Microhardness and Electrical Properties a Bulk Crystal Grown from Mixture of Two Different Compositions of InSbBi Bulk Crystals

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Abstract- Vertical Directional Solidification (VDS) Technique was used for the bulk growths of $InSb_{1-x}Bi_x$ semiconductor without seed for x = 0.04 and x = 0.06. The source materials were filled with argon in the quartz ampoules at the pressure 200 torr. The ampoules were synthesized for 48 hr at temperature 850 ^oC. Temperature gradient at the solid- liquid interface was of the order of 20 $^oC/cm$. The ampoules were rotated at the speed of 12 rpm during the synthesis as well as the growth. The grown ingots were annealed at 250 ^oC for 48 hr. These two growths were carried out at the growth rate of 5mm/hr. In both the cases the composition of the source materials (Indium, antimony and Bismuth) in the grown crystals was estimated by EDAX analysis. An equal amount of InSbBi from each of the two ingots was filled in the new ampoule with argon pressure 200 torr and the growth was carried out at the growth rate of 3 mm/hr and rotation speed 12 rpm. The grown ingot was n-type semiconductor with mobility 1530 cm^2/Vs and microhardness 200 Kg/mm^2.

Index Terms- bulk crystal growth, microhardness, growth rate, microstructures

I. INTRODUCTION

Detectors for near infrared region and their operations at room temperature are of most importance in the devices used in military, medical diagnostics, pollution monitoring, industrial process controls, etc. [1] Thus there is extensive interest on the devices operating in the 8-12 µm wavelength region [2]. So far this field is being dominated by (MCT). But this material has some serious drawbacks, such as: (i) material parameters change with time [3] (ii) very unstable to the various external conditions [4], (iii) poor compositional uniformity over a large area due to high Hg- vapour pressure and (iv) weak Hg- bond, hindering the further development of infrared technology [5, 6]. As far as detector operations in near IR are concerned, problems mentioned above are the real challenges. These problems seem to be less severe for III / V alloys as compared to II / VI alloys. The only reason due to which II / VI alloys are still preferred is inability of conventional III / V alloys to reach higher end of near IR (12 µm) at the common operating temperature of 77 K [7]. Owing to these problems, $InSb_{1-x}Bi_x$ seems to be a strong contender to HgCdTe, which can operate even at room temperature. Incorporation of small amount of trace impurity of Bi into the host InSb lattice, changes the band gap from 0.18 eV to -1.5 eV to cover the NIR region (8–12 µm) [8]. Recent studies showed that, it is relatively difficult to grow ternary and quaternary large single crystals of high quality. Reported problems in such growths are: constitutional super cooling appear in front of the growth interface which results to sudden transition from single to polycrystals and local compositional inhomogeneity in the solid leads to cracking of the crystals [9]. In recent years, the growing field of semiconductor micromechanics has created an increasing demand for strength data on semiconductors and for adequate tests and evaluations of their mechanical properties [10] Microhardness is a good general measure of the mechanical properties, bond-strength, and structural stability. InSb1-xBix is one of the III-V psudobinary alloys suggested on the basis of stabilization by solution hardening of InSb [11].

II. EXPERIMENTAL

Bulk growths of $InSb_{1-x}Bi_x$ semiconductor without seed for x = 0.04 and x = 0.06 were carried out by Vertical Directional Solidification (VDS) technique [12]. Proper composition of the source materials were filled with argon in the quartz ampoules at the pressure 200 torr. The ampoules were synthesized for 48 hr at temperature 850 ^oC. Temperature gradient at the solid- liquid interface was of the order of 20 $^oC/cm$. To increase stirring effect, relative motion between the growing surface and the solution was employed by rotating the ampoule [13]. The ampoules were rotated at the speed of 12 rpm during the synthesis as well as the growth. The grown ingots were annealed at 250 ^oC for 48 hr. These two individual growths were carried out at the growth rate of 5mm/hr by lowering the ampoules in the temperature zone. In both the cases the composition of the source materials (Indium, antimony and Bismuth) in the grown crystals was estimated by EDAX analysis. An equal amount of InSbBi from each of the two ingots was filled in the new ampoule with argon pressure 200 torr. To improve the crystal quality the growth was carried out at the growth rate of 3 mm/hr [14] and rotation speed 12 rpm. The grown ingot was cut along the axis and perpendicular to the axis to obtain the substrates. The wafers were polished to mirror finishing by lapping and fine polishing [15]. These substrates were characterized by etching and microstructures, Hall measurements, mobility, optical FTIR, x-ray diffraction, SEM, EDAX and Microhardness.

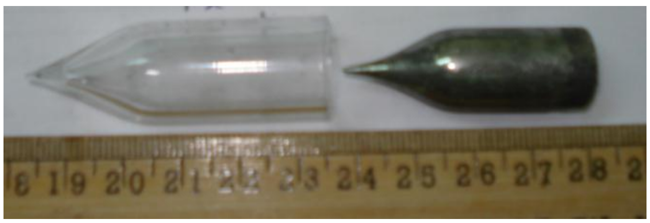
III. RESULTS AND DISCUSSION

One of the important factors of good quality crystal growth is detachment of the as grown ingot from the ampoule wall. Fig. 1

ISSN 2250-3153

shows just grown ingot in the ampoule easily comes out of the ampoule without having attachment with inner wall of the ampoule. This was verified by the uniform microstructures on the wafers and constant resistivity at different positions on the wafer even up to edge of the wafer.

(a)



(b)

Fig.1 Grown ingot in the ampoule and out of the ampoule.

A four point probe was used to measure resistivity. Average resistivity is $9.71 \times 10^{-4}\ \Omega$ cm and Hall coefficient 1.486 cm^3/C, with the mobility 1530 cm^2/Vs. Polarity of the Hall voltage showed that the grown crystal is n-type semiconductor. Linear variation of the Hall voltage with the applied magnetic field (from 1×10^3 gauss to 6×10^3 gauss) confirmed the homogenous crystal growth.

Optical FTIR patterns of one of the wafer of initial growth (x=0.04) and that of the mixed growth were obtained by FTIR (PERKIN-ELMER) in transmission mode (fig. 2).

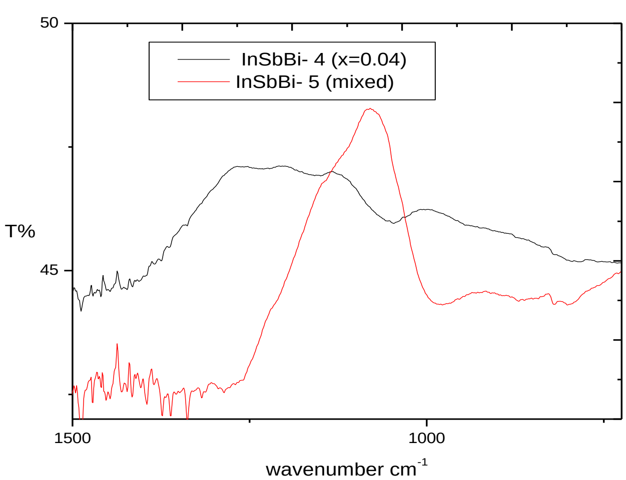


Fig. 2 .FTIR curves of one of the initial growth and the mixed growth.

The FTIR curves indicate the energy band gap of crystal grown from the mixture is 0.148 eV, while the band gap calculated from the variation of resistivity with the temperature is 0.150 eV. This slight reduction in the band gap from 0.172 eV shows small amount of Bi is incorporated in InSb lattice. X-ray diffraction (XRD) pattern of a wafer of the crystal revels that the grown crystal is polycrystalline.

Hardness of a wafer of pure InSb and the wafer of the as grown material were measured using a micro-hardness tester MODEL: 5 Auto, YOM: 2011. The indentation marks corresponding to the applied force F for the dwell-time 10s were obtained as shown in fig.3. Arithmetic mean d of the two diagonals in the indentation mark was calculated to obtain hardness. The hardness of pure InSb and InSbBi were found to be 200 Kg/mm^2. No appreciable change in the hardness was observed due to presence of Bi.

$$HV = 1.854 \frac{F}{d^2}$$

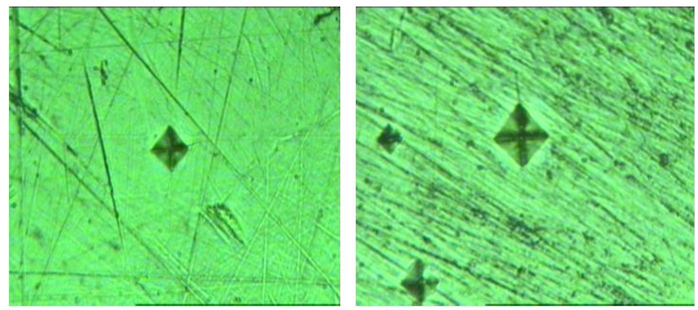


Fig. 3 Indentation marks on InSb and InSbBi wafers.

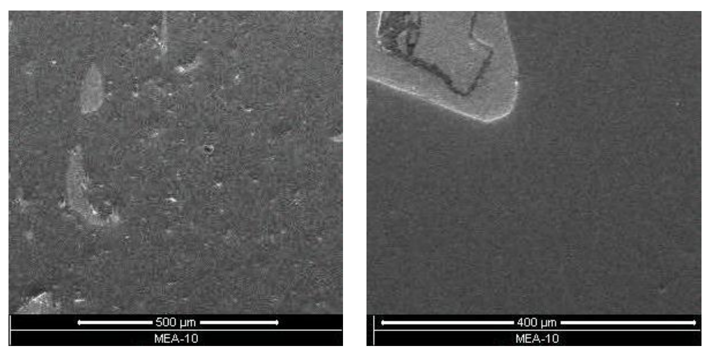


Fig. 4 SEM image of (a) first growth (x=0.04) and (b) mixed growth.

This Microstructures and SEM images of the wafers of the first two individual growths show more surface defects fig.4 (a) than the wafers of the crystal grown from the mixture of the two individual growths. Although segregation of Bi was observed in all the crystal growths.

IV. CONCLUSION

Hardness of the individual $InSb_{1-x}Bi_x$ compositions and the crystal grown from the mixture of the two compositions is nearly same but there is reduction in mobility from approx. 4500 Cm^2/Vs to 1530 Cm^2/Vs.

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