X-RAY IDENTIFICATION OF THE DEFORMATION TENSOR IN Bi MONOCRYSTALS AND $Bi_{1-x}Sb_x$ ($x \leq 0.1$) ALLOYS WITH STRONG ANISOTROPIC DEFORMATION IN ANNULAR RINGS

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All of the components of the tensor of deformation of ε_{ij} are acquired using a method of X-ray diffractometry at liquid nitrogen temperature and the nature of the stressed state of samples of monocrystalline Bi and $Bi_{1-x}Sb_x$ ($x \leq 0.1$) alloys, deformed in annular rings using a method which has been widely used to create strong anisotropic deformations in semimetals, is determined.

Work [1] describes a technique for strong anisotropic deformation of monocrystals which has been successfully used in investigating the band structure [2 and 3], the electron topological transitions [2 and 4], and the propagation of magnetoplasma waves [5 and 6] in Bi, Sb, and their alloys.

This technique includes locking the slippage planes by rigid attachment of the disk shaped sample in an annular ring subjected to stretching by two concentrated forces F (Fig. 1). The locking of the planes of the easiest slippage (the (111) plane in semimetals) advances the elasticity limit into a range of much greater deformations. It is substantive that the deformation takes place at low temperatures.

Despite the large volume of different experimental data acquired using the method in [1], the question about the size and nature of deformations in such a system, in essence, remains unanswered. This is associated with the difficulties of direct measurement of deformation in small samples at low temperature using resistance strain gages. On the other hand, calculation of deformations in a sample-ring system performed within an isotropic planar problem in the theory of elasticity, which was used until recently [1 and 2], contains a number of assumptions, such as exclusion of the layer of polymerized Araldit resin which fills the gap of ~20 μm between the sample and the ring from examination, as well as the potential for inelastic effects at the stress concentration points. The reason for performing the X-ray investigation was the impossibility of quantitative description of the deformed state using the tensor of deformation calculated in [1], in particular, the absence of any sort of agreement between the acquired components of the tensor of deformational potential and the available literary data [7 and 8]. Since the technique [1] has already been quite widely used, experimental identification of the nature of the deformations created using it is required.

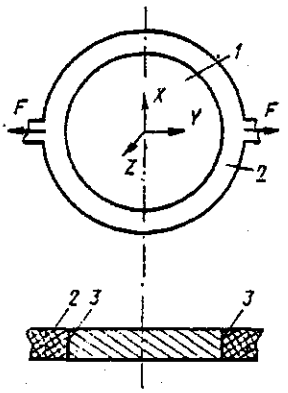

Fig. 1

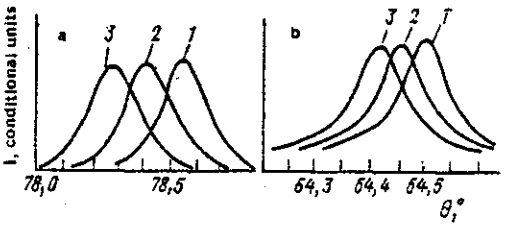

Fig. 2

Fig. 1. A device for anisotropic deformation [1]: 1) an annular ring with a diameter of 4 mm and a thickness of 0.8 mm made of 40KhNYu nonmagnetic steel; 2) a disk shaped sample with a diameter of 3 mm and a thickness of 0.75 mm glued into the ring (the triangular axis C_3 is parallel to the Z axis), and 3) a layer of Araldit resin.

Fig. 2. The shift of the diffraction maximums for copper radiation in the $F \| C_3$ (a) orientation and for molybdenum radiation in the $F \| C_1$ (b) orientation (b) with a change in loading: a) the (5 5 5) plane; F = = 0 (1), 140 (2), and 280 N (3) and b) the plane (10 10 10); F = 0 (1), 100 (2), and 200 N (3).

The purpose of this work was to take direct low temperature X-ray measurements of the change in the diffraction angles from different crystallographic planes with deformation of samples of Bi and $Bi_{1-x}Sb_x$ ($x \leq 0.1$) samples using a technique from [1] and to identify using these data the specific values of the components of the deformation tensor in the investigated system.

Measurement results and the samples. The shift in the diffraction maximums of reflection of the X-rays relative to the deforming force, whose value could be changed, was investigated at liquid nitrogen temperature. Special low temperature attachments to a DRON-2 X-ray diffractometer with a GUR-5 goniometer, whose layout is described in detail in work [9], were developed for this purpose.

Twenty-six samples of Bi and $Bi_{1-x}Sb_x$ ($x \leq 0.1$), whose elastic moduli in a range of small concentrations of antimony differed by not more than 3-4% [10], were investigated. The mirror reflecting surface (the (111) plane) was produced through shearing in liquid nitrogen. Just as in all of the previously performed investigations using the technique from [1], the samples differed in orientation of the crystallographic axes relative to the stretching force F: 1) $F \| C_2$; 2) $F \| C_1$. The above cited number of studied samples made it possible to collect satisfactory statistics for a sufficiently precise identification of ε_{ij} in both orientations.

The work was performed in the $K_{\alpha 1}$ line of copper and more rigid molybdenum X-ray radiation, where the $K_{\alpha 1}$—$K_{\alpha 2}$ doublet for the planes used ($\theta > 60°$) is permitted for more than 0.5°. For the Cu $K_{\alpha 1}$ and Mo $K_{\alpha 1}$ lines the penetration

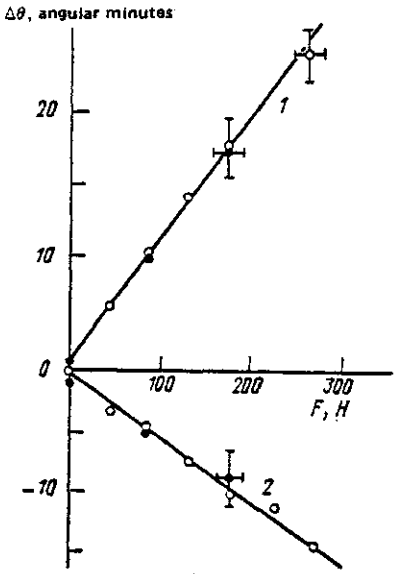

Fig. 3. Change in the diffraction angle from the planes (6 3 3) (1) and (5 5 5) (2) with deformation of a $Bi_{0.95}Sb_{0.05}$ sample (copper radiation and an FIC$_2$ orientation). The open points are acquired with application of loading and the darkened are with removal of the loading.

depth is $2.8 \cdot 10^{-4}$ and $5.2 \cdot 10^{-4}$ cm, respectively. Planes with rhombohedral indices of (10 10 10), (12 4 8), (4 12 8), (4 10 10), and (12 6 6) for molybdenum radiation and of (5 5 5), (2 5 5), (6 3 3), and (3 5 5) for copper radiation were investigated.

Several deformation cycles were performed in each sample; recording of the diffraction maximums was performed both with the application and with removal of the loading. As an example, Fig. 2 illustrates the shift of the diffraction maximum from the (5 5 5) plane for copper radiation and the (10 10 10) plane for molybdenum radiation in a sample of $Bi_{0.95}Sb_{0.05}$ with a change in loading. The dependence of the shift of the Bragg angle of reflection $\Delta\theta$ from the applied force F is linear and reversible (Fig. 3), which indicates the elastic nature of the deformation right up to F = (200-250) N. Graphs analogous to those cited in Fig. 3 are built for each loading cycle in each of the investigated samples. The slants of these graphs are used in solving a system of equations for determining ε_{ij}.

X-Ray identification of the deformation tensor from the change in the interplanar distances in the monocrystal is based on the fact that when the perpendicular to any plane (h, k, l) forms angles of α_i with the axes, in which the deformation tensor ε_{ij} is assigned, then the change in its interplanar distance $(\Delta d/d)^{hkl}$ is determined by the relation $(\Delta d/d)^{hkl} = \sum_{ij}^{3} \varepsilon_{ij} \cos\alpha_i \cos\alpha_j$. Having selected a sufficient number of crystallographic planes (1-6 depending on the number of non-zero components of the deformation tensor) in the deformed monocrystal, and having determined the change in their interplanar distances based on the change in the diffraction angle of the X-rays, it is possible to write a system of equations for finding the unknown ε_{ij}. The change in the Bragg angle $\Delta\theta$ and

Table 1

The Components of the Tensor of Deformations and Stresses in $Bi_{1-x}Sb_x$ ($0 \leq x \leq 0.1$) with Deformation in Angular Rings

ε_{ij}	$\varepsilon_{ij} \cdot 10^6$, N^{-1}			σ_{ij}	σ_{ij} in N/cm² at F = 10 N
	experiment, this work		theoretical calculation [1]		
	F ∥ C_2	F ∥ C_1			
ε_{XX}	4,2±0,6	4,1±0,8	14	σ_{XX}	56±35
ε_{YY}	−9,7±0,5	−9,8±1,0	−9	σ_{YY}	−404±35
ε_{ZZ}	3,2±0,3	3,3±0,6	−2	σ_{ZZ}	−6,0±20
ε_{ZX}	0,2±0,2	4,2±0,5	—	σ_{ZX}	0
ε_{ZY}	−4,4±0,3	0,1±0,3	—	σ_{ZY}	0,0±10
ε_{XY}	—	—	0	σ_{XY}	0

$\Delta d/d$ are associated with sufficiently small deformations by the relation $\Delta d/d = -\Delta\theta \operatorname{ctg}\theta$ [11]. The nonlinear corrections to this dependence in this work are less than 1-2% and may be ignored. In light of the symmetry of the studied system presented in Fig. 1 and the assumption that the shift deformation ε_{xy} is ignorably small in the central part of the sample, the changes in the interplanar distances must be determined in five different planes which are sufficiently slanted to the binary C_2, the bissector C_1, and the triangular C_3 axes of the crystal. It is also required that the Bragg angles of reflection of these planes θ be sufficiently large ($\theta \geq 60°$) since only in this case do the small deformational changes in the interplanar distances lead to a noticeable shift in the diffraction maximums.

Due to the design characteristics of the low temperature attachments used [9], five components of the deformation tensor ε_{xx}, ε_{yy}, ε_{zz}, ε_{xz}, and ε_{yz} for each was determined in a series of two deformation cycles: three planes which pass through the X axis were investigated and the components ε_{yy}, ε_{yz}, and ε_{zz} were determined in the first cycle and in the second, in a second type attachment, the planes which pass through the Y axis and the ε_{xx}, ε_{xz}, and ε_{zz} (repeated) components.

In each set of three planes one ("perpendicular") coincided with the surface of the sample (the "XoY" plane), while the other two ("the oblique") had different Bragg angles and slant angles ϕ to the surface of the sample equal in size, but different in angle. With such selection of the planes, the system of equations is simplified and has the form, for instance, for determining ε_{zz}, ε_{xx}, and ε_{xz} of:

$$\begin{cases} -\Delta\theta_n \operatorname{ctg}\theta_n = \varepsilon_{zz}, \\ -\Delta\theta_{k_1} \operatorname{ctg}\theta_k = \varepsilon_{zz}\cos^2\varphi + \varepsilon_{xx}\sin^2\varphi + \varepsilon_{xz}\sin 2\varphi, \\ -\Delta\theta_{k_2} \operatorname{ctg}\theta_k = \varepsilon_{zz}\cos^2\varphi + \varepsilon_{xx}\sin^2\varphi - \varepsilon_{xz}\sin 2\varphi, \end{cases}$$

where θ_n and θ_k are the Bragg angles of the "perpendicular" and "oblique" planes, while $\Delta\theta_n$, $\Delta\theta_{k_1}$, and $\Delta\theta_{k_2}$ are the changes in these angles with deformation. The system of equations for determining ε_{zz}, ε_{yy}, and ε_{yz} looks similar.

The acquired values of the components of the tensor of deformation are cited in Table 1.

In light of the isotropy of the elastic properties of Bi in a basis plane, the diagonal components ε_{ij} are identical at $F \| C_2$ and $F \| C_1$, while the shift deformations in accordance with the type of matrix of the constants of rigidity in Bi are different.

The components of the tensor of stresses σ_{ij} (Table 1) are calcualted by using Hook's law and the experimental values of ε_{ij} and the values of the components of the matrix of elastic constants from work [10]. The stresses coincide for the two orientations which is also caused by isotropy of the compressibility of Bi in the basis plane.

It should be noted that the values presented in Table 1 correspond only to the central part of a sample with a surface of approximately 1 mm^2. During the measurements the entire peripheral part of the sample was covered with lead foil (a mask) approximately 0.05 mm thick, which completely absorbs the X-ray radiation. Positioning an opening in the lead mask in different sectors of the periphery, it was possible to determine that near the point of force application (the X-periphery) and in a perpendicular direction (the Y-periphery) the values of the deformations differ by approximately 20-30% from the values in the central part, so that on the whole, the deformation throughout the sample is more uniform than follows from the calculation in [1].

The lack of change in the amplitude and form of the X-ray diffraction maximums with deformation, which is normally the case for the overwhelming majority of samples (see Fig. 2), indicates the high degree of uniformity of deformation in the central part of the sample. The fact that the values of deformations are the same for both types of radiation used is also significant. This indicates the absence of a noticeable gradient in deformation in the direction perpendicular to the surface and supports the fact that the deformational changes in the near surface layer are characteristic to the entire volume of the sample.

Discussion of the results. A noteworthy fact is that the deformations acquired for the central part of the disk do not coincide (with the exception of the ε_{yy} component) with the results of the theoretical calculation [1]: ε_{xx} is smaller by almost a factor of four times; ε_{zz} has a different sign and is a factor of 1.5 times larger than the calculated in absolute value; and ε_{zx} ($F \| C_2$) or ε_{zx} ($F \| C_1$) differ greatly from zero. The results of the calculation of σ_{ij} (see Table 1) show that a planar stressed state is actually realized in the central part of the studied system, as was hypothesized in [1]. However, unlike the calculation, the compressing stress σ_{yy} exceeds the stretching stress σ_{xx} by almost an order of magnitude, i.e., the nature of the anisotropic deformation corresponds more to monoaxial compression than to monoaxial stretching, as was previously proposed. A comparison of the calculated [1] and experimentally determined nature of the stressed state in the peripheral sectors of the sample shows that the stretching stress is not released in the thickness of the sample, but in a thin layer on the boundary of the sample and the ring. In fact, even in sectors of the X-periphery where, according to the calculation, stretching stress σ_{xx} must dominate, in fact the stretching stress σ_{yy} dominates.

The most probable cause of this is the presence of a thin (approximately

15-20 μm) layer of the polymerized Araldit resin, unconsidered in the calculations, which links the sample with the ring. In those sectors where the layer is subjected to compression (the Y-periphery) it fully transmits the compressing force to the sample, while with stretching (the X-periphery), it does not provide rigid attachment between the sample and ring, as a result of which the stretching stress is released. More likely, this occurs as a result of layering off of the resin in the small range of the maximal stress concentration near the points of force F application, which is not evident to the naked eye. The probability of plastic effects in the samples themselves in this range is apparently unlikely in light of the linear dependence $\Delta\theta(F)$ (see Fig. 3), although the release of stretching stresses due to the formation of microscopic cracks along the secondary junction planes ($\bar{1}11$) in the field of the X-periphery with loads of $F < 10$ N must not be completely ruled out. On the whole, however, the general nature of deformation in the sample cannot be attributed to simple monoaxial compression to which the nonzero value of the σ_{xx} component which is beyond the measurement error indicates.

Calculation of the components of the tensor of the deformational potential for electrons and holes based on data about the change in the energy spectrum of bismuth with deformation in the investigated system [2] with the use of the values of ε_{ij} from this work produces good agreement with the results of works [7 and 8]. It should be noted that the values of ε_{ij} determined in a system with dimensions as indicated in the subscript to Fig. 1, apparently, will be valid for samples of any diameter with preservation of the similarity of the entire system as a whole and an increase in F in accordance with its transverse dimensions.

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