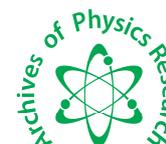




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Growth and X ray Characteristics of Dilute Nitride of Indium Antimonide

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ABSTRACT

The bandwidth of Indium antimonide can be reduced by a considerable value with addition of very small amount nitrogen. The resultant material will be suitable for infrared detection in long wavelength region (about 8-10 μm). This material that is, the dilute nitride of Indium Antimonide is grown by vertical directional solidification technique. In this technique the growth of a single crystal occurs without providing seed from outside. The Indium and antimony in pure form (6N) are mixed in Stoichiometric proportion and sealed inside a quartz ampoule. The ampoule is conical from the lower side. The ampoule is sealed at low pressure (200 torr) of argon. The growth temperature is 525 degree Celsius. The ingot is removed from ampoule and cut into wafers for further analysis. EDAX analysis confirms uniform distribution of nitrogen inside the ingot. The material shows n type behavior with large carrier concentration and low mobility compared to indium antimonide grown under same conditions. Using powder X ray method, lattice parameter is determined.

Keywords: Bulk crystals, InSb, EDAX, powder x ray

INTRODUCTION

The energy band gap of Indium antimonide is 0.17eV, which corresponds to band gap of 7 μm . The addition of nitrogen is expected to reduce the bandgap further. Thus the band gap of Indium antimonide can be suitably adjusted for detection of infra red radiation in 8-10 μm atmospheric transmission range. [1, 2] The bulk crystals of Indium antimonide nitride are grown in the lab using vertical directional solidification technique. This technique is known to give quality crystals of antimonide binary/ ternary compounds. The Indium antimonide crystals grown in the lab are of superior quality compared to other methods of growth. The method is especially advantageous for the growth of dilute nitride of indium antimony as it reduces the need for continuous flow of nitrogen gas and other complications in the design

MATERIALS AND METHODS

The bulk crystal of dilute nitride of Indium antimonide is grown by VDS. The Furnace is of 100 mm diameter. The temperature profile of furnace is as shown in figure1, where zero cm on y axis indicates bottom of the furnace and 30 cm above the bottom is the center of the tube. The heater is placed at the center outside the quartz tube chamber. The advantage of VDS-technique is that the unidirectional growth process can control the crystal nucleation and detached growth of the crystals inside the ampoule. Also, the crystal is grown without seed. [3, 4] The Indium, Antimony and Indium nitride powder were sealed in a quartz ampoule at pressure 10^{-3} bar. Presence of nitrogen, changes the band gap of InSb. For percentage above 1, the material will have negative band gap. Therefore the exact

percentage of Indium nitride powder was added to Indium and Antimony. High quality (In, Sb) (6N) were used as the source materials.

The material was kept for mixing at temperature 800°C for 18 hours for synthesis and homogeneous mixing of melt composition. There exist a temperature gradient inside the furnace as shown in figure 1 and ampoule is pulled down at a constant speed of 3mm/h. [5] To ensure uniformity of the composition, ampoule was rotated with the speed 10 rpm. Cr -Al thermocouple is used for temperature measurement.

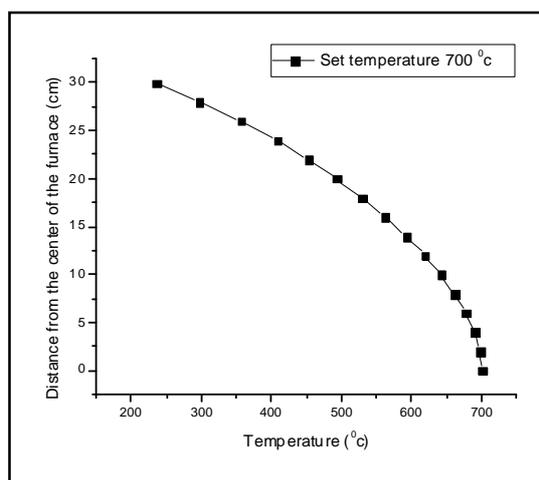


Figure 1 Temperature Profile of Furnace

The growth was started around 50°C above the melting point of InSb (525°C). About 30mm crystal was grown as shown figure 2 and figure 3. At 350°C, the downward movement of the crystal was halted. The crystal was kept at this temperature for 10 hours before removing from furnace. The crystal was found detached from the quartz ampoule on growth. As a result of which it was easy to remove from the quartz ampoule. The process of removal of the crystal from the ampoule is shown in figures 3. It can be easily seen that the crystal is removed without breaking ampoule wall. It is observed that quartz ampoule is grown detached in a closed quartz ampoule.

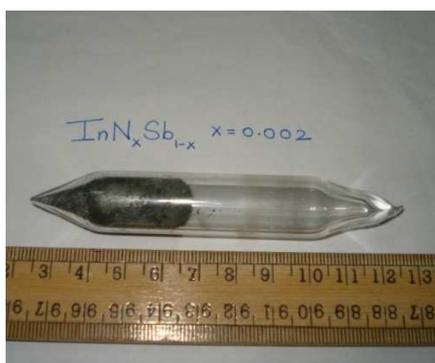


Figure 2 Crystal inside sealed ampoule



Figure 3 Detached ingot

We got single crystals of about 10 mm diameter which is slightly smaller than inner diameter of the quartz ampoule and 30-40 mm in length. Using this method three bulk crystals of dilute nitride Indium Antimonide are grown with nitrogen atomic percentage of 0.1% and 0.2% and 0.3% respectively.

RESULTS AND DISCUSSION

EDAX analysis:

Composition of all three samples is done using energy dispersive X ray analysis at ICON international laboratories at Mumbai branch. The analysis is done using electrons of energy 10-30kV. The peak on graph clearly indicates presence of nitrogen in the sample. The EDAX analysis shows uniform incorporation of nitrogen throughout the samples. The exact amount of nitrogen cannot be detected by this analysis as the method is not suitable for low atomic number elements like nitrogen. The analysis just proves the presence of nitrogen, for detail about composition other methods like electron probe micro analysis are utilized. With increase in composition of nitrogen, it can be observed that there is steady increase reflected in atomic and weight percentage. The tabulated values of Indium and Antimony are obtained using EDAX, where as nitrogen content is found using wavelength dispersive spectroscopy (WDS).

Table 1 composition of dilute nitride of InSb

Crystal	N %	In %	Sb%
I	0.1-0.25	44	45
II	0.2-0.25	42	40
III	0.1-0.3	45	47.7

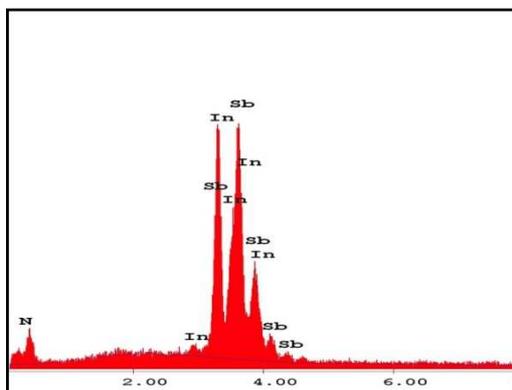


Figure 4 EDS analysis of InSb:N

Powder X ray analysis

From each sample fine powder is used for the powder x ray analysis. A typical graph of intensity versus 2theta is shown in figure1. With the presence of nitrogen though the shift in peak angle is only 0.15% for InSb1-xNx (x=0.002), there is change in intensity for every peak when compared with undoped InSb.

Table 2 powder x ray analysis

hkl	InSb	InSbN x=0.001	InSbN x=0.002
		Major peak	Major Peak
111	23.7701	23.8	23.9096
220	39.3098	39.36	39.4086
311	46.4563	46.58	46.5639
400	56.7790	56.92	56.9008
331	62.4416	62.46	62.5620
422	71.2108	71.38	71.3684
		76.54	76.64387

rom the table, we calculated interplaner spacing using formula given in equation1.

$$d_{hkl} = \frac{n\lambda}{2\sin\theta} \tag{1}$$

The lattice parameter for cubic InSb was calculated from each value of interplaner spacing using following relation for cubic crystal

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad (2)$$

Various systematic errors can happen while taking readings of intensity for different 2theta values such as use of a flat specimen instead of a curved one, absorption in the specimen, displacement of specimen from diffractometer axis. There are various extrapolation functions to take care of different errors, but no extrapolation function is completely satisfactory. A Nelson-Riley extrapolation function is considered here as it take care of most errors.[6] Figure5 shows Lattice constant versus Extrapolation function is plotted for sample with nitrogen content 0.5% and values obtained for the same and other samples from intercept on y axis are tabulated in the table. The lattice parameter of undoped InSb is given as 6.47 AU

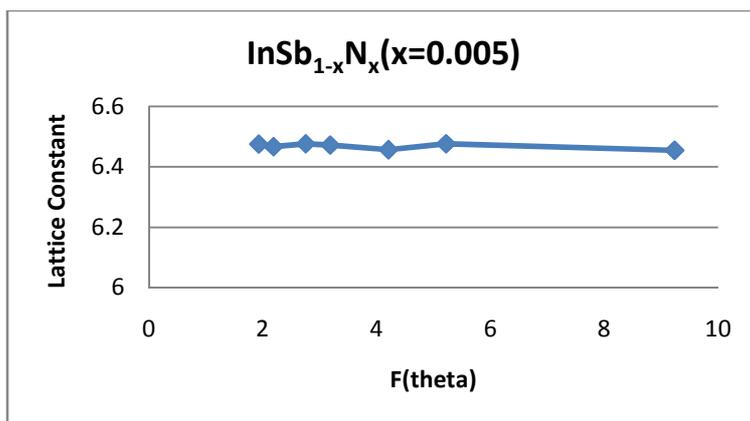


Figure 5 Lattice const vs. F (theta)

Table 3 lattice constants

InSb _{1-x} N _x	Lattice constant (a) in AU
X=0.001	6.48
X = 0.002	6.48
X = 0.005	6.4873

CONCLUSION

We could successfully incorporate nitrogen in bulk crystals of InSb though all ingots are not uniformly doped with nitrogen. Lattice constant was calculated from powder X ray analysis and increases marginally with increase in nitrogen content as shown in table3.

Acknowledgements

The powder XRD analysis is done at T. I. F. R., Mumbai.

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