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Study of Nitrogen Percent in InSbN Bulk CrystalGrown by VDS method

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Abstract*: Three bulk ingots of InSbN with 12mm diameter were grown using vertical directional solidification process with nitrogen composition 0.1%, 0.2% and 0.5%. The composition was studied using various methods. The EDS and secondary ion mass spectroscopy results indicated that the nitrogen was uniformly distributed throughout the crystal. The optical and electrical properties were studied. The optical and electrical properties varied with change in composition of nitrogen in Indium antimony.*

Keywords*: EDS; InSb:N, bulk crystal, semiconductors*

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I. Introduction

The low wavelength IR region of the spectrum is useful for detection of atmospheric gases, thermal imaging and infrared astronomy [1]. The dilute nitride doped Indium antimony (InSb:N) is sensitive to this range and is a suitable material for device fabrication for its high mobility and electrical resistivity [2, 3]. The antimony based devices work at room temperature against the mct based materials which work at low temperature. The bulk crystal of InSb:N is grown by vertical directional solidification technique. The material InSb has band gap 0.17 eV. Addition of nitrogen reduces the band gap grastically. Addition of 1% nitrogen reduces the bandgap by 100meV owing to small size of nitrogen. These highly mismatched materials are suitable for band gap modification and extensive study of the thin films of such materials can be found in literature [1]. However for device fabrication purpose, bulk crystals are requred.

Three bulk crystals of InSb:N were grown using vertical directional solidification technique in thw laboratory. The crystals were detached from wall. The detached crystals were polished and used for compositional analysis. The composition is calculated directly from electron dispersive spectroscopy (EDS) and the bonding between atoms is oberved with secondary ion mass spectroscopy (SIMS) analysis. The effect of composition on electrical and optical characteristics was studied elsewhere[4, 5].

II. Experimental

The crystals grown by VDS method are detached from ampoule wall after growth resulting in contactless growth [6] and the details of growth are also described in previous work [5, 6]. Three ingots of InSb:N were grown with nitrogen percentage 0.1% and 0.2% and 0.5%. The compositional analysis was performed on polished ingot along the axis as shown in Figure 1.

2.1 Compositional Analysis

Compositional analysis was performed using energy dispersive X- ray analysis. The electrical and optical characteristics changed as per the composition, confirming the results obtained by the EDS analysis. The SIMS analysis was performed to study the bonds formed by nitrogen in the compound.

2.2 Energy Dispersive Spectroscopic Analysis

The Energy Dispersive Spectroscopic instrument is an attachment to the scanning tunneling microscope.The scanned images are shown in Figure 1.

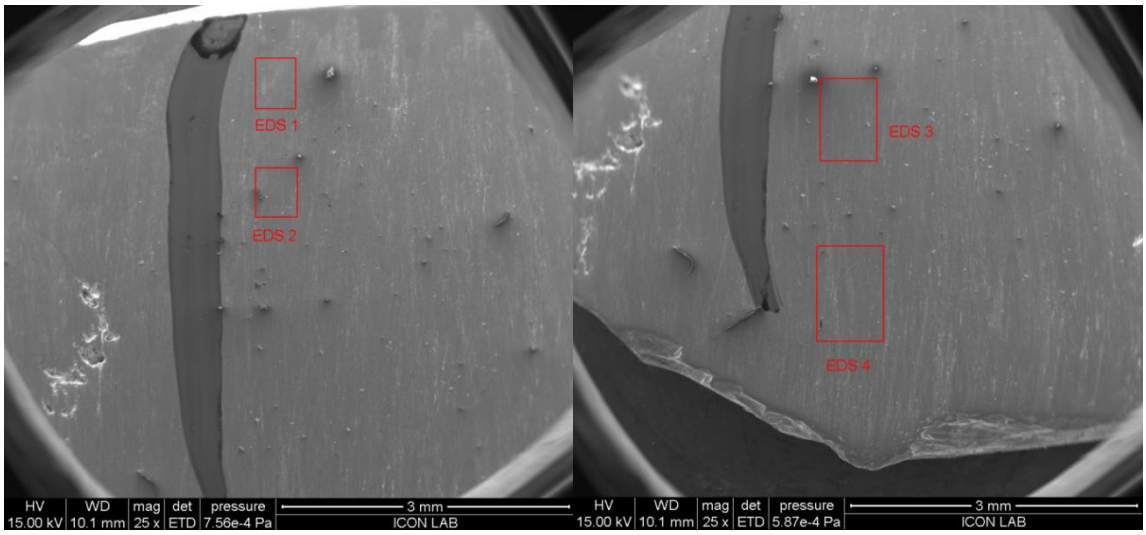

Figure 1 SEM images of InSb:N substrate

The electron beam in the electron microscope interacts with material under observation and generates characteristic X-rays. The material is identified from the energy or wavelength of the X- rays is generated. The EDS instrument identifies the element from the energy of the intensity peak and measures the K ratio of X-rays produced for each element in the compound. K Ratio is ratio of K series of X-rays emitted by different elements in the compound. K ratio for In in InSb:N will be defined as follows

$$K_{In} = \frac{I(In)}{I(In)+I(Sb)+I(N)+I(Impurities\ )} \qquad (1)$$

where I(In) is the intensity of all K series X rays emitted by the element In, I(Sb) and I(N) are the intensities of K series of X rays emitted by Sb and N and I(Impurities) is the intensities of K series of X rays of impurities present in the compound.The position ofEDS peak is indication of element whereas composition is deduced from the intensity of X rays.

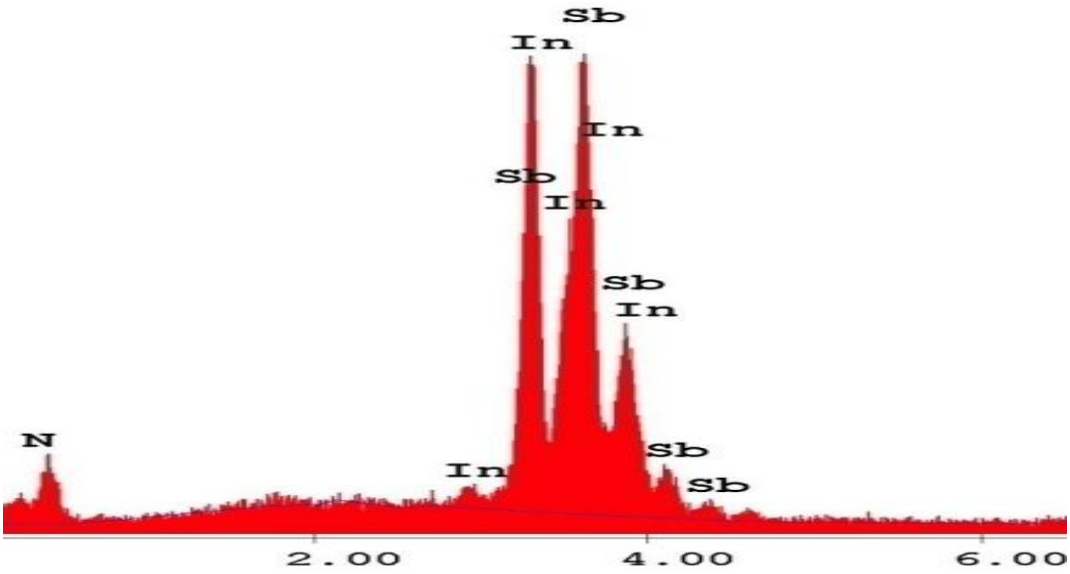

Figure 2 EDS scan of InSb:N sample

Special software DTSA II was used for quantifying and simulating EDS spectrum. The software is developed by the Surface and Microanalysis Science Division at the National Institute of Standards and Technology. The K ratio of given materials is compared with the K ratio of the standard materials using this software DTSA II. The absorption of X rays, backscattering and fluorescence are higher in the case of alloy as compared to those for pure elements. Hence it is more appropriate to use k ratio of alloys as standard value when the material under study is an alloy. Figure 2 shows the graph of sample InSb:N, N=0.5% as obtained

from EDS instrument whereas Table 1 gives all details of the analysis. From the comparison, the composition of the compound is determined. Thus k ratio of a dilute nitride was used as standard value to determine nitrogen percentage and k ratio of undoped InSb was used as a standard to determine Indium and Antimony percentage. The comparative graph of sample and simulation is shown in Figure 3. For the third sample with 0.5% nitrogen, the amount of nitrogen incorporated was 0.1% on average. For other samples the percentage of nitrogen varied from conical tip of ingot (minimum) to the cylindrical end of the ingot.

Table 1 EDS quantification with K ratio

	k ratio of In	Atomic %	k ratio of Sb	Atomic %	k ratio of N	Atomic %
1	0.4519	47.645	0.4790	52.261	0.0298	0.094
2	0.4485	47.843	0.4713	52.045	0.0352	0.112
3	0.4483	47.930	0.4694	51.955	0.0362	0.115
4	0.4525	48.032	0.4708	51.866	0.0335	0.103
5	0.4219	46.499	0.4661	53.420	0.0259	0.081

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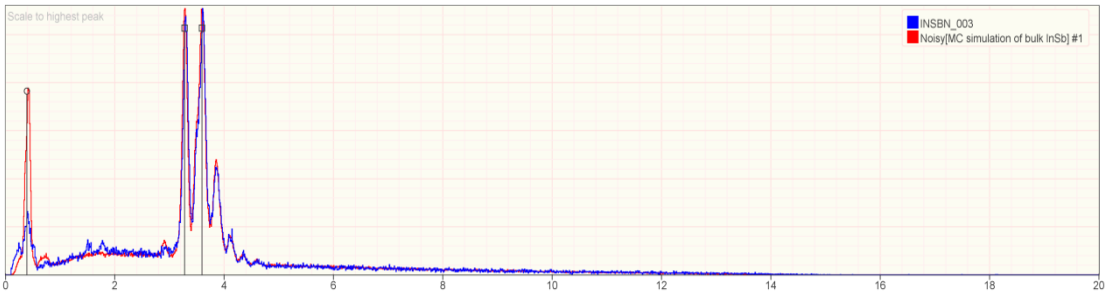

Figure 3 Simulation and actual EDS overlapped on the single graph

III. SIMS Analysis

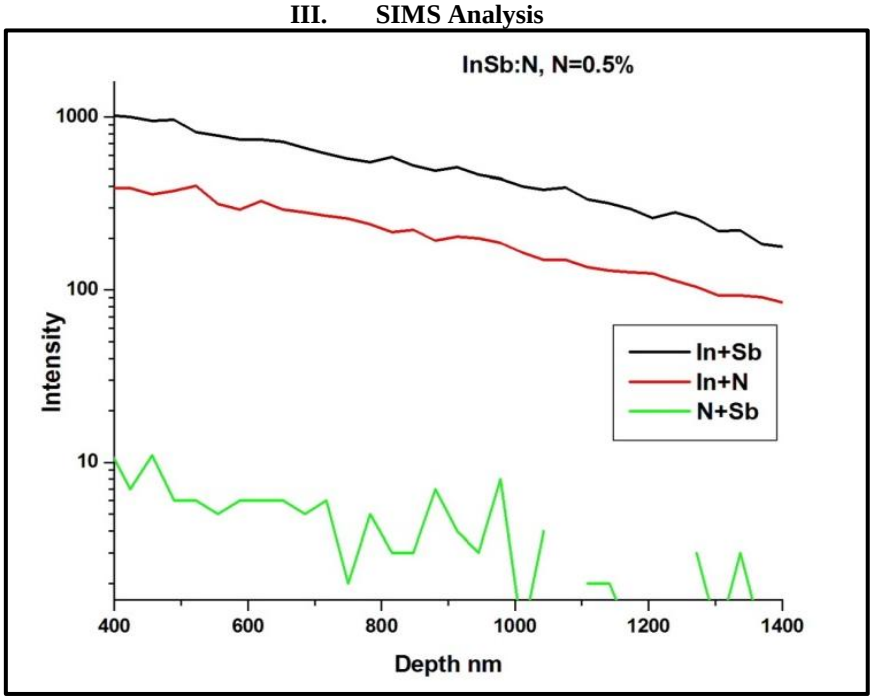

Figure 4 SIMS analysis of InSb:N

An oxygen ion beam is used for removing layers for depth profiling of atoms and molecules. The Gallium beam was used as primary ions for mass spectroscopy at a particular depth. The depth profile of crater created by

sputtering was measured using stylus profiler. Secondary ions generated during the process are analyzed in mass spectrograph as shown in Figure 4.

IV Conclusion

Information given by EDS instrument was successfully analyzed by DTSA II software to obtain composition for different growths of InSb:N crystals. The compositional analysis indicated that the indium and antimony were uniformly distributed in the ingot but nitrogen composition increased from conical tip of the ingot (0.29%) to cylindrical end. At the cylindrical section, the composition is in the range 0.35%, again slightly reducing in the end to 0.3%. The presence of nitrogen is confirmed using SIMS analysis qualitatively. Almost parallel lines for the different bonds (InN, SbN and InSbN) indicates uniformity of composition over different regions. This sample was from mid- section of the ingot and not from conical region.

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