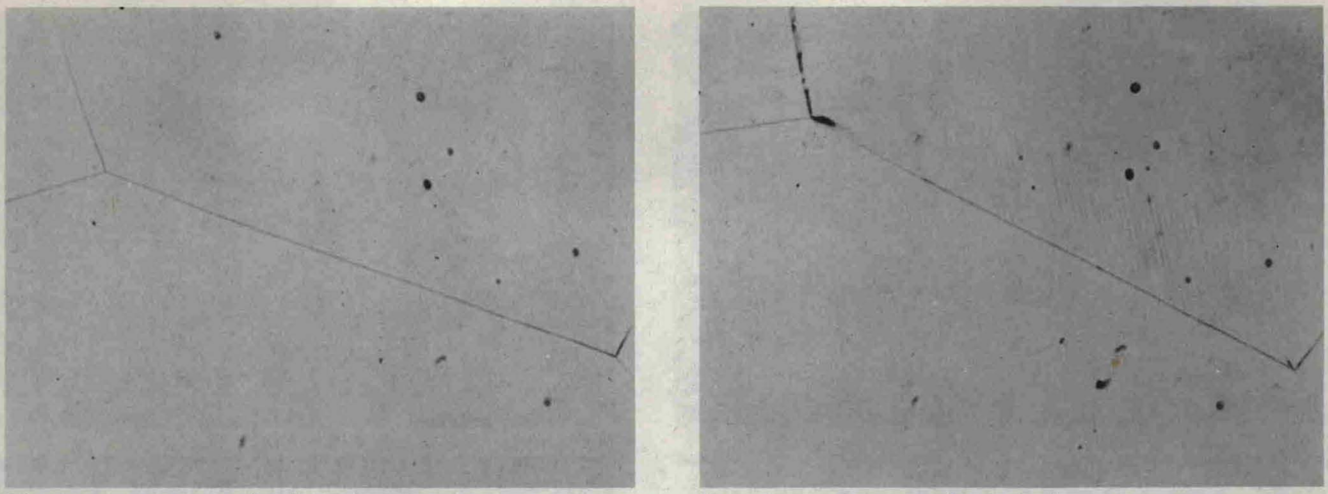


(c) 15,000 atm

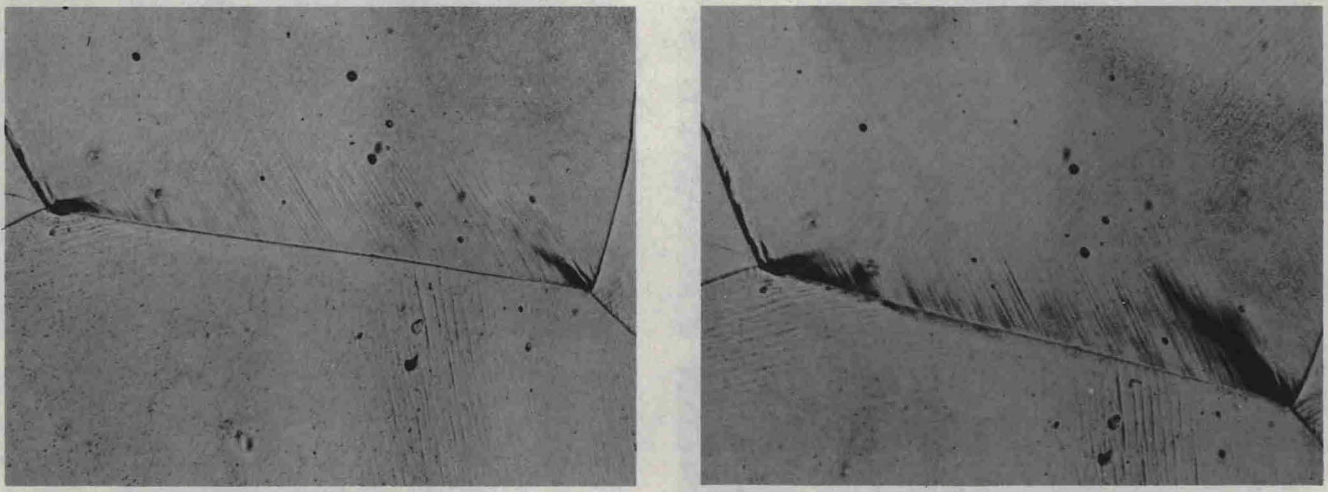


(d) 20,000 atm

Fig. 7—Typical structural change in as-extruded bismuth. X100. Enlarged approximately 2 pct for reproduction.



(*a*) 0 pct strain (*b*) 1 pct strain



(*c*) 3 pct strain (*d*) 5 pct strain

Fig. 8—Bismuth deformed in uniaxial compression showing preponderance of slip and cross-slip. X100. Enlarged approximately 4 pct for reproduction.

zation. To prevent distortion, the surface was prepared by electropolishing in a supersaturated KI solution with 1 pct by volume concentrated HCl. Added polishing time was approximately 4 min with a 6 v potential and moderate agitation.

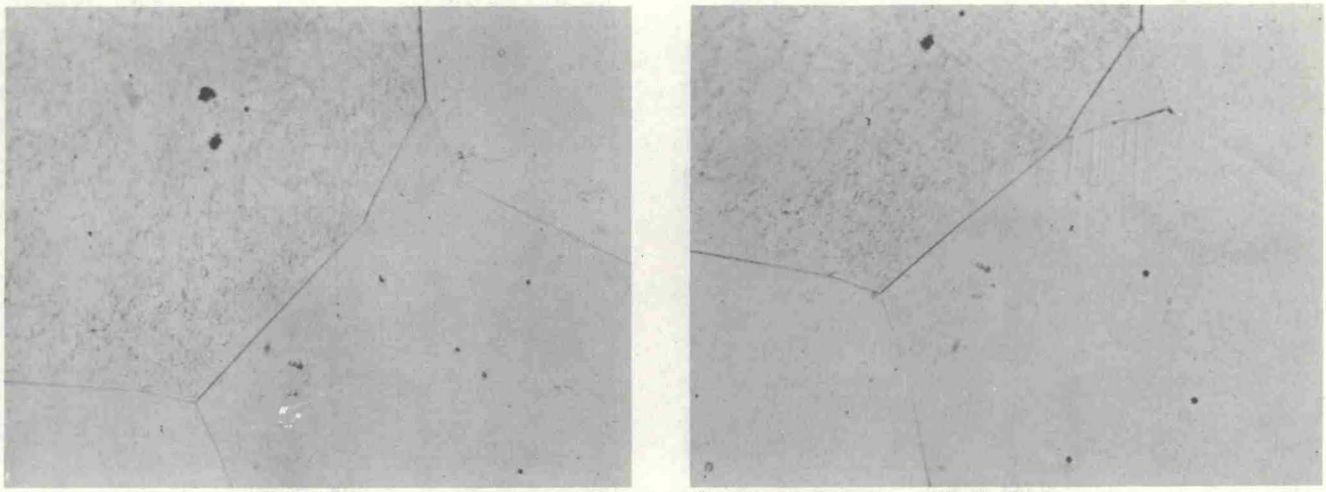
Pressure System. The pressure system used in this investigation is shown schematically in Fig. 2. The system operates by precharging the main cylinder, using the precharge pump and liquid-gas cylinder, to a pressure of approximately 2000 atm. The pressure is further built up by driving the piston into the cylinder using the upper jack as shown in Fig. 1. As the piston moves, it passes the precharge port, thus preventing high pressures from entering the precharge system. The high-pressure piston, which is schematically shown, is of the unsupported area type, using brass wedge rings against the cylinder wall.

In order to keep the main cylinder in triaxial compression simultaneous with the build-up of pressure, it is forced into a tapered jacket by means of the lower jack. With this arrangement, the system has a capacity of 30,000 atm.

Pressure measurement is by means of a manganin coil mounted in the bottom closure as also shown in Fig. 1. The pressure was recorded by a Foxboro recorder mounted on the control panel. Although it was not used in this investigation, a furnace, along with three thermocouples, can also be placed inside of the main cylinder. The leads for the manganin cell, along with those for the furnace and thermocouples, enter the main cylinder through the bottom closure.

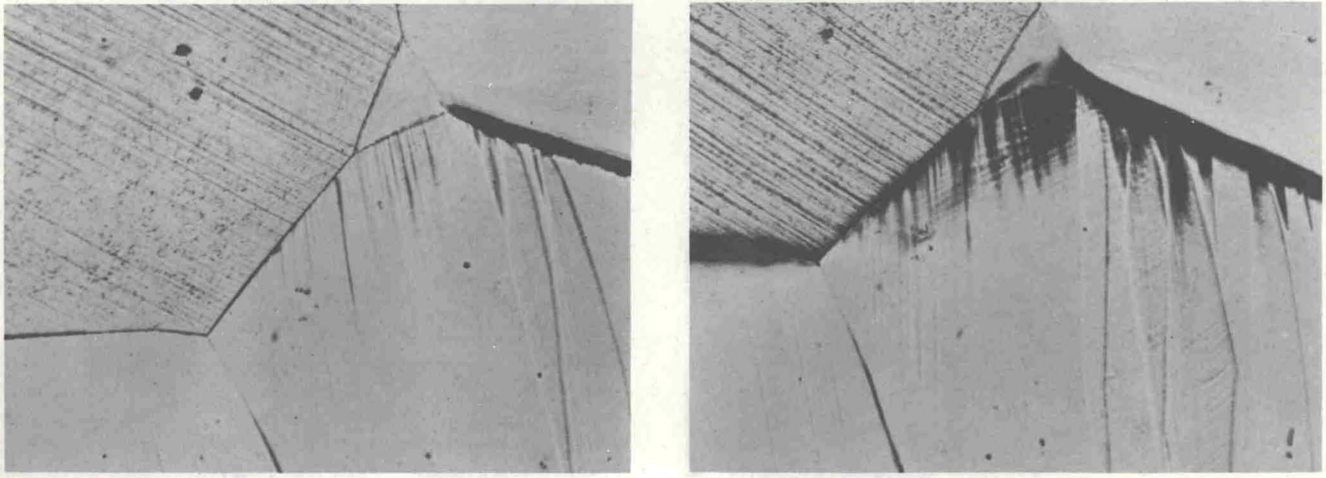
This pressure system is similar to that used by Bridgman[3] and Birch and Robertson,[4] and further details may be obtained from subject references.

For testing, the metallographically prepared specimens were placed inside of a holding cylinder in order to enhance handling, and this cylinder placed in the main cylinder of the pressure device. Extreme care was necessary in the handling of the specimen in order to prevent accidental deformation. In all pressure runs, single crystal control samples were simultaneously tested along with the polycrystalline samples to insure that there were no extraneous nonhydrostatic stress components. The pressure was held at each of the investigated levels of 5000, 10,000, 15,000, and 20,000 atm for 1/2 hr.



(*a*) 0 pct strain

(*b*) 1 pct strain



(*c*) 3 pct strain

(*d*) 5 pct strain

Fig. 9—Bismuth deformed in uniaxial compression showing slip in early and twinning in later stages. X100. Enlarged approximately 5 pct for reproduction.

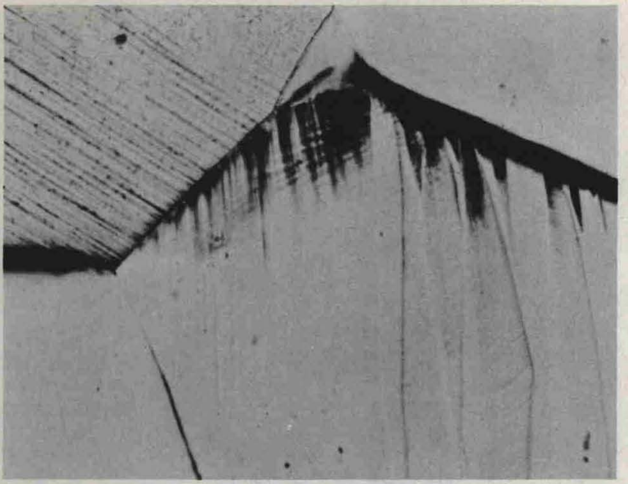
For pressures above 10,000 atm, proportionate amounts of a pentane-iso-pentane mixture was added.

RESULTS AND DISCUSSION

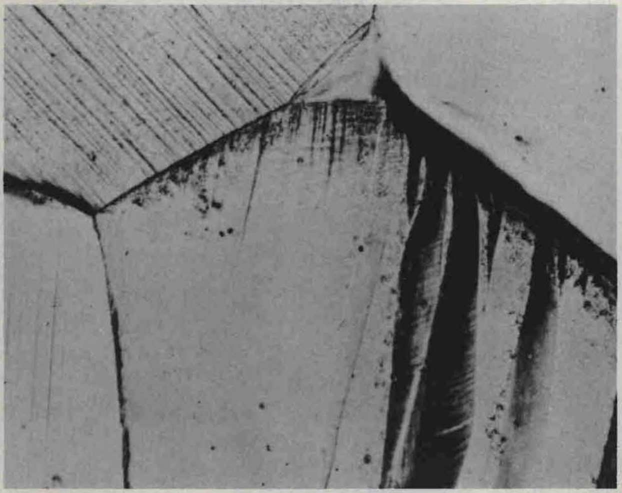
Using the experimental procedure and apparatus described, Figs. 2 through 7 are representative of the type and magnitude of the plastic deformation found in many polycrystalline bismuth samples subjected to pressures of up to 20,000 atm. The single crystals tested simultaneously with all polycrystalline samples exhibited no signs of plastic deformation. Even in such an anisotropic material as bismuth, the deformation in a single crystal under hydrostatic compression should be completely elastic in nature. Finding no permanent deformation in the single crystals, then, substantiates that there were no extraneous nonhydrostatic force components introduced to the polycrystalline samples during pressurization. The plastic deformation shown then must result from the localized shear stresses induced by the hydrostatic compression of a polycrystalline material exhibiting a high degree of anisotropy in its elastic properties.

The pressure-induced plastic deformation is initially highly localized, occurring first between those grains having a relative crystallographic orientation resulting in the highest induced shear stress which, when resolved, exceeds the yield stress of the grain, or grains concerned. In samples with 6-10 grains intersecting the polished surface, deformation is readily detectable in isolated areas at pressures as low as 5000 atm as shown in Figs. 2 through 6. Although not shown, very slight deformation was noted between grains in isolated cases at pressures as low as 2000 atm. As the pressure level is increased, the deformation increases in intensity and randomness throughout the entire sample. The widespread nature and randomness of this deformation, particularly at the higher pressure levels, can readily be seen in Fig. 7 which is representative of the smaller grained as-extruded structure.

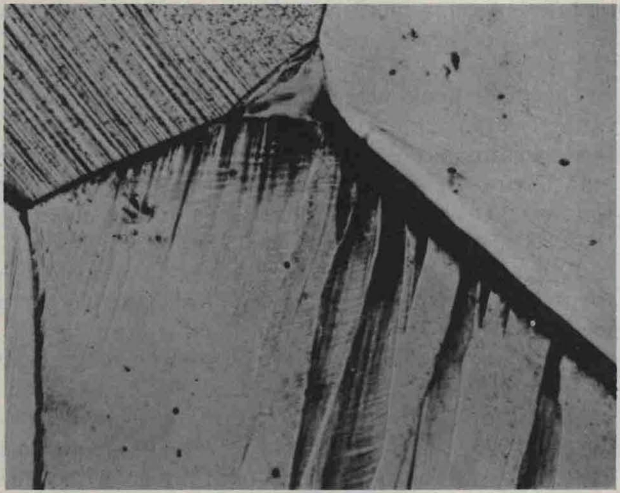
The observed plastic deformation was of two characteristic types. The first type, shown in various degrees in Figs. 2 through 7, is concentrated along the grain boundaries. As the pressure is increased, this area of highly concentrated deforma-



(*a*) 0 pct strain



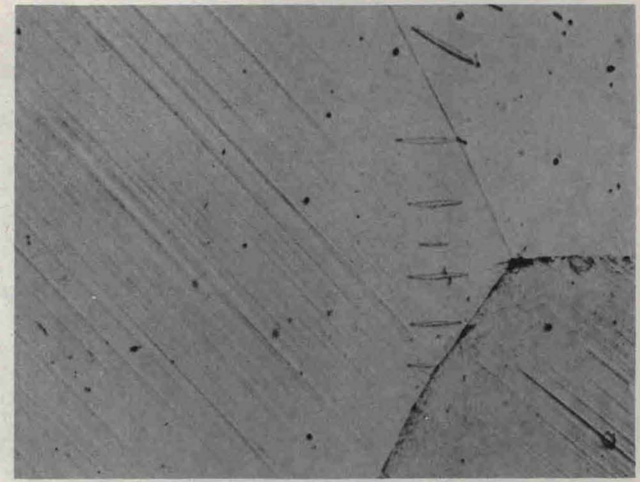
(*b*) 15,000 atm



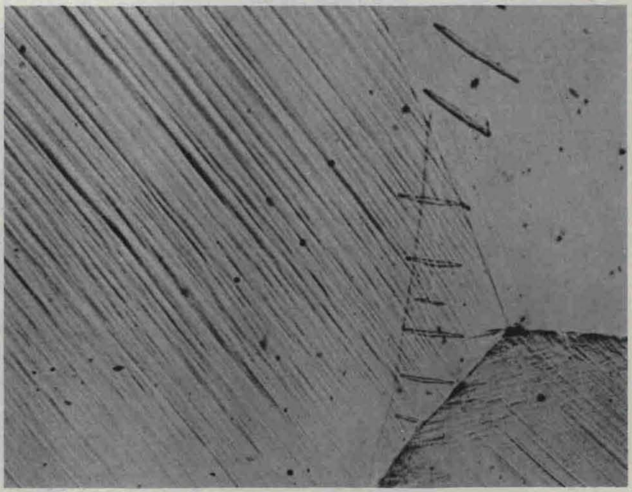
(*c*) 20,000 atm

Fig. 10—Extreme pressure induced deformation superimposed on bismuth previously deformed 5 pct in uniaxial compression. X100. Enlarged approximately 5 pct for reproduction.

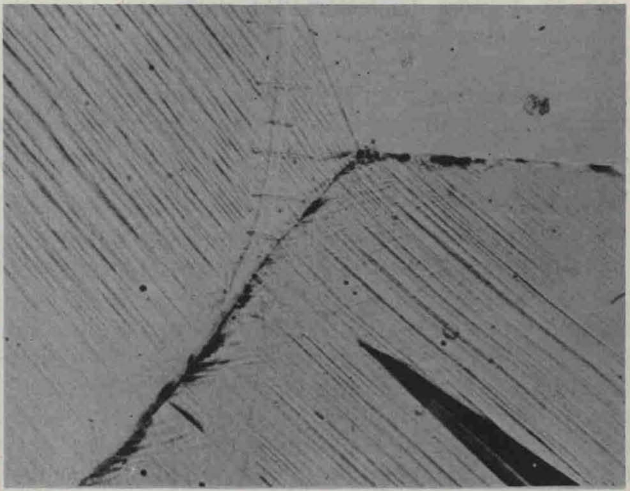
tion expands towards the center of the grain in a readily visible series of steps corresponding to the various pressure test levels. This deformation occurs only on one side of the boundary or the other.



(*a*) 0 pct strain



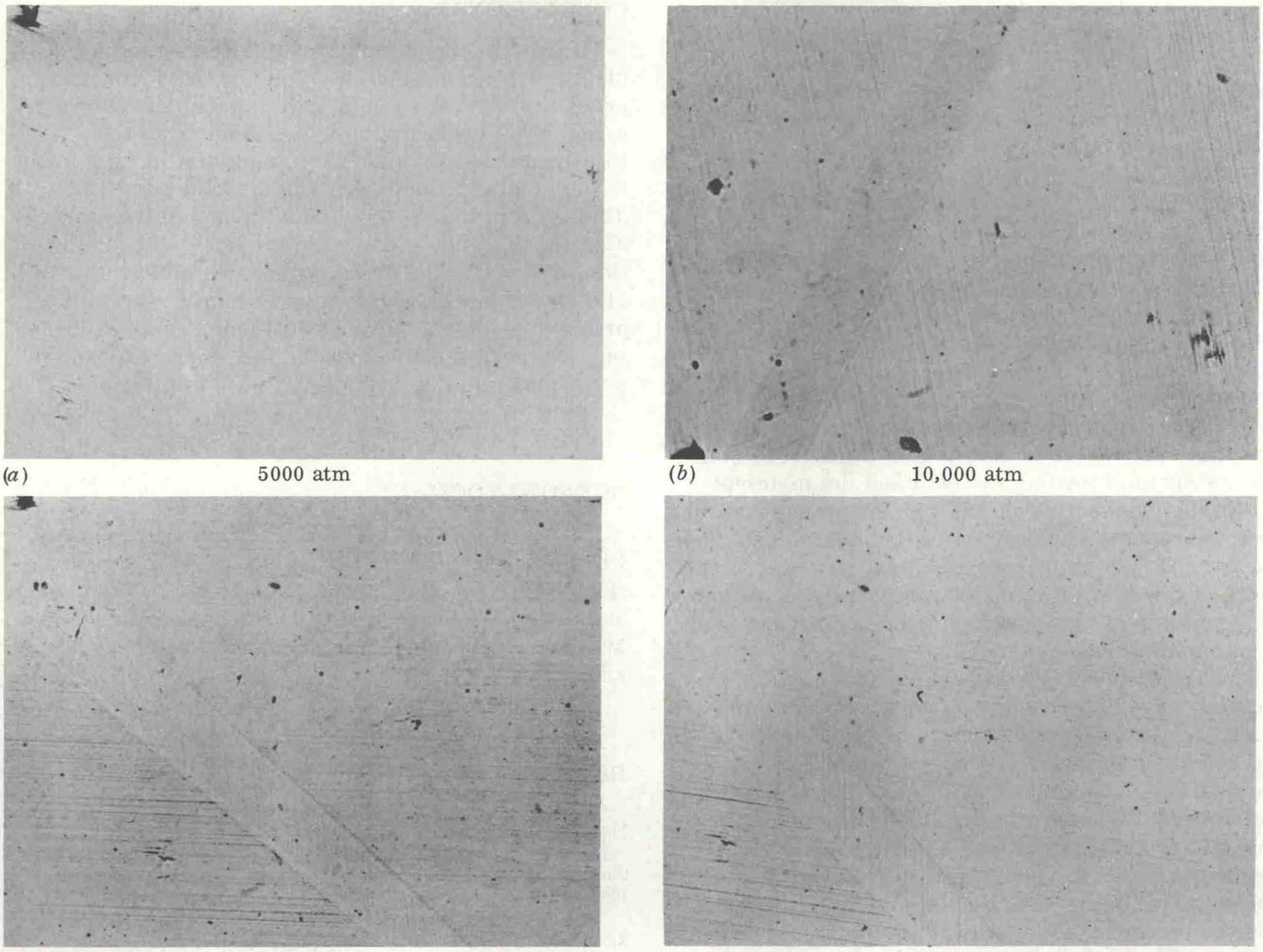
(*b*) 3 pct strain



(*c*) 5 pct strain

Fig. 11—Uniaxial compression of specimen previously subjected to 20,000 atm pressure. X100. Enlarged approximately 6 pct for reproduction.

Although it is difficult to ascertain precisely what mechanism, or mechanisms, are involved in this type of plastic deformation, close observation yields some indications. As clearly shown in Figs. 2 and 3, the slip lines of the grain, in which only



(a) 5000 atm

(b) 10,000 atm

(c) 15,000 atm

(d) 20,000 atm

Fig. 12—Mechanically twinned area subsequently subjected to extreme pressure. X100. Enlarged approximately 5 pct for reproduction.

slip has occurred, are almost continuous across the original grain boundary to the point where the concentrated deformation has progressively occurred. It appears, then, that in some manner the material in the region between the original boundary and the area of concentrated deformation has rotated to an orientation closely approximating that of the adjacent grain. A plausible explanation is that what appears to be the concentrated deformation is actually a form of grain boundary migration. Since the testing temperature of approximately 293 °K is somewhat greater than 1/2 of the absolute melting temperature of 544 °K for bismuth, the test conditions are within the range where boundary migration might be expected.

Other less plausible mechanisms are that the lattice adjacent to the boundary has undergone severe and concentrated bending or even fragmentation by cleavage along the (111) plane. Studies now underway to determine the effects of time and temperature on the type and magnitude of the observed phenomenon should lead to a better understanding of the mechanism involved.

The second general type of deformation observed is that of generalized slip and, in the later stages, multiple glide as shown in Figs. 5 and 6. This type of deformation is quite general throughout most of the prepared surface, becoming more generalized and increasing in intensity as the pressure is increased.

It is also interesting to note that there are substantial differences between the general deformation picture induced by extreme pressure as compared to that associated with uniaxial compression as shown in Figs. 8 and 9 for a series of strains ranging from 0 to 5 pct. As can be seen, initial deformation is predominantly by slip and at somewhat higher strains by a combination of slip and twinning which has been previously discussed by Gyndyn and Startsev.[5] Although there is some deformation concentrated near the boundaries, it is apparently, a bending type phenomenon, and, as evidenced from the comparison of Fig. 9 with 2 through 4, not the same as that induced by hydrostatic compression. A further comparison is shown in Fig. 10 which consists of a specimen uniaxially strained as shown in Fig. 9, then exposed to pressures of 20,000 atm. The boundary type phenomenon characteristic of extreme pressure can be readily seen superimposed on the results of uniaxial straining. The specimen, shown in Fig. 11, was first deformed by an extreme pressure of 20,000 atm, then uniaxially compressed 3 pct. The difference between the two types of deformation caused by the different stress states is again obvious.

It is interesting to note that although twinning is a primary mode of deformation in uniaxial compression, none has been detected in the many specimens deformed by extreme pressure. Fig. 12 shows a twin, mechanically induced prior to subjecting the specimen to pressure. As can be seen, there is a narrow band along each side of the original twin boundary that tends to increase in width as the pressure is increased. It is interesting to note, however, that this narrow band exhibits slip parallel to both the matrix surrounding the twin and the material within the twinned region. It appears, then, that this band represents somewhat of a transition zone, and may or may not represent an actual increase in the width of the twin. A more detailed study of the effects of extreme pressure on twinning and the twin mechanism is now underway.

The results presented herein cover only the effects of extreme pressure on polycrystalline bismuth. Work is either underway or planned for near future execution into the study of the effects of extreme pressure on materials of varied structures and degrees of anisotropy. In these studies, bicrystals of varied relative orientation will be examined in order to understand more fully the conrolling parameters including symmetry, orientation, and temperature, associated with anisotropy induced deformation, and to gain quantitative data which may be compared to theory. It is felt that a knowledge of how a hydrostatic stress state effects the basic structure of materials is necessary in order to study and explain accurately many of the extreme pressure phenomena exhibited by materials in the polycrystalline form.

CONCLUSIONS

Polycrystalline bismuth exhibits substantial plastic deformation when subjected to hydrostatic pressures as low as 5000 atm and ambient temperatures. This deformation is attributable to the localized shear stresses arising from the anisotropy in the elastic properties. The deformation, which differs from that associated with uniaxial compression, is characterized by severe localized distortion adjacent to the grain boundaries and generalized slip and cross-slip. Twinning, which is one of the primary modes of deformation under uniaxial loading, has not been observed in specimens exposed to pressures up to 20,000 atm.

ACKNOWLEDGMENT

The work performed by Mr. Richard Hyserman, Physical Science Aide, in assisting in the preparation of samples and operation of pressure equipment, and the helpful discussions on the part of R. V. Milligan, Mechanical Engineer, are gratefully acknowledge.

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