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# Study of Vickers' Micro Hardness on $InBi_{0.85}Sb_{0.15}$ Single Crystals

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**Abstract**

$InBi_{0.85}Sb_{0.15}$ single crystals have been grown by zone-melting method. The growth velocity was 0.8 cm/h and the freezing interface temperature was 35 °C/cm. A Vickers' projection microscope was used for the study of micro hardness of the crystals. Hardness tests were carried out on the as-cleaved, cold-worked and annealed surfaces of the crystals. The effect of perfection of crystals on micro hardness with respect to the cold-worked, as-cleaved and annealed crystals have been studied and the results are reported.

**Key Words:** Vickers' micro hardness, Cold-worked crystals, Annealed crystals, Zone-melting method.

## 1. Introduction

For metals and alloys, the crystal growth from melt, such as Bridgman and zone-melting techniques, has been widely used to obtain large-sized crystals. Such methods have been used by different authors [1–5] for InBi single crystal growth. This intermetallic compound belongs to the tetragonal system, having $c/a = 0.955$ with a vary low melting point of 109 °C. InSb, like InBi, is a III-V compound, but its properties very significantly from those of InBi. The lattice of InBi is close to cubic type in terms of its unit cell parameters. Therefore it is expected Sb can substitute Bi in the InBi lattice. Indeed, Sb has been added to study its effect on crystal perfection and hardness of InBi [6]. This paper reports microhardness has been found to be load dependent in low load range (LLR). The crystals were cold-worked and annealed and, following these treatments, the nature of load dependence on micro hardness has been studied. The results have been explained in terms of deformation-induced coherent regions.

## 2. Experimental Procedure

The stoichiometric amounts of elements such as In, Bi and Sb all of 5N (99.999%) purity were sealed in quartz ampoules under pressure of $10^{-5}$ Torr. The melting point of $InBi_{0.85}Sb_{0.15}$ is 129 °C. The ampoule containing the charge was placed in a horizontal alloy-mixing furnace at a temperature of about 175 °C for 50 h. The ampoule was continuously rocked and rotated for proper mixing and reaction. The ingots were then cooled to room temperature over a period of 24 h. the crystals were grown by zone-melting method under a temperature gradient of 35 °C/cm and a lowering rate of 0.8 cm/h.

$InBi_{0.85}Sb_{0.15}$ crystals thus obtained were cleaved along (001) planes at ice temperature. For the cold-worked treatment, the cleaved samples were compressed for 24 h under a load of 1.5 kg. The crystals were then cleaved to obtain 1–2 mm thick slices. Microhardness measurements were carried out on the cleavage planes using a Vickers' pyramidal diamond indenter. The indentations were made at different loads ranging from 1.0 gm to 100 gm for fixed azimuthal orientations of the indenter to avoid anisotropic variations. For annealed samples, the samples were placed in an ampoule at $10^{-4}$ Torr pressure. The temperature of the annealed sample was kept at 90 °C for 24 h, then cooled to room temperature. The Vickers' microhardness measurements were studied on the annealed samples in the same range as those cold worked. Indentation time was 30 seconds for cold-worked, annealed and as-cleaved samples. All necessary precautions were taken to see that the experimental errors in the measurements were as small as possible.

## 3. Results and Discussion

Figure shows the Vickers' hardness number (VHN) as a function of load for the cold-worked and annealed crystals. For comparison, also shown is a similar plot obtained for the untreated crystal (denoted as-cleaved). Note the variation in hardness with load at low values; this is a common feature observed in all the plots. The hardness reached a peak and displays complex variation with increase in load, becoming constant at sufficiently high loads. The bulk characteristic hardness is usually represented by the value in the saturation region. Thus, it can be seen that the strain hardening due to the cold-work is more in the $InBi_{0.85}Sb_{0.15}$ crystal, raising the VHN 11.5% from 9.3 kg/mm$^2$ to 10.8 kg/mm$^2$. Softer the crystal, more mobile would be the dislocations resulting in the crystal, showing a higher tendency to strain-harden.

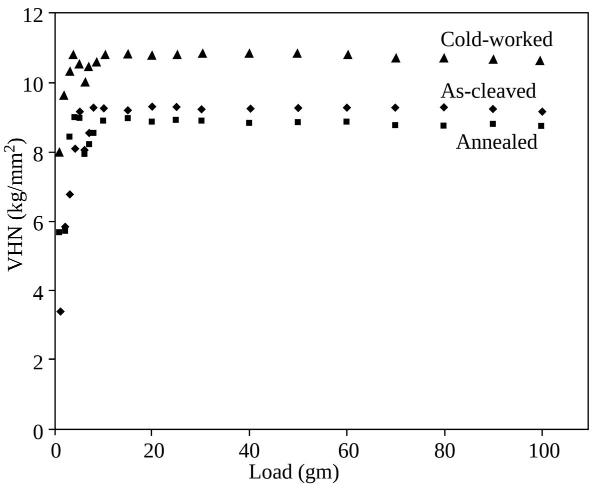


**Figure.** Plots of VHN versus load for $InBi_{0.85}Sb_{0.15}$ single crystals.

The effect of cold-working on low-load hardness may be explained as follows. The deformation at low load is normally accommodated by rosette dislocations creating intersecting jogs and dipoles with the progress of penetration by the indenter. Consequently, this structure becomes filled with complex network of immobile dislocations acting as strong barrier to the motion of new dislocations. At such depths, the plastic flow gets limited by the strength of interactions between the barriers and dislocations and the hardness exhibits a peak. The depth zone at which this occurs is known as deformation induced coherent region and is the characteristics of mechanical state of crystals [7–9].

From Figure, in the low load range, hardness load-dependent, while at higher loads it hardness is largely load-independent. The hardness value of $InBi_{0.85}Sb_{0.15}$ crystal obtained in this plot is 8.8 kg/mm$^2$, which is

9.4% less than the hardness observed in the as-cleaved sample. That is to say, annealing leads to considerable softening of the crystal. Annealing is known to decrease dislocation density and to free immobile dislocation tangles, thus causing the plastic softening of the crystals; this phenomena is largely dependent on dislocation mobility.

## 4. Conclusions

In the cold-worked sample, the independent hardness value increased 11.5% over the as-cleaved sample. In the annealed sample, the independent hardness value decreased to 9.4% of the as-cleaved sample.

The perfection of the crystals increased in the order: cold-worked, as-cleaved and annealed samples; as the hardness of the samples are reduced, respectively.

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