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The Study of the Structural and Transformation Characteristics of the Pressure-Induced Polymorphs in Bismuth

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It is known from the early work of Bridgman that the two lowest-pressure transitions (I-II and II-III) are accompanied by substantial and abrupt changes in resistivity and volume. However, unlike the temperature-induced allotropic transformations observed in such elements as lithium, cobalt, tin, and so forth, there is little actually known about many of the characteristics of the pressure-induced transitions. This current work involves an examination of the structural and transformation characteristics of the bismuth I-II and II-III transitions under hydrostatic pressures. The relationship of initial structure to the transformation pressure, rate, resistivity change, and resultant structure is discussed. It is shown that the transition pressure and transformation rate are independent of the presence of grain boundaries and associated anisotropy-induced deformation. An observed hysteresis in both the I-II and II-III transitions is shown.

BISMUTH is one of the most interesting of the elements exhibiting pressure-induced polymorphs since it undergoes several allotropic transformations at pressures below 90,000 atm. It is known from the early work of Bridgman[1,2] that the two lowest-pressure transitions (I-II and II-III) are accompanied by substantial and abrupt changes in resistance and volume. However, unlike the temperature-induced allotropic transformations observed in such elements as lithium, cobalt, tin, and so forth, there is little actually known about many of the characteristics of these pressure-induced transitions. It is the purpose of this work to examine some of the structural and transformation characteristics of the bismuth I-II and II-III transitions under hydrostatic pressures.

Another interesting characteristic of bismuth is that, in its polycrystalline form, hydrostatic pressures of sufficient magnitude will induce severe progressive plastic deformation in the region of the grain boundaries.[3] This deformation, which has also been observed in several other metals, is attributed to the high degree of anisotropy in the linear compressibility of bismuth, resulting in shear stresses in the grain boundaries when it is exposed to hydrostatic pressure.

Most thermally induced allotropic transformations in metals, whether of the diffusionless athermal (martensitic) or isothermal nucleation and growth types, are dependent upon structure and prior history,[4] viz., grain boundaries, deformation, and so forth. One logically wonders then whether the transformation characteristics of the pressure-induced polymorphs in bismuth might also depend upon initial structure, particularly with respect to the presence of grain boundaries and associated plastic deformation.

In this investigation, the role of grain boundaries and plastic deformation on the characteristics of the bismuth I-II and II-III transitions will be established. The rather unique residual microstructural changes associated with these transitions will be presented and discussed. The occurrence of a measurable hysteresis in both the I-II and II-III transitions will be demonstrated. The type of transformation mechanism based on the observed transformation rate will be discussed.

EXPERIMENTAL PROCEDURE

A) Apparatus. The hydrostatic pressure system utilized in this investigation is similar to that previously reported by Bridgman[1] and Birch and Robertson,[5] and has been previously described.[3]

For the purpose of this work, the pressure medium utilized was a 1:1 mixture of pentane and isopentane. Pressure measurement was by means of a manganin coil in conjunction with a Foxboro Recorder. The manganin coil was mounted in the bottom closure and inserted inside the pressure cavity. Based on calibration against a controlled clearance piston gage at approximately 10,000 atm, the estimated error in the pressure measurement was ±2 pct. Assuming the nonlinearity in the pressure coefficient of resistivity between 10,000 and 28,000 atm to be not greater than 1 pct, then the estimated error in the range of the I-II and II-III transitions was ±3 pct.

B) Specimen Material and Preparation. The bismuth utilized throughout this investigation was of

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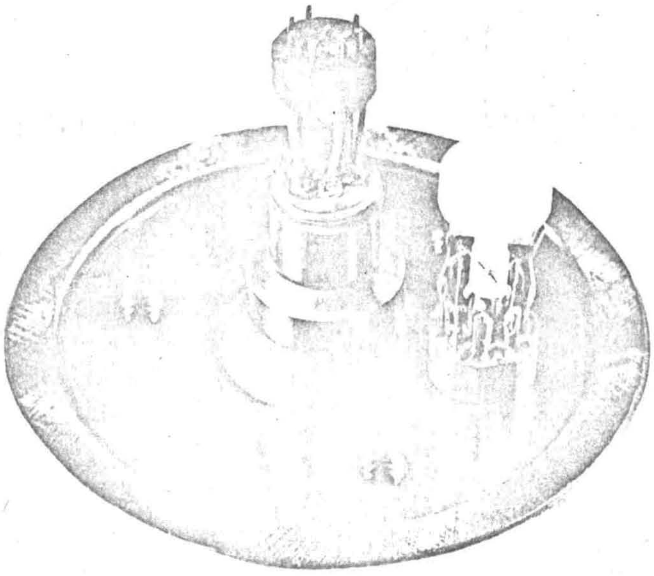

Fig. 1—Specimen holder and bottom closure for hydrostatic pressure system.

high-purity single-crystalline stock having the following detectable impurity levels:

Pb - 0.6 ppm
Fe - 0.9 ppm
Cu - 0.1 ppm
Cd - 0.3 ppm

In order to minimize variations in impurity content between the single and polycrystalline specimens, the polycrystalline samples were produced from the original single crystals by remelting into a 3/4-in. billet and extrusion at 220°C to a 0.17-in. diam. The smaller grain size samples were prepared directly from the as-extruded material. For the larger grain size, the as-extruded material was subjected to an annealing treatment of 220°C for 5 hr. It was attempted to obtain polycrystalline samples by progressive compressive deformation of the single crystals with intermediate annealing. Although moderate grain sizes could be obtained in this manner, this technique was abandoned in favor of the former.

The specimens were 0.16 in. in diam and 0.22 in. long. Prior to pressurization, a plane parallel to the longitudinal axis of the specimen was metallographically prepared by electropolishing using a saturated KI solution with 2 pct by volume of concentrated HCl.

C) Resistance Measurement. Electrical resistance as a function of pressure was measured by means of a conventional potential-drop method using a K-3 type potentiometer and recording oscillograph. Two specimens were connected in series to a current source, and the potential drop across each was measured separately. The arrangement of the two specimens in the sample holder is shown in Fig. 1 along with the bottom pressure closure. For the pressure run, the sample holder was simply connected to the seven-anode insulating block of the closure. There is a total of seven electrical leads emerging through the bottom closure with two being used for the current through the specimens, four for voltage measurements, and the remaining one for the manganin coil inserted inside of the insulating block.

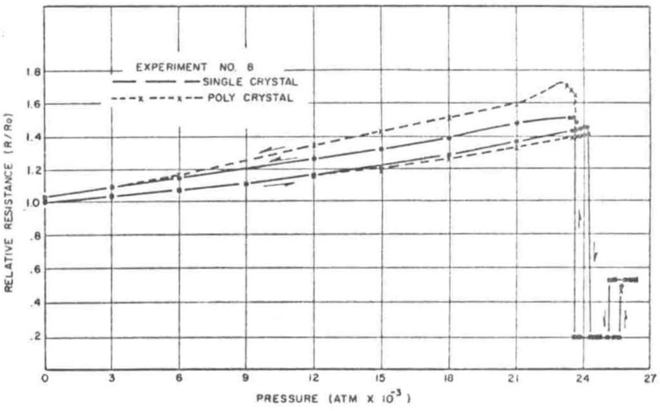

Fig. 2—Relative resistance vs pressure.

D) Procedure. In all experiments, pressure was increased by 3000-atm increments up to 21,000 atm and a 2000-atm increment to 23,000 atm with a 5- to 10-min hold period between each pressure change to permit stabilization of the pressure and voltage readings due to thermal effects. Beyond 23,000 atm, four different procedures, consisting of *a*) 100-, *b*) 250-, and *c*) 500-atm increments with 5-min stabilization periods, and *d*) continuous pressurization at the rate of 50 atm per min, were utilized. The depressurization rate closely approximated the procedure used for increasing pressure.

RESULTS AND DISCUSSION

A) Transition Pressure. In order to establish the effect of initial structure on the transition variables, a single crystal and a polycrystalline sample, connected in series in the manner previously described, were simultaneously exposed to the pressure. Typical relative resistance vs pressure curves utilizing this procedure are shown in Fig. 2.

As is demonstrated in Fig. 2, the I-II and II-III transitions, upon increasing pressure, occur isobarically with the transformation pressure being independent of initial structure. Similarly, the III-II transition, upon decreasing pressure, is also isobaric and structure-insensitive. However, the II-I transition is not completely isobaric, but exhibits some sluggishness near the completion of transformation. This deviation from isobaric conditions is small, usually not exceeding 300 atm.

Under the condition of this experiment, there is a substantial hysteresis in both the I-II and II-III transitions. The magnitude of this hysteresis is independent of structure, but somewhat dependent upon pressurization rate as is shown in the following tables.

The single value of the pressures and pressure difference shown is the average for all tests (only

I-II Transition

Pressurization Procedure	$P_{\text{I-II}}$, K-atm	$P_{\text{II-I}}$, K-atm	$\Delta P_{\text{I-II}}$, K-atm
500-atm increments	24.23 (24.10-24.50)	23.29 (22.90-23.50)	0.94 (0.60-1.20)
250-atm increments	24.25 (24.20-24.30)	23.20 (23.00-23.50)	1.05 (0.75-1.30)
100-atm increments	24.20 (24.05-24.30)	23.49 (23.40 23.70)	0.71 (0.55-0.90)
Uninterrupted at 50 atm per min.	24.31 (24.25-24.35)	23.20 (23.15-23.40)	1.11 (0.95-1.20)

II-III Transition

Pressurization Procedure	$P_{\text{II-III}}$, K-atm	$P_{\text{III-II}}$, K-atm	$\Delta P_{\text{II-III}}$, K-atm
500-atm increments	25.55 (25.50-25.60)	24.84 (24.70-25.10)	0.71 (0.50-0.80)
250-atm increments	25.65 (25.60-25.70)	24.98 (24.95-25.00)	0.67 (0.65-0.70)
100-atm increments	25.70 25.70	25.15 (24.95-25.30)	0.55 (0.40-0.75
Uninterrupted at 50 atm per min	25.75 25.75	25.13 (25.10-25.15)	0.62 (0.60-0.65)

the first cycle in case of recycling) in the specific category and the double values, in parentheses, the data spread. Although the differences are relatively small, it can be seen that the magnitude of the hysteresis in the I-II transitions tends to increase with increasing pressurization rates, which most probably is due to nonequilibrium conditions resulting in "over-driving" of the transitions.

Recycling several times through either of the transitions seems to have little, if any, effect on the magnitude of the hysteresis. For example, passing through the I-II transitions three times does not result in any consistent change in the hysteresis. Recycling four times through the II-III transition resulted in a reduction from 750 to 500 atm.

It is of concern whether the observed hysteresis is, in fact, a real phenomenon associated with these transitions or simply a function of the experiment. From the standpoint of the experiment, the two possible causes of observed hysteresis are substantial nonequilibrium associated with too rapid a pressurization rate and/or natural hysteresis in the manganin-coil pressure transducer.

Considering the first possibility, using 100-atm increments with a 5-min stabilization period there was usually a substantial period of elapsed time at the transition pressure before the transformation initiated. This is clearly shown in Fig. 3 which is a typical plot of relative resistance vs time as obtained from the continuous oscillographic recording of voltage change for simultaneously pressurized single and polycrystalline samples. Time = 0 on the abscissa corresponds to the time at which the transition pressure was reached. In any given experiment, the transitions normally did not initiate simultaneously in both samples. However, considering the curve for the single crystal shown in Fig. 3 as an example, approximately 3 min elapsed before the I-II transition initiated and 7 min before the initiation of the II-III. For decreasing pressure, the elapsed time was approximately 1 and 2 min, respectively, for the III-II and II-I. Apparently, then, using 100-atm increments with a stabilization period is close enough to equilibrium conditions so that the hysteresis is not attributable to "overdriving" of the transitions. Using the larger pressure increments, the transitions often occurred during the increase or decrease of pressure, resulting in little or no elapsed time at the transition pressure and short durations of transformations. In these instances, there is a greater tendency to "overdrive" the transitions, resulting in an increase in the magnitude of the hysteresis.

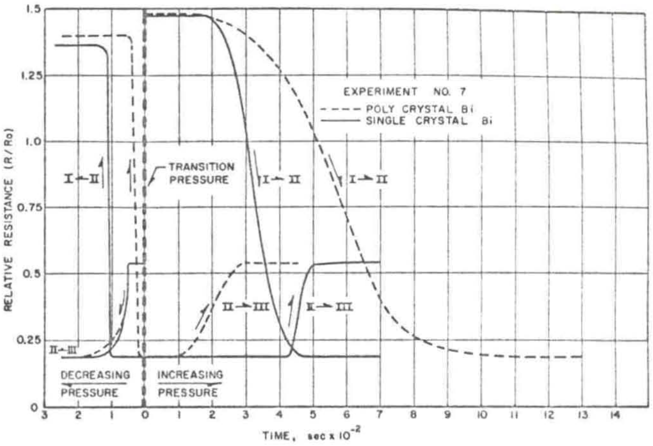

Fig. 3—Typical relative resistance vs time curves for I-II and II-III transitions.

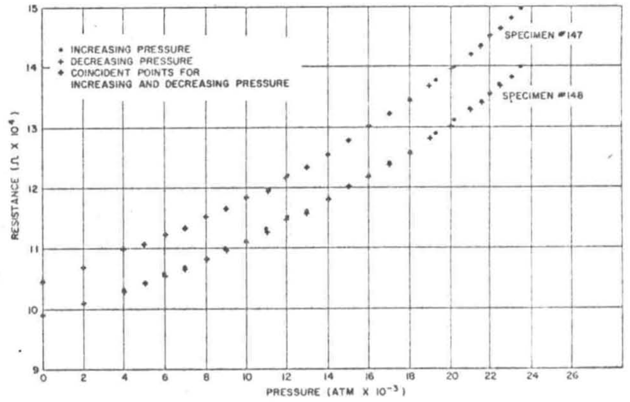

Fig. 4—Resistance vs pressure for single crystals.

That the observed hysteresis is not due to the manganin-coil transducer is based on the following two considerations. First, any hysteresis occurring in the pressure measurement would vanish at the peak pressure and be a maximum at the midpressure range. However, the hysteresis in the transition was observed even when the applied pressure just exceeded the transition pressure by a few hundred atmospheres, and also in the case of the partial I-II transformation. Second, as a further check, two bismuth single crystals were pressurized to just below the I-II transition. The relative resistance vs pressure data for this experiment is shown in Fig. 4. Since there was no observed structure change in the samples, one would not expect to see any natural hysteresis in the bismuth. It is evident from the figure, therefore, that up to 23,500 atm the hysteresis due to the manganin coil is negligible.

Based on the above considerations, it must be concluded that there is a real hysteresis in the bismuth I-II and II-III transitions that, under equilibrium or near equilibrium conditions, has an average magnitude of 730 and 550 atm, respectively. The fact that this hysteresis was not detected by Bridgman[1] is most probably due to his utilizing 1000-atm increments in the region of the transition. Since the pressure increments used in his research were approximately the same size as the hysteresis observed in this work, it could have readily gone without detection.

B) Relative Resistance. Some mention should be made of the resistance changes associated with the bismuth transitions, particularly with respect to their structural implications.

Relative resistance (R_p/R_0) data at the transition pressure from all experiments are summarized as follows according to initial structure, where the single value represents the average from all experiments (for first cycle only in case of multiple cycles) and the double value, in parentheses, the data spread.

	Increasing Pressure			Decreasing Pressure	
	R_{I}	R_{II}	R_{III}	R_{I}	R_{II}
Single crystal	1.470 (1.280-1.890)	0.191 (0.160-0.254)	0.559 (0.430-0.650)	1.560 (1.31-1.940)	0.199 (0.134-0.254)
Polycrystal	1.455 (1.340-1.570)	0.204 (0.180-0.230)	0.548 (0.507-0.570)	1.520 (1.390-1.720)	0.204 (0.180-0.210)
Bridgman[1]	1.514	0.239	0.616	—	—

As can be noted, the average values of the relative resistance for the three phases at the point of transition upon increasing pressure are similar to those reported by Bridgman for single crystals. Since the single crystals in this program were of random orientation, the values vary considerably, which simply reflects anisotropy in the resistivity and pressure coefficient of resistivity. As would be expected due to its randomizing effect on anisotropy, the data spread for the polycrystalline specimens is somewhat smaller than that observed for the single crystals.

There is a difference, again being larger in the case of the single crystals, between the relative resistance of Phase I and II observed under increasing and decreasing pressure. Bridgman[1] also observed a similar effect and mentioned that it might be associated with a change from a single to polycrystalline structure as a result of passing through the I-II transition, thus reducing, or eliminating, the anisotropy inherent in the original single crystals. As will subsequently be shown, the original single-crystalline structure is lost as a result of passing through the I-II transition, which could account for such an observed difference. However, even in the case of the original polycrystalline samples where one naturally assumes complete, or near complete, isotropy, there is a marked difference between the relative resistance at the point of transition, particularly of phase I, for increasing and decreasing pressure. This, along with the fact that the final resistance differs from the original, indicates that the polycrystalline structure formed as a result of passing through the II-I transition may exhibit substantial orientational regularity ("texturing"), thus differing from the initial isotropic polycrystalline structure. This residual polycrystalline structure common to all initial structural conditions will be subsequently discussed.

C) Transformation Rate. The transformation rate associated with subject transition varies widely from test to test and within any given pressure run. For example, whereas in Fig. 3 the single-crystal sample transforms more rapidly in the case of the I-II transition, in other experiments the polycrystal transforms at a higher rate. There appears, however, to be no reproducible effect of initial structure upon the transformation rate or any indication that one of the transitions was more sluggish than the other.

As would be expected, the rate of transformation is highly dependent upon the pressurization rate. At the higher rates, the transitions often initiate while pressure is being changed, in which case it proceeds rapidly, reaching completion in as little as 10 sec. At the slower pressurization rates (Fig. 3 being a typical example of the 100-atm increment procedure), the transition may require several minutes to go to completion.

If one assumes that relative resistance change is proportional to the volume percentage of the two phases present at any given time, then the curves, shown in Fig. 3, are similar in shape to that predicted based on nucleation and growth theory for an isothermal transformation.[6] It is indicated, therefore, that the pressure-induced I-II and II-III transitions in bismuth are thermally activated nucleation and growth-type processes in contrast to the very rapid nonthermally activated diffusionless type (often called martensitic) as observed in such pure metals as lithium, cobalt, and zirconium.

D) Microstructural Analysis. Typical residual structures for single and polycrystalline bismuth after cycling through the I-II transition are shown in Figs. 5 and 6 and for I-II and II-III transitions in

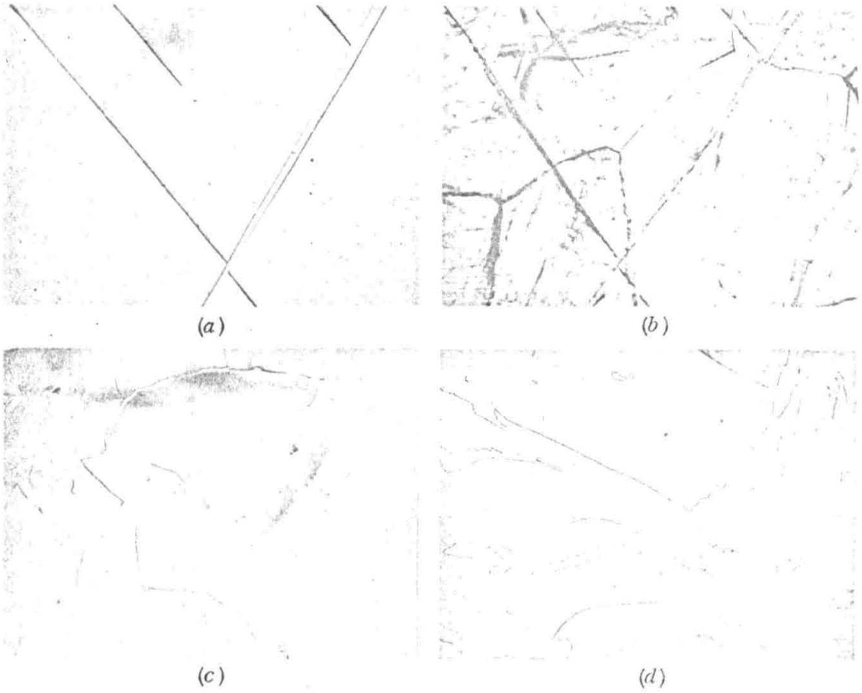

Fig. 5—Structural changes in bismuth single crystal after I-II transition. (*a*) Original structure; (*b*) after transition; (*c*) intermediate repolish; (*d*) final structure. X100. Reduced approximately 42 pct for reproduction.

Figs. 7 and 8. In these figures, the original structure (*a*) represents the metallographically prepared surface prior to pressurization. The structure after transition (*b*) shows the same region as the original area immediately after removing the sample from the pressure cell but without further metallographic preparation. One can readily see the "ghost" image of the original structure, *i.e.*, original Phase I grain boundaries and twin bands, along with an image of the structure in the Phase II or Phase II and III regions. Considering Figs. 5 and 6, one can readily see pronounced grain boundaries depicting the polycrystalline nature of Phase II, along with substantial surface distortion due to volume change. When the sample passes through both the I-II and II-III transitions, as shown in Figs. 7 and 8, the structure is much more diffuse since two volume changes occurred. It is still possible, however, to see an image of the original structure as well as some grain boundaries probably associated with Phase II bismuth.

That the structure observed directly after transition is actually a "ghost" image depicting the history of the sample is demonstrated by its removal

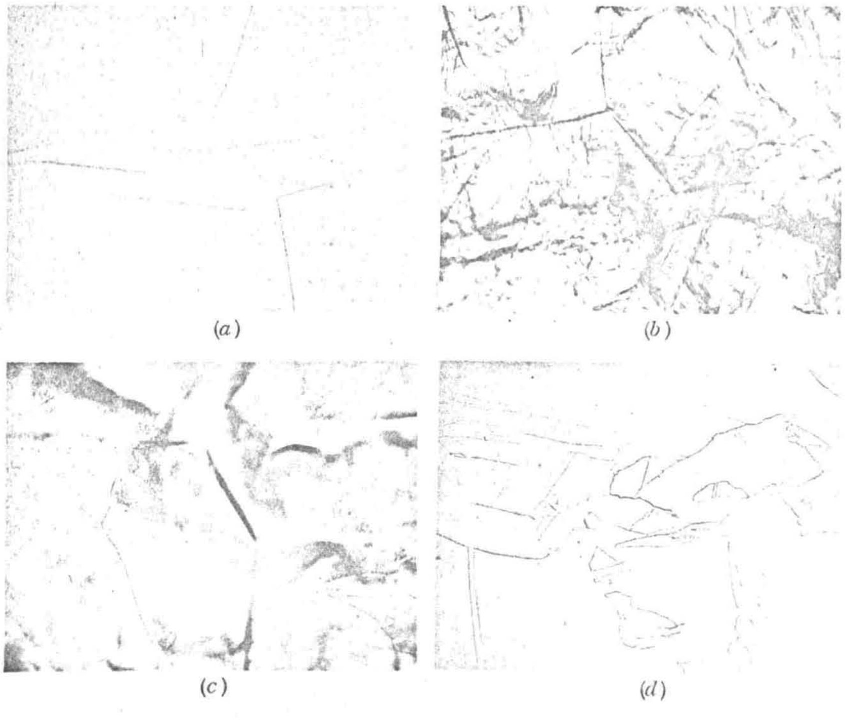

Fig. 6—Structural changes in polycrystalline bismuth after I-II transition. (*a*) Original structure; (*b*) after transition; (*c*) intermediate repolish; (*d*) final structure. X100. Reduced approximately 42 pct for reproduction.

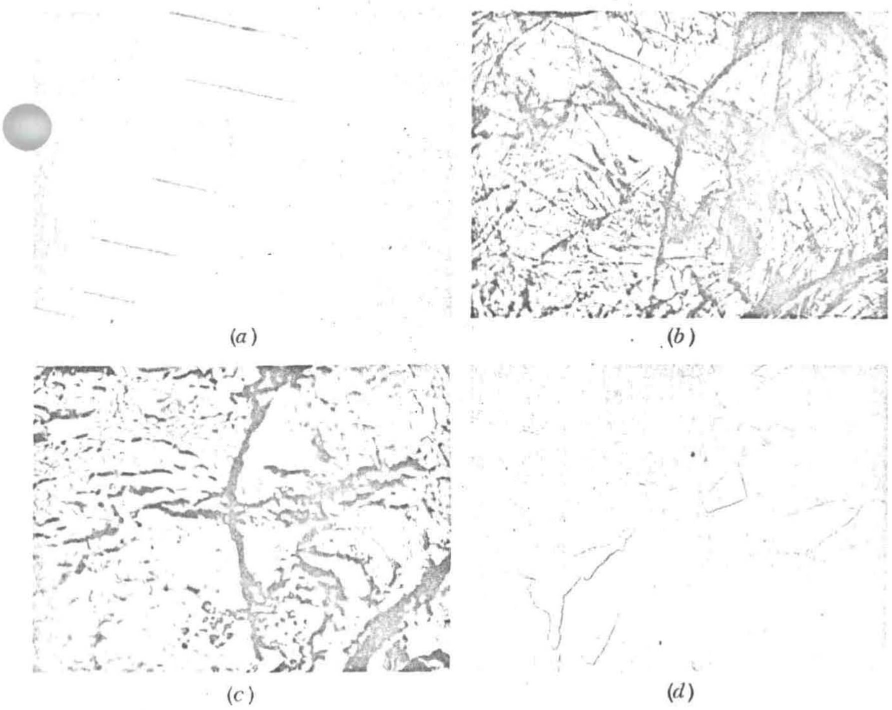
(a) (b) (c) (d)

Fig. 7—Structural changes in single-crystal bismuth after I-II-III transitions. (*a*) Original structure; (*b*) after transition; (*c*) intermediate repolish; (*d*) final structure. X100. Reduced approximately 42 pct for reproduction.

by slight repolishing as shown in (*c*) of the subject figures where the gradual disappearance of the original Phase I and superimposed Phase II boundaries is evident.

As can be noted, the final or residual structure, after complete repolishing, is polycrystalline in nature and is the same whether the sample was originally single or polycrystalline or passed through the I-II or I-II and II-III transitions. It is characterized by some twinned regions, as is commonly encountered in this material, and many apparently isolated grains completely surrounded by a larger grain or matrix region.

An interesting characteristic of the polycrystalline structure resulting from cycling through the transitions is its drastically enhanced grain boundary migrations. It was observed that, regardless of the initial structure or whether it passed through the I-II or II-III transition, this residual polycrystalline structure becomes single crystalline, based on an optical observation of the polished surface, after annealing at 160°C for 3 hr. Furthermore, at 120°C for 3 hr, the residual structure consisted of only two to three grains intersecting the polished surface. As a matter of comparison, the as-extruded material, which had a grain size comparable

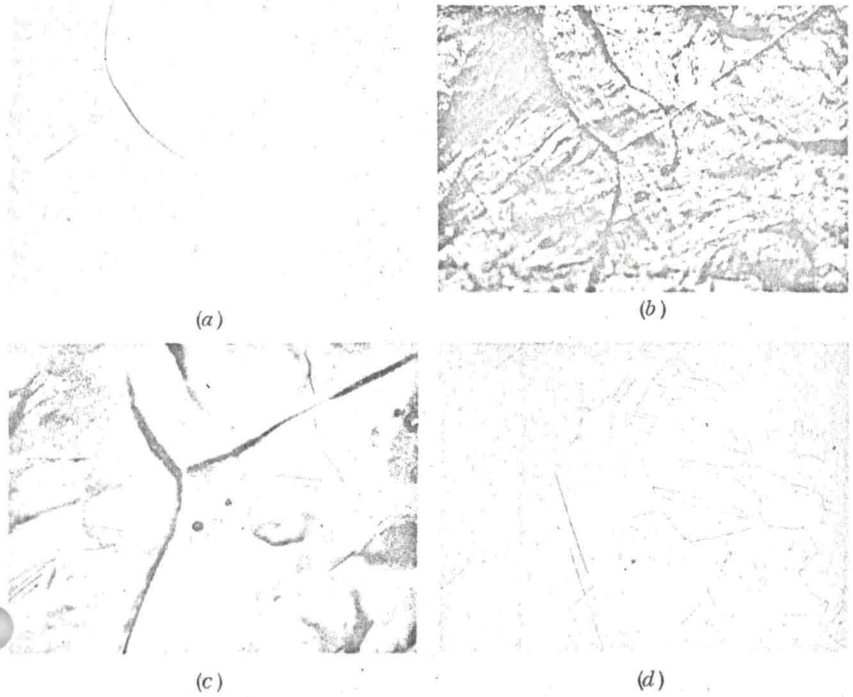
(a) (b) (c) (d)

Fig. 8—Structural changes in polycrystalline bismuth after I-II-III transitions. (*a*) Original structure; (*b*) after transition; (*c*) intermediate repolish; (*d*) final structure. X100. Reduced approximately 42 pct for reproduction.

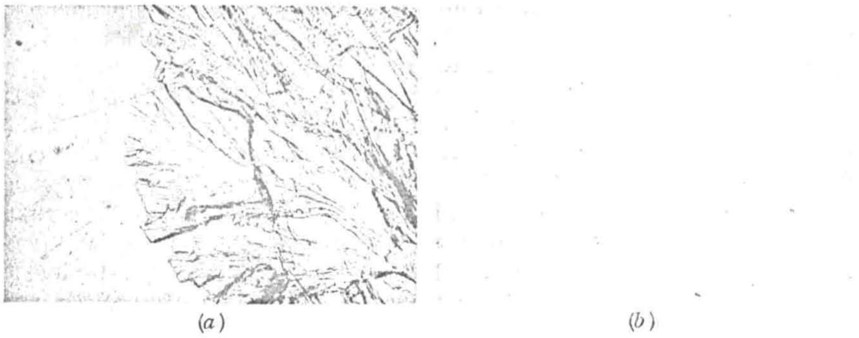
(a) (b)

Fig. 9—Structural changes in single-crystal bismuth after 50 pct completion of I-II transitions. (*a*) After 50 pct completion; (*b*) final structure in transformed region after repolishing. X100. Reduced approximately 42 pct for reproduction.

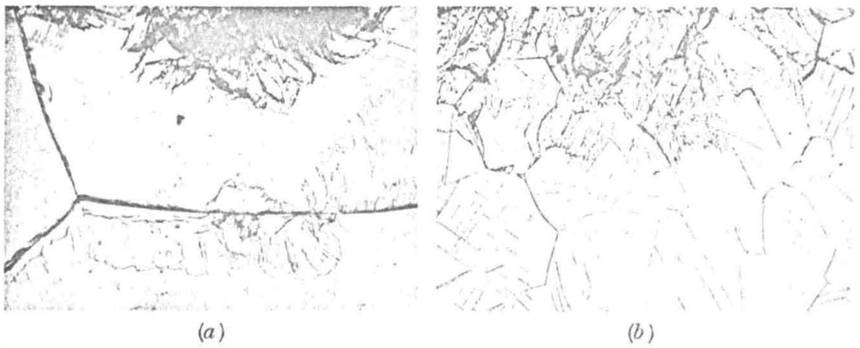
(a) (b)

Fig. 10—Structural changes in polycrystalline bismuth after various stages of completion of I-II transition. (*a*) After 38 pct completion; (*b*) after 41 pct completion. X100. Reduced approximately 42 pct for reproduction.

to that of the residual structure after transition, required an annealing treatment of 220°C for 5 hr which resulted in ten to twenty grains intersecting an equivalent polished area as demonstrated in the original structure photomicrographs of Figs. 6 and 8.

Two factors that could contribute to the enhanced boundary migration are stored strain energy and preferential orientation of the structure as indicated by the difference in resistivity after pressurization. The stored strain energy arises from the fact that, just as increasing pressure progressively induces shear stresses in the grain boundaries of Phase I bismuth, so also will decreasing pressure once the material has transformed back from Phase II to I. Undoubtedly, substantial boundary migration and deformation occur progressively in Phase I reaching a maximum at atmospheric pressure. The available stored strain energy at atmospheric pressure due to this deformation could materially affect the rate of boundary migration.

A better understanding of how the transformations occur and the rather unusual residual structure can be gained from the examination of partially transformed specimens. For the purpose of obtaining partially transformed samples for examination, the I-II transition was interrupted after a certain percentage of voltage change, as indicated by the oscilloscopic recorder, by simply reducing the pressure by 100-atm increments. Partial transformation in a single-crystal sample is shown in Fig. 9.

Referring to (*a*) of Fig. 9, the boundary between the transformed and untransformed region is quite evident. The transformed region is typical of that observed in all cases for the I-II transition containing an image of the polycrystalline nature of Phase II. There is a small amount of slip in the untransformed area, undoubtedly due to deformation induced by the volume change of the transformed portion of the sample. Photomicrograph (*b*), which is of the transformed area in the region of the initial transformed-untransformed boundary after repolishing, shows an interesting characteristic. The initial boundary is gone, and the only visible variation from the original single-crystal structure consists of a very few small isolated grains, one of which is shown in the center of the photomicrographs.

There appear to be at least two plausible explanations for this reversion back to near single-crystalline form and the presence of the isolated regions. First, substantial boundary migration occurs to reduce the progressively increasing shear stresses in the polycrystalline form of Phase I transformed from the Phase II region, as was previously described. The second possibility is that the untransformed Phase I region controls the direction of growth and possibly the orientation of the nuclei of Phase I transformed from Phase II, similar in effect to the growth of single crystals from a "seed" crystal. This is probably due, at least in part, to the large anisotropy of the linear compressibility of bismuth which, under pressure, could tend to enhance growth of nuclei similarly oriented to the untransformed volume and, due to the greater energy involved, retard the growth rate of those with a different orientation. This effect would be more prevalent in the region of the interface between the transformed and untransformed region. At a distance removed from the interface, nucleation and growth of unfavorably oriented material occur to a limited extent, as shown by the small isolated regions. However, the enhanced growth rate of the favorably oriented nuclei effectively overruns these unfavorable regions, resulting in isolated regions surrounded by material of the same orientation as the untransformed region. The specimen would undoubtedly revert completely back to a single crystal if given enough time during the transition period or at elevated temperatures where diffusion would be enhanced.

Which of the above phenomena is the more important cannot be ascertained from available evidence. It is likely that the final structure is a manifestation of the combined effects of both mechanisms.

When the specimen completely transforms, as shown in Figs. 5 through 8, Phase I nucleates and grows from Phase II in many initially independent regions. However, as was previously discussed, it is felt that the high anisotropy of the linear compressibility again enhances the growth of some grains during the decrease of pressure, while retarding the growth of others to such an extent that some unfavorably oriented areas are effectively overrun and surrounded.

Partial I-II transformation in annealed and as-extruded polycrystalline structures is shown in Figs. 10(*a*) and 10(*b*), respectively. As can be noted, the original Phase I grain boundaries, including those along which severe anisotropy-induced deformation has occurred, do not appear to be preferential sites for Phase II nucleation. Although in photomicrograph (*a*) of this figure transformation appears to be associated with a Phase I boundary in one area, in another region it is independent. This lack of any significant dependency of the I-II transformation on Phase I boundaries is more clearly shown in (*b*) which bears out the insensitivity of the transition pressures and rate to initial structure. The formation of Phase III from II is even further removed from the original Phase I structure; thus one would not expect to encounter any structural sensitivity.

CONCLUSIONS

1) The bismuth I-II and II-III transition pressure and transformation rate are structure-insensitive with respect to the presence of grain boundaries and associated localized plastic deformation.

2) The I-II and II-III transitions are completely isobaric for increasing hydrostatic pressures. The III-II also occurs isobarically but, at very slow pressurization rates, there is a tendency for the final stages of the II-I transition to extend over a narrow range of 200 to 300 atm.

3) Even under very slow pressurization rates consisting of 100-atm increments with 5-min stabilization periods, there is a measurable hysteresis in both the I-II and II-III transitions amounting to an average of 0.71 and 0.55 k-atm, respectively. The magnitude of the hysteresis in the transitions increases with increasing pressurization rates.

4) The final structure is the same regardless of initial structure or whether the specimen had passed through the I-II or the I-II and II-III transitions. This residual structure is polycrystalline and contains many small isolated grains. This structure will become single crystalline as a result of a low-temperature short-time anneal that has little effect on unpressurized polycrystalline material. This enhancement of boundary migration is attributed, in part, to the release of stored elastic strain energy resulting from the anisotropy of the linear compressibility.

5) The formation of Phase II, which is polycrystalline, from Phase I occurs independently of any original grain boundaries. In the case of partial transformation, the orientation of Phase I formed from Phase II upon decreasing pressure is effectively the same as the original Phase I in the untransformed regions.

6) Under equilibrium, or near equilibrium, pressurization, there is a substantial waiting period at pressure before initiation of transformation. Under these conditions, the actual transitions will require up to several minutes for completion in a manner indicating a nucleation and growth-type transformation mechanism. Under faster pressurization rates, the transformation may be completed in as little as a few seconds.

ACKNOWLEDGMENT

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