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Effects of anisotropy and annealing on microhardness of $In_xBi_{2-x}Te_3$ ($x = 0{\cdot}1$ to $0{\cdot}5$) single crystals

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Abstract. The crystals of $In_xBi_{2-x}Te_3$ ($x = 0{\cdot}1$ to $0{\cdot}5$) have been grown by zone-melting method. In order to study anisotropy exhibited by the (0001) plane of the crystals, the directional hardness was determined by producing indentations at various azimuthal orientations of the indentor with respect to the surface over a range 0–180°. The crystal was rotated about the indentor axis in steps of 15° while keeping applied load and loading time constant at 50 g and 20 sec, respectively. For annealing study, the sample was kept at a temperature of 375°C. It was observed that softening of crystal takes place and the hardness decreases to a considerable extent.

Keywords. Anisotropy; annealing; hardness.

1. Introduction

The V–VI group compounds are low band gap semiconductors and known to find applications ranging from photoconductive targets in TV cameras to IR detectors (Arivuoli *et al* 1988). Among these, Bi_2Te_3 is the most potential material for thermo-electric devices (Jansa *et al* 1992). It crystallizes into hexagonal structure. Its melting point is about 573°C and has a direct band gap of about 0·16 eV. There has been an ample study reported on crystal growth and polycrystalline thin films of both pure and indium doped Bi_2Te_3 apart from the semiconducting, optoelectronic and thermoelectric properties (Drabble 1963; Sagar and Faust 1967; Testradi and Burstein 1972; Ha *et al* 1994). It has been shown that on exceeding a certain limiting concentration of indium in Bi_2Te_3 the conductivity changes from *p*-type to *n*-type, for $x = 0$–$0{\cdot}32$ (Jansa *et al* 1992). However, there are very few reports on microhardness of single crystals of Bi_2Te_3. This is particularly so in the case of indium doped crystals. Microhardness is a general macroprobe for assessing the bond strength, in addition to being a measure of the bulk strength. In this work the Vickers microhardness of $In_xBi_{2-x}Te_3$ ($x = 0{\cdot}1$–$0{\cdot}5$) single crystals as a function of applied load, temperature and orientation on the cleavage surfaces have been reported.

2. Experimental

The single crystals were obtained from stoichiometric mixtures of the respective elements of 5 N purity, using zone-melting method. The vacuum pressure used to seal the quartz ampoules containing the charge was of the order of 10^{-4} Pa and the growth velocity and the furnace gradient were kept at 3·5 mm/h and 45°/cm, respectively.

The specimens were in the form of 2–3 mm thick (0001) cleavage slices obtained at ice temperature to minimize deformation. A Vickers projection microscope with diamond pyramidal indentor was used to produce indentations on the (0001) plane and later on measure the same. The indentation diagonals were measured to an accuracy of 0·125 mm.

In order to study anisotropy exhibited by (0001) plane of the crystals, the directional hardness was determined by producing indentation at various azimuthal orientations of the indenter with respect to the surface over a range 0–180°. The crystal was rotated about the indentor axis in steps of 15° while keeping the applied load and loading time constant at 50 g and 20 sec, respectively.

For annealing study, the sample was sealed in an ampoule at 10^{-4} Pa. It was kept in the furnace at temperature of 375°C for about 48 h and gradually cooled down to room temperature following which microhardness was calculated using the standard formula. At least three indentations for each loading time were produced. The results present averages of the data obtained.

3. Results and discussion

The surface anisotropic variation of H_v in the three cases are represented in figure 1 in the form of plots of H_v vs orientation angle, θ. The four-fold symmetry of the

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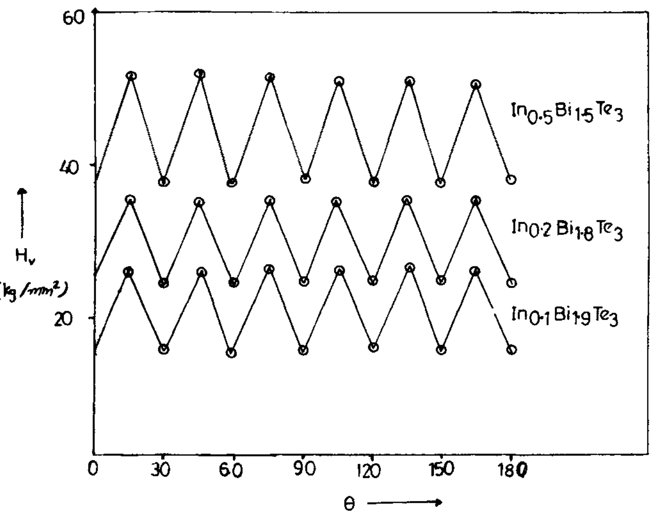

Figure 1. Plots of H_v vs θ.

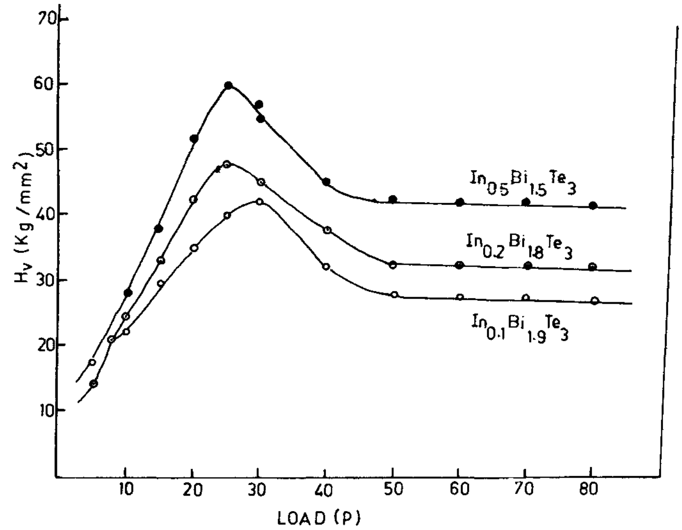

Figure 2. Plots of H_v vs P.

indentor and six-fold symmetry axis normal to the cleavage plane combine to result into twelve-fold symmetry. This is evident in the plot (Pandya *et al* 1977). The hardness values repeat at every 30° interval.

Figure 2 shows the plots of H_v vs load (P) obtained for samples annealed at 375°C. In the low load range the hardness remains dependent on load while at higher load it remains particularly independent of load. The complexity observed in the load dependence of hardness closely parallels many a report on variety of crystals (Buckle 1951; Bhatt *et al* 1983; Jani *et al* 1994). The hardness peaks are in turn explained in terms of the resulting deformation induced coherent regions. Beyond a certain depth of penetration, which corresponds to the expanse of the coherent region and to the load at the peak hardness, the indentor penetrates the virgin layers which easily favour nucleation and multiplication of dislocations (Braunovic 1973; Pandya *et al* 1977; Vyas *et al* 1995). It is observed (figure 2) that the hardness is independent of load for loads beyond 50 g and represents the true hardness of the bulk of the crystal. Accordingly, the hardness values of $In_{0.1}Bi_{1.9}Te_3$, $In_{0.2}Bi_{1.8}Te_3$ and $In_{0.5}Bi_{1.5}Te_3$ crystals are 27, 32 and 41 kg/mm^2, respectively. It is observed that softening of crystal takes place and the hardness decreases to a considerable extent as a result of annealing. Annealing is known to decrease dislocation density and to free immobile dislocation tangles, thus causing the plastic softening of the crystals.

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