CHAPTER-6

SUMMARY AND CONCLUSIONS

In this last chapter, a brief summary and conclusions have been discussed. The present study deals with the characterization and fabrication of n-InSb thin films. We have studied broadly three major things:

1) Composition dependent properties
2) Thickness dependent properties
3) Substrate temperature dependent properties

6.1 CONCLUSION OF COMPOSITION DEPENDENT n-TYPE InSb THIN FILMS.

The n-type InSb thin films have been deposited on borosil glass substrate at room temperature of 300 nm thickness by electron beam evaporation technique with use of different starting materials. The particle size (D), dislocation density (δ) and strain (ε) are change with increase of composition ratio (In/Sb) in starting material and thin films.

1. It observed here that the crystallites have zinc blende structures and orientation along the (111), (220), (311) planes.

2. It is observed that the crystallite size of crystallite, lattice parameter, strain and dislocation density in starting material and thin film are changing with composition ratio (In/Sb).
3. The big grain size 13.97nm, low dislocation density 5.12 x 10^{15} \text{line/m}^2, low strain 3.20 x 10^{3}\text{line}^2 \text{m}^{-4} have been observed for (111) plane in n-InSb thin film prepared with composition of starting material In_{0.66}Sb_{0.34}.

4. The low value of the dislocation density and strain in thin films planes indicate the formation of high-quality thin films of n-type InSb.

5. The increase in grain size of crystallites in thin film may be due to decrease in strain value or coalescence of small crystallites.

6. It is cleared from micrographs that the deposited n-InSb films are homogenous, without cracks or holes, well covered to the glass substrate and the size of crystallites is of the order of nanometers which are confirmed by X-ray measurement.

7. The composition of In and Sb is better for deposition of n-InSb thin film because the film have better structural parameter.

6.2 CONCLUSION OF THICKNESS DEPENDENT n-TYPE InSb

THIN FILMS.

The n-type InSb thin films have been deposited on borosil glass substrate at room temperature of 700-1300 nm thickness by electron beam evaporation technique with use of the starting materials In_{0.66}Sb_{0.34}. The particle size (D), dislocation density (\delta) and strain (\epsilon) are change with increase of thickness.

1. The crystallite size D increases from 28.64 nm to 84.40 nm when the thickness of the film was increased from 700 nm to 1300 nm. The 900 nm thickness of the thin film gives the largest grain size (84.51nm).
2. From the XRD data patterns, it is observed that the crystallites have cubic structure with preferential orientation along (111) plane.

3. The low line strain (1.74 X10^{-3} \text{ line}^2 \text{ m}^{-4}) and minimum dislocation density (1.40 X10^{14} \text{ linem}^{-2}) has been observed for film thickness of 900 nm.

4. The crystallites size increase from 700nm to 900nm thickness and after it very slowly decreases. This is happening because with increase of film thickness up to 900nm there is fast growth of crystallites and after 900nm, the growth is slow.

5. The dislocation density and strain value decreases which is due to improvement of crystallinity.

6. SEM images show that the films are uniform, homogeneous, polycrystalline, fully covered on substrate without crack or hole.

7. The n-InSb thin film deposited at thickness 900nm is the best among the other films at different thickness (700nm-1300nm).

6.3 **CONCLUSION OF SUBSTRATE TEMPERATURE DEPENDENT n-InSb THIN FILMS.**

Indium Antimonide thin films were deposited by the technique of electron beam evaporation with thickness 900nm with different substrate temperature 323K-473K.

1. The grain size increases from 85.99-92.23 nm. It is observed that the grain size of crystallite in thin films increase with increase of substrate temperature from 323K-398K and then it slowly decrease. It is due to increase of growth
of crystallites with increase of substrate temperature. The crystallites have cubic structure with preferential orientation along (111) plane. The larger grain size is 92.23nm for (111) plane.

2. The (111) plane has been taken because the (111) plane always gives the Gaussian curve which is helpful to determine the full width half maxima value and it also appears as the strongest peak in the XRD spectrum, that's why I showed the grain size only of the (111) plane.

3. The low strain 1.6 x 10^{-4}\text{line}^{-2}\text{m}^{-4} and low dislocation density 1.18 x 10^{14} \text{line/m}^2 has been observed, and which indicates better lattice arrangement.

4. The n-InSb thin film deposited at substrate temperature 398K is the best among the other films at substrate temperature range (323K-473K).

5. From Scanning Electron Microscopy, the images show that the films are uniform, homogeneous, polycrystalline, fully covered on substrate without crack or hole.

In view of experimental results, it is concluded that the improved structural properties are obtained by using optimized In_{0.60}Sb_{0.34} for electron beam evaporation technique at substrate temperature 398K because of the larger grain size 92.23nm, low strain 1.6 x 10^{-4}\text{line}^{-2}\text{m}^{-4} and low dislocation density 1.18 x 10^{14} \text{line/m}^2 have been obtained for this film. This is the best compare to other films which can be used in speed electronic device and optoelectronic device.

6.4 Future Scope of the Work

In the present thesis we have fabricated n-type InSb thin film with electron beam evaporation technique by varying the composition materials, the
thickness and substrate temperature of the film. Various characterization techniques were used to investigate the character and morphology of the thin films like XRD, EDAX and SEM. By using these methods we have optimized the composition of starting material and thin film. For industrial application point of view, InSb thin films are used to develop many electronic devices, sensors and detectors. InSb is a fascinating material because its high electron mobility is appropriate for high speed electronic device and Hall-effect devices. Its narrow band gap is also suitable for infrared applications. n- InSb are used for producing galvanomagnetic devices, optoelectronic devices for environmental control. InSb detectors have an extra benefit over conventional existing photoconductive cells. Because, InSb detectors are stable in air at least, many months. These detectors have resistances of about 1 to 10 k Ohm as compared with the 1 to 100 Mega Ohm related with PbTe cells. n-InSb can be use in bio-sensor which is to detect the bacteria.
Fabrication and characterization of \(n\)-InSb thin film of different thicknesses

S R Vishwakarma, Anil Kumar, R S N Tripathi, Rahul & Sasmita Das
Department of Physics & Electronics, Dr R M L Avadh University, Faizabad 224 001, India
E-mail: srvzkb@rediffmail.com

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The \(n\)-type indium antimonide thin films of the thickness 300-1200 nm were fabricated by electron beam evaporation technique on ultrasonically cleaned glass substrates at room temperature using optimized starting material. The X-ray diffraction patterns reveal that films are polycrystalline with zincblende structure. The dependence of structural, electrical and optical properties on film thickness was studied and optimized the film thickness. The Scanning Electron Microscope (SEM) micrographs show that the films are smooth and compact with larger grains. The electrical resistivity decreases \((0.66-0.13) \times 10^5 \ \text{ohm.cm}\) with increase of film thickness. These fabricated thin films show semiconducting behaviour because its conductivity increases with increase of temperature. The Hall effect measurement indicates that the films are of \(n\)-type having carrier concentration \((0.45-0.17) \times 10^{18} \ \text{cm}^{-3}\) and mobility \((2.11-30.42) \times 10^2 \ \text{cm}^2/\text{Vs}\). The direct band gap has been calculated by Fourier Transform Infrared (FTIR) transmission spectra recorded at room temperature which decreases from 0.22 to 0.19 eV with increase of films thickness.

Keywords: \(n\)-InSb thin films, Grain size, Resistivity, Carrier concentration, Direct band gap

1 Introduction

In recent years, the growth of InSb thin films has attracted attention as a potential material for infrared detectors and high speed devices because of small band gap. The III-V semiconductors, due to their structure, conventionally play a major role in scientific research and applications. Among the III-V binary compound semiconductors, indium antimonide shows \(n\)-type and \(p\)-type semiconductivity, polycrystallinity and melts at 525°C. It is a narrow band gap semiconductor\(^1\) \(0.17 \ \text{eV at 300 K}\) and \(0.23 \ \text{eV at 80 K}\). In \(n\)-type indium antimonide (due to anion vacancies), the electrons have high mobility \((80,000 \ \text{cm}^2/\text{Vs})\) due to their smaller effective mass. Similarly in \(p\)-type InSb (due to cation vacancies), the holes have the mobility 1250 \(\text{cm}^2/\text{Vs}\). Because of high mobility of charge carrier, InSb is used in magnetic-field sensing devices such as Hall sensor and magnetoresistors\(^2\), speed-sensitive sensors\(^3\) and magnetic sensors\(^4\). The infrared detectors are fabricated with \(n\)-type InSb thin films and sensitive between 3-5 \(\mu\text{m}\) wavelengths\(^5\). The \(n\)-type InSb \((n\)-InSb\) thin film can also used as bio-sensor to detect the bacteria. Many reports are available on the growth of InSb thin films using different deposition techniques such as molecular beam epitaxy\(^6\) (MBE), metal organic chemical vapour deposition\(^7\) (MOCVD) and vacuum evaporation\(^8\). The non-stoichiometry in structure or vacancies in film is created during deposition of thin film. The anion vacancies i.e. indium enriched thin film exhibit \(n\)-type semiconductivity while the cation vacancies exhibit \(p\)-type semiconductivity. The aim of present study is to fabricate \(n\)-type indium antimonide thin films of different thickness, therefore, prepared indium enriched non-stoichio-metric InSb powder of composition In\(_{0.60}\)Sb\(_{0.34}\). This is novel method to create controlled amount of non-stoichiometry in thin films. The composition of indium and antimony in starting material had been optimized as In\(_{0.60}\)Sb\(_{0.34}\) on the basis of electrical, optical and structural properties\(^9\). In present work, \(n\)-InSb thin films of different thicknesses have been fabricated by electron beam evaporation technique using aforesaid starting material. The electron beam evaporation technique is more suitable among physical evaporation techniques because during the deposition material directly comes into vapour state\(^10\). This paper reports variation of structural, electrical, optical and surface morphological parameters with film thickness. The film thickness has been optimized on the basis of these parameters.

2 Experimental Details

Before deposition of thin films, borosil glass substrates were boiled in chromic acid for two hours,
then washed using distilled water and rinsed by acetone. They were finally cleaned with distilled water in ultrasonic bath and dried at 423 K in oven. The n-InSb thin films of different thicknesses (300-1200 nm) were deposited at room temperature on cleaned glass substrate in Hind Hvac Vacuum Coating unit under a vacuum better than 10⁻⁵ Torr. The source distance from glass substrate was maintained at 125 mm for all cases. The non-stoichiometric starting material of composition In₀.₆₆Sb₀.₃₄ was used as source material. The deposition rate was maintained constant by controlling the electrical current of electron beam gun. The film thickness and deposition rate were measured by film thickness monitor (FTM) with quartz crystal sensor which is vibrating at frequency 6 MHz. The sensor of FTM has attached parallel to the substrate. The thicknesses of film have increased by increasing the deposition time.

These fabricated films were characterized for their structural properties by using D-8, Discover XRD-diffractometer (Bruker), using CuKα₁ radiation (λ=1.540598Å) with Ni-filter at University of Delhi, Delhi, India. The Scanning electron microscope (SEM) model Zeiss-EVO 18 (special edition) was used to study the surface morphology of these thin films at Indian Institute of Technology Delhi, India. The electrical resistivity of the samples has been measured using standard four probes technique. This technique is widely used for the measurement of electrical resistivity of thin films. A four probe measurement is performed by making four electrical contacts to a sample surface through probe arranged in set-up, two of the probes are used as source current and remaining other two probes are used to measure voltage. The advantage of the four probes is to eliminate the occurrence of errors due to the probe resistance and spreading resistance under contact resistance between metal probes and thin film. The Hall effect studied by applying a magnetic field perpendicular to the current direction (in surface of n-InSb thin film). Under such condition, Hall voltage is developed perpendicular to the current and magnetic field. The Hall effect parameters of fabricated films were measured using Hall set-up designed by Scientific Equipment and Services, Roorkee, India.

The optical property of thin films has been studied using optical transmission recorded by FTIR Spectrophotometer (spectrum 65, Perkin Elmer ) in the wavelength range 1000-6000 nm at Department of Physics, Banaras Hindu University, Varanasi, India.

3 Results and Discussion

3.1. X-Ray diffraction analysis

The X-ray diffractogram of n-InSb thin films of thicknesses 600 to 1200 nm were scanned in the 2θ range from of 20° to 70° as shown in Fig. 1. The XRD data were indexed with the help of JCPDS data file no (00-019-0577). The data was analyzed and found that n-InSb thin films were polycrystalline and crystallites are oriented along different planes i.e. (111), (220), and (311). From the XRD data patterns, it is observed that the crystallites have cubic structure with preferred orientation along (111) plane. The intensity of diffraction peaks increases with increase of film thickness.

The lattice constant (c) of n-InSb film of thicknesses 600 nm to 1200 nm for (111) plane was calculated using Eq. (1) and given in Table 1.

\[ c = d \sqrt{h^2 + k^2 + l^2} \]  

where d is inter planar spacing and (h k l) is Miller indices.

The grain size of thin films was calculated using diffractogram and Debye-Scherer’s formula.

\[ D = \frac{0.94 \lambda}{B \cos \theta} \] 

![Fig. 1 — XRD pattern of n-InSb thin films of different thickness](image-url)
where \( \lambda \) is wavelength of X-ray (1.540598 Å) and \( \theta \) is diffraction angle, \( \beta \) is full width half maxima.

The dislocation density \( (\delta) \) and line strain \( (\varepsilon) \) of films were calculated using equations\(^5\).

\[
\delta = \frac{n}{D^2}
\]

…(3)

where \( n \) is a factor, which equals unity giving minimum dislocation density.

\[
\varepsilon = \beta \cos \frac{\theta}{4}
\]

…(4)

The calculated values of grain size \( (D) \), dislocation density \( (\delta) \) and line strain \( (\varepsilon) \) of n-InSb thin films of different thicknesses (600 nm to 1200 nm) using diffraction pattern for (111) plane are listed in Table 1. It has been observed from Table 1 that the crystallite size \( (D) \) is increasing from 28.64 nm to 84.40 nm with increase of film thickness from 600 nm to 900 nm and after 900 nm thickness it very slowly decreases. This is happening because with increase of film thickness up to 900 nm there is fast growth of crystallite and after 900 nm growth is slow. The line strain \( (\varepsilon) \) decreases from 5.14×10^{-4} line^{-2} m^{-2} to 1.74×10^{-4} line^{-2} m^{-2} and dislocation density \( (\delta) \) also decreases from 12.19×10^{14} line m^{-2} to 1.40×10^{14} line m^{-2} with increase of films thicknesses up to 900 nm, which is also due to improvement of crystallinity. The lattice constant \( (c) \) was nearly equal for each film. The larger grain size (84.51 nm), the minimum dislocation density \( (1.40×10^{14} \text{line m}^{-2}) \), low line strain \( (1.74×10^{14} \text{line m}^{-2}) \) and lattice parameter \( (6.53 \text{nm}) \) have been observed for film of thickness 900 nm.

### Table 1 — Variation of structural parameters of n-InSb thin films

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>Thickness (nm)</th>
<th>Plane (h k l)</th>
<th>Lattice spacing ‘d’ (nm)</th>
<th>Grain size ‘D’ (nm)</th>
<th>Strain ( (\varepsilon) \times 10^{-4} ) (line^{-2} m^{-2})</th>
<th>Dislocation density ( (\delta) \times 10^{14} ) (line m^{-2})</th>
<th>Lattices constant ‘c’ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>600</td>
<td>111</td>
<td>3.77</td>
<td>28.64</td>
<td>5.14</td>
<td>12.19</td>
<td>6.52</td>
</tr>
<tr>
<td>2</td>
<td>700</td>
<td>111</td>
<td>3.77</td>
<td>32.80</td>
<td>4.49</td>
<td>9.30</td>
<td>6.53</td>
</tr>
<tr>
<td>3</td>
<td>800</td>
<td>111</td>
<td>3.77</td>
<td>50.46</td>
<td>2.92</td>
<td>3.93</td>
<td>6.53</td>
</tr>
<tr>
<td>4</td>
<td>900</td>
<td>111</td>
<td>3.77</td>
<td>84.51</td>
<td>1.74</td>
<td>1.40</td>
<td>6.53</td>
</tr>
<tr>
<td>5</td>
<td>1000</td>
<td>111</td>
<td>3.77</td>
<td>84.51</td>
<td>1.74</td>
<td>1.40</td>
<td>6.52</td>
</tr>
<tr>
<td>6</td>
<td>1100</td>
<td>111</td>
<td>3.77</td>
<td>84.49</td>
<td>1.74</td>
<td>1.40</td>
<td>6.53</td>
</tr>
<tr>
<td>7</td>
<td>1200</td>
<td>111</td>
<td>3.77</td>
<td>84.40</td>
<td>1.74</td>
<td>1.40</td>
<td>6.53</td>
</tr>
</tbody>
</table>

thin films of thickness 600 nm-1100 nm are shown in Fig. 2. It is clearly observed from the surface morphological study that the n-InSb films were homogeneous and well covered on the glass substrate. The nano-crystalline nature is confirmed by the morphology of the films as seen from SEM micrographs. The SEM images reveal that the crystallite size increases with increase of film thickness.

#### 3.3 Electrical analysis

Electrical resistivity \( (\rho) \) of n-InSb thin films of different thicknesses 300 nm-1100 nm was calculated using Eq. (5).

\[
\rho = \frac{2\pi SV}{CIT}
\]

…(5)

where \( C = \frac{2S}{\log 2} \), \( w = \text{thickness of the film and probe distance S= 0.2 cm.} \)

For this apply a constant current \( (I) \) through the outer pair of probes and measuring the voltage \( (V) \) between the inner pair of probes. The calculated resistivities are shown in Fig. 3. It is observed from Fig. 3 that the resistivity of n-InSb thin film decreases \( (0.66-0.13)\times10^{-2} \text{ohm-cm} \) for film of thicknesses 300-900 nm and after this resistivity remains constant for other thicknesses. The lowest resistivity \( (0.13)\times10^{-2} \text{Ohm-cm} \) is found for film of thickness 900 nm.

The electrical conductivity of films increases with increase of temperature i.e. films are of semiconducting nature\(^3\). The variation of \( \text{ln} (\rho) \) (Ω cm)\(^{-1}\)with (1000/\(T\)) K\(^{-1}\) for n-InSb thin films of different thicknesses (300-1200 nm) is shown in Fig. 4. The activation energy is calculated with the help of the slope of curves given in Fig. 4 and its variation with film thickness is shown in Fig. 5. It is observed from Fig. 5 that the activation energy of films decreases from 0.0675 eV to 0.0448 eV with

3.2 Scanning electron microscopic analysis

The Scanning Electron Microscopy (SEM) is a convenient and versatile tool to study microstructure of the thin film. The SEM images of fabricated n-InSb
Fig. 2 — SEM images of $n$-InSb thin films of different thicknesses
The lower activation energies were observed which shows that donor level lies just below the bottom of conduction band for each thickness of the film. Therefore, electronic conduction occurs in n-InSb thin films at room temperature due to transfer of electrons from this donor level to conduction band. The activation energy observed in present investigation is similar with that observed by Al-Ani et al.\textsuperscript{14}

The Hall effect measurement of all semiconducting InSb thin films shows that these films exhibit n-type behaviour. The carrier concentration in n-InSb thin films of different thicknesses (300-1200 nm) was calculated with use of Hall coefficient measured at room temperature and variation carrier concentration with film thickness is shown in Fig. 6. The carrier concentration decreases with film thicknesses of (300-900 nm) and very slowly increases for other thickness. The lowest carrier concentration (0.14) $10^{19}$/cm$^3$ was found for n-InSb thin films of the thickness 900 nm. The mobility of charge carriers in n-InSb thin films of different thicknesses (300-1200 nm) is calculated with help of measured values of Hall coefficient and resistivity. The variation of electron mobility with film thickness is shown in Fig. 7. It is observed from Fig. 7 that mobility of electron increases with increase of film thickness up to 900 nm due to decrease in the grain boundary scattering and mobility slowly decreases after 900 nm due to minute decrease in grain size. Also maximum mobility is observed at film thickness of 900 nm which has larger grain size. The observed values of carrier concentration and electron mobility are in good agreement with the observations of n-InSb films by other researchers\textsuperscript{15-17}.

The observed carrier concentration is affected by the parallel conduction at the surface. Therefore, the

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**Fig. 3** — Variation of Resistivity with thickness of film

**Fig. 4** — Variation of ln($\rho$) (Ω cm)$^{-1}$ with reciprocal of temperature for film of different thicknesses: (a) 300 nm, (b) 400 nm, (c) 500 nm, (d) 600 nm, (e) 700 nm, (f) 800 nm, (g) 900 nm, (h) 1000 nm, (i) 1100 nm, (n) 1200 nm

**Fig. 5** — Variation of activation energy with thickness of film deposited at room temperature

increase of film thickness up to 900 nm due to shifting of donor level towards the bottom of conduction band. After 900 nm film thickness, the activation energy becomes nearly constant because shifting has stopped.

**Fig. 6** — Variation of carrier concentration with thickness of n-InSb film deposited at room temperature
measured value in thin films is higher than that of the bulk material. The decrease in carrier concentration with increase of film thickness is due to the defect caused by the lattice mismatch of the materials. The high carrier concentration and low mobility are observed in the present study, because films are deposited at lower substrate temperature which associated large number of their structural defect.

3.4 Optical analysis

The transmission data for n-type InSb film of different thicknesses (700-1100 nm) is recorded using FTIR spectrometer. From the transmission data, the absorption coefficient ($\alpha_{\text{tr}}$) is calculated for all films in the strong absorption region using the relation:

$$\alpha_{\text{tr}} = \frac{1}{d} \ln \left( \frac{1}{T} \right)$$

where $\alpha_{\text{tr}}$ is absorption coefficient at particular wavelength, $T$ is transmittance at same wavelength and $d$ is film thickness.

The calculated FTIR absorption spectra for n-type InSb thin film of different thicknesses (700-1100 nm) are shown in Fig. 8. It is clear from these spectra that the absorption increases with increase of film thickness which is due to grain boundary scattering of infrared radiation. This increase of absorption with film thickness is related to grain size of crystallite in the films which is increasing with film thickness. It has also been observed from absorption spectra that the absorption is decreasing with increase of wavelength. The absorption rapidly decreases from wavelength 2500 to 5000 nm which indicates $n$-InSb is more active in this region.

The optical energy band gap of thin film is calculated by using Tauc relation:

$$\alpha_{\text{tr}} = A(hv - E_g)^n$$

where $hv$ is photon energy, $E_g$ is optical band gap, $A$ is constant, $n = \frac{1}{2}$ for direct band gap material.

The indium antimonide (InSb) is a direct band gap material, therefore, variation of $(\alpha_{\text{tr}})^2$ with photon energy for $n$-InSb thin film of different thicknesses (700-1100 nm) is as shown in Fig. 9. The extrapolation of straight-line portion, gives the value of direct band gap. It is found that the direct band gap decreases (0.22-0.19 eV) with increase of film thickness. This decrease in direct band gap with film thickness is due to reduction in the number of unsaturated defects. These unsaturated defects decrease localized density of state in the band structure. The minimum direct band gap (0.19 eV) is
observed for \textit{n}-InSb thin film of 900 nm thickness. The band gaps observed in present investigation are similar with that observed by other researchers\textsuperscript{19-20}.

\section{4 Conclusions}

Indium antimonide thin films were deposited by the technique of electron beam evaporation within the thickness 300-1200 nm. Hall measurements indicate that the films were of \textit{n}-type, having carrier concentration \(\sim 10^{18} \text{ cm}^{-3}\) and mobility \(\sim 10^3 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}\) for the film thickness of 300-1200 nm. It is also observed that the carrier concentration (\(n\)) and resistivity (\(\rho\)) decrease while the Hall mobility (\(\mu\)) increases with the increase of film thickness. Optical band gap varies from 0.19 to 0.22 eV with film thickness. Thin films having 900 nm thickness is better because it has maximum electron mobility, lowest resistivity and activation energy.

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\section{References}


Study of structural property of $n$-type indium antimonide thin films

S R Vishwakarma*, A K Verma, R S N Tripathi, S Das & Rahul
Department of Physics & Electronics, Dr R M L Avadh University, Faizabad 224 001, India
*E-mail: svrfb@rediffmail.com
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In present study, the $n$-type indium antimonide (InSb) thin films of thickness 300 nm were deposited on glass substrate at room temperature under the high vacuum ~10$^{-3}$ torr using starting materials. The starting materials have been prepared under vacuum ~10$^{-3}$ torr in vacuum coating unit using indium (99.999%) and antimony (99.999%) metal powder as source materials with various non-stoichiometric composition as In$_{x}$Sb$_{y}$ (0.2 < x < 0.4). The Energy Dispersive Analysis of X-rays (EDAX) measurement provides the information of chemical composition (In:Sb) in thin films. X-ray diffraction studies of starting materials and thin films confirmed the formation of InSb with polycrystallinity and orientation of crystallites along the (111) and (220) planes. The surface morphological study of thin films by scanning electron microscope reveals the crystalline nature which was found to be in good agreement with the XRD crystallinity analysis. The particle size (D), dislocation density (d) and strain (c) have been evaluated using XRD data for the starting materials and thin films. It is observed from X-ray diffraction patterns and scanning electron micrographs that particle size, dislocation density and strain are changing with composition ratio (In:Sb) in starting materials and their thin films.

Keywords: InSb thin films, Particle size, Dislocation density, Strain, Lattice parameter

1 Introduction

In recent years, the growth of InSb thin films has attracted attention as a potential material for infrared detectors and high speed devices because of its small band gap. The III-V semiconductors, due to their structure, conventionally play a major role in scientific research and its applications. Among the III-V binary compound semiconductors, indium antimonide shows $n$-type and $p$-type semiconductivity, polycrystallinity and melts at 525°C. It is a narrow band gap semiconductor with an energy band gap of 0.17 eV at 300 K and 0.23 eV at 80 K. In $n$-type indium antimonide (anion vacancy), the electrons have high electron mobility (80,000 cm$^2$/Vs) due to their smaller effective mass. Similarly in $p$-type InSb (cation vacancy), the holes have the mobility 1250 cm$^2$/Vs. Therefore, InSb is a material available for magnetic-field sensing devices such as Hall sensor and magnetoresistors, speed-sensitive sensors, and magnetic sensors. The infrared detectors fabricated with $n$-type InSb thin films are sensitive between 3-5 μm wavelengths. These $n$-type InSb thin films can also be used as bio-sensor to detect the bacteria. Many reports are available on the growth of InSb thin films using different deposition techniques such as molecular beam epitaxy, metal organic chemical vapour deposition and vacuum evaporation. In the non-stoichiometry in thin films of indium antimonide created during their deposition. The anion vacancies i.e. indium enriched exhibit $n$-type semiconductivity and the cation vacancies exhibit $p$-type semiconductivity due to excess of antimony. The aim of present study is to fabricate $n$-type indium antimonide thin films, therefore, prepared indium enriched non-stoichiometric InSb powder with different composition of indium and antimony. This is a novel method to create controlled amount of non-stoichiometry in thin films. We have fabricated $n$-type InSb thin films by electron beam evaporation technique using starting materials which have controlled non-stoichiometric composition. The electron beam evaporation technique is more suitable among physical evaporation because during the deposition, materials come into vapour state without changing in liquid state. Finally, we have optimized the composition of starting materials on the basis of structural parameters.

2 Experimental Details

2.1 Preparation of starting materials

Indium and antimony metal powders of purity 99.999% have been purchased from Alfa-Aesar Ltd, USA. To prepare the non-stoichiometric starting materials for fabrication of $n$-type indium antimonide thin films, first we take different amount of In (indium) and Sb (antimony) metal powder using
composition In$_{0.2}$Sb$_{0.8}$ (0.2 ≤ s ≤ 0.4). For each composition, the indium and antimony metal powder were mixed by grinding with mortar rod and then mixed powder was heated at 50°C in vacuum unit (Hind Hivic Company Ltd., India) using molybdenum boat under a vacuum $2 \times 10^{-5}$ torr for ten hours and cooled up to room temperature in the same vacuum condition. This cooled mixture material again grinded with mortar rod and heated in same vacuum condition with enhanced temperature. This process was repeated five times with different temperature some higher than previous to each composition for formation of crystalline $n$-type InSb through solid reaction.

2.2 Preparation of InSb thin films

Before deposition of thin films, borosil glass substrates were boiled in chromic acid for two hours, then washed using distilled water and rinsed by acetone. They were finally cleaned with distilled water in ultrasonic bath and dried at 423 K in oven. The $n$-type InSb thin films of thickness 300 nm were grown on glass substrate at temperature 300 K. For this, prepared starting materials were taken in the graphite crucible and targeted by beam of electrons in vacuum ($\sim 10^{-5}$ torr), where the vacuum system equipped with liquid nitrogen trap. The source materials kept at a distance of 125 mm from the substrate holder. The deposition rates (0.5-18) mm/s were adjusted by changing the filament current of electron beam gun power supply. The thickness and deposition rate were measured by digital film thickness monitor (VICO, DTM-10) using a quartz crystal sensor set at 6 MHz.

2.3 Chemical analysis of thin films

The chemical compositional analysis of some representative thin films has been studied by Energy Dispersive Analysis of X-rays (EDAX) technique using scanning electron microscope Carl Zeiss EVO40 series from Advance instrument research facility, Jiwaharlal Nehru University, New Delhi.

2.4 Structural measurement of starting materials and thin films

The X-Ray diffraction pattern of starting materials obtained by X-Ray Diffractometer (Rigaku D/max-2200) using Cu$k\alpha$ radiation of wavelength 1.5404 Å at 40 kV and 20 mA in scanning angle between 20° to 90° at the Centre of Science & Nanotechnology, University of Allahabad, Allahabad, India. The X-Ray diffractogram of $n$-type InSb thin films have been obtained by Philips Analytical X-Ray diffractometer (PW-3710) using Cu$k\alpha$ radiation ($\lambda = 1.5418$ Å) with Ni-filter at 35 kV and 30 mA in scanning angle between 20° to 70° at Department of Physics, JMI University, New Delhi, India. The scanning electron micrograph (SEM) of $n$-type InSb thin films have been taken by Scanning Electron Microscope (Technai G2 300 kV) operated at 20 kV at Centre of Science & Nanotechnology, Allahabad University, Allahabad, India.

2.5 Optical measurement of thin film

Optical transmittance of $n$-type InSb thin film fabricated from starting material has composition In$_{0.50}$Sb$_{0.50}$ is recorded using Fourier transform infrared spectrophotometer (FTIR) model No.Jasco/4100 in wave number range 1000-3000 cm$^{-1}$ from Department of Physics, The M S University, Vadodara (Gujarat). From the transmission data, the absorption coefficient ($\alpha$) is calculated for deposited thin film in the region of strong absorption using the relation$^{11}$:

$$\alpha = 1 / d \left[ \ln \left( \frac{1}{T} \right) \right]$$

where $\alpha$ is absorption coefficient at particular wavelength, $T$ the transmittance at same wavelength and $d$ is film thickness.

The direct band gap of thin film is calculated by using Tauc relation$^{12}$:

$$\alpha h\nu = A (h\nu - E_g)^{2}$$

where $h\nu$ is Photon energy, $E_g$ is band gap, $A$ is constant, $n = \frac{1}{2}$ for direct band gap material.

3 Results and Discussion

The X-Ray Diffractogram of starting materials are shown in Fig. 1-10. In diffractogram, large number of diffraction peaks of InSb with different $(h k l)$ planes

![Fig. 1 — X-ray diffraction of starting material of composition In$_{0.50}$Sb$_{0.50}$](image-url)
Fig. 2 — X-ray diffraction of starting material of composition $\text{In}_{0.5}\text{Sb}_{0.5}$

Fig. 3 — X-ray diffraction of starting material of composition $\text{In}_{0.1}\text{Sb}_{0.9}$

Fig. 4 — X-ray diffraction of starting material of composition $\text{In}_{0.3}\text{Sb}_{0.7}$

Fig. 5 — X-ray diffraction of starting material of composition $\text{In}_{0.4}\text{Sb}_{0.6}$

Fig. 6 — X-ray diffraction of starting material of composition $\text{In}_{0.3}\text{Sb}_{0.7}$

Fig. 7 — X-ray diffraction of starting material of composition $\text{In}_{0.7}\text{Sb}_{0.3}$
of high intensity are present but few peaks of In and Sb with less intensity are also present. Thus, X-ray diffraction patterns confirmed the formation of InSb with polycrystalline nature in starting materials.

The X-ray diffractogram of \( n \)-type thin films, prepared by using starting materials, are shown in Figs. 11-15. These patterns confirmed the polycrystalline nature of the \( n \)-InSb thin films. From the XRD patterns, it has been observed that the crystallites have zinc blende structures with orientation along the (111) and (220) planes. The intensity of diffraction peaks of indium antimonide change with increase of the composition ratio of In/Sb in starting materials. The diffraction peaks of indium

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**Fig. 8** — X-ray diffraction of starting material of composition In\(_{0.33}\)Sb\(_{0.67}\)

**Fig. 9** — X-ray diffraction of starting material of composition In\(_{0.15}\)Sb\(_{0.85}\)

**Fig. 10** — X-ray diffraction of starting material of composition In\(_{0.75}\)Sb\(_{0.25}\)

**Fig. 11** — X-ray diffraction of \( n \)-type InSb thin films prepared using starting materials of composition In\(_{0.60}\)Sb\(_{0.40}\)

**Fig. 12** — X-ray diffraction of \( n \)-type InSb thin films prepared using starting materials of composition In\(_{0.85}\)Sb\(_{0.15}\)
and antimony also appeared. The crystallites in a polycrystalline material normally have a different orientation plane from its neighbours, a similar result has also been observed in present study. The orientation of the crystallites is randomly distributed with respect to some selected frame of reference. The diffraction patterns of n-type InSb thin films are highly oriented along (111) and (220) planes in the present investigation, a similar planes and results have been also observed by other investigators Tahar et al., Senathilokumar and Singh.

The experimental inter planer space (d) value recorded by diffractometer for starting and thin films, their corresponding (h k l) planes obtained with the help of Joint Committee on Powder Diffraction Standard (JCPDS card no. PDF#06-0208) data are given in Table 1.

The crystallite size (D) of the starting materials and n-type InSb thin films are calculated by Debye Scherrer’s formula \( D = \frac{0.94\lambda}{\beta \cos \theta} \) (3)

where \( \beta \) is the FWHM of diffraction peak, \( \lambda \) is wavelength of used X-ray and \( \theta \) is diffraction angle.

The strain value (\( \varepsilon \)) for n-type InSb thin films calculated for (111) and (220) planes by use of following relation and presented in Table 3.

\[ \beta = \frac{\lambda}{D \cos \theta} - \varepsilon \tan \theta \] (4)

The dislocation density (\( \delta \)) defined as the length of dislocation lines per unit volume of the crystal and its value calculated with help of crystallite size (D) for the starting materials and n-type thin films, using the following formula (5) and given in Tables 2 and 3.

\[ \delta = \frac{1}{D^2} \] (5)

<table>
<thead>
<tr>
<th>S. No</th>
<th>Diffraction angle (2θ), deg.</th>
<th>Observed 'd' value (Å)</th>
<th>Standard 'd' value (Å)</th>
<th>(h k l) plane corresponding to InSb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>23.64</td>
<td>3.751</td>
<td>3.740</td>
<td>(111)</td>
</tr>
<tr>
<td>2.</td>
<td>39.17</td>
<td>2.287</td>
<td>2.290</td>
<td>(220)</td>
</tr>
<tr>
<td>3.</td>
<td>56.66</td>
<td>1.619</td>
<td>1.620</td>
<td>(400)</td>
</tr>
<tr>
<td>4.</td>
<td>46.38</td>
<td>1.952</td>
<td>1.953</td>
<td>(311)</td>
</tr>
<tr>
<td>5.</td>
<td>76.33</td>
<td>1.247</td>
<td>1.247</td>
<td>(511)</td>
</tr>
</tbody>
</table>
The lattice parameter 'a' calculated for crystals of starting materials and n-type InSb thin films with the help of (111), (220) planes and following equation 15, are presented in Tables 2 and 3.

\[ d = a \sqrt{h^2 + k^2 + l^2} \]  
\[ \quad \text{...}(6) \]

where \( d \) is interplaner space.

The chemical compositional analysis data of InSb thin films are presented in Table 3. From Table 3, it can be seen that the composition of indium and antimony in thin film and starting materials are nearly equal and have minute difference approximately one per cent. This difference decreases with increase of indium in starting material and found to be minimum for InSb in composition of starting material.

It has been observed from Tables 2 and 3 that the crystalline size (\( D \)) of crystallite, lattice parameters (a) and dislocation density (\( \delta \)) in starting materials and thin films are changing with composition ratio (InSb) in starting materials and orientation of the planes. The strain value (\( \epsilon \)) observed in thin films is also varying with composition ratio in starting materials and orientation of the planes. The big grain (8.42 nm), large lattice parameters (6.512×10^{-10} m) and low dislocation density (1.41×10^{16} line/m^2) have been observed for (111) plane in InSb composition of starting materials. While big grain (14.20 nm), large lattice parameters (6.490×10^{-10} m) and low dislocation density (4.96×10^{15} line/m^2) have been observed for (220) plane in n-type InSb thin films prepared with composition InSb of starting materials. The minimum strain (2.14×10^{-3} m^-2) in InSb thin films prepared with InSb composition of starting materials.

The grain size and lattice parameter increase but dislocation density decreases when thin films prepared using same composition of starting materials with change of orientation of the plane. The increase in grain size of crystallites in thin films may be due to decrease in strain value or coalescence of small crystallites 15. Since dislocation density and strains are the manifestation of dislocation network in the films. The observed structural parameters of n-type InSb thin films in present study are found to be good agreement with the results observed by other investigators. 10,13,14 The low values of the dislocation density and strain in thin films for (220) planes...
indicate the formation of high-quality thin films\textsuperscript{13,15} of $n$-type InSb.

Scanning electron micrographs (SEM) have been used for the analysis of surface morphology $n$-type InSb thin films. The SEM pictures of $n$-InSb films on glass substrate are shown in Figs 16-20. It is cleared from micrographs that the deposited $n$-InSb films are homogenous, without cracks or holes, well covered to the glass substrate and the size of crystallites is of the order of nanometers which are confirmed by X-ray measurement. The variation of crystallites size with composition ratio in starting materials is also confirmed by scanning electron microscope patterns.

The indium antimonide (InSb) is a direct band gap material, therefore, variation of $(2h \nu)^{2}$ with photon energy for InSb thin films of thickness 300 nm fabricated with In$_{0.66}$Sb$_{0.34}$ composition starting

Fig. 16 — SEM of $n$-type InSb thin films prepared from starting materials of composition-In$_{0.66}$Sb$_{0.34}$

Fig. 17 — SEM of $n$-type InSb thin films prepared from starting materials of composition-In$_{0.62}$Sb$_{0.38}$

Fig. 18 — SEM of $n$-type InSb thin films prepared from starting materials of composition-In$_{0.64}$Sb$_{0.36}$

Fig. 19 — SEM of $n$-type InSb thin films prepared from starting materials of composition-In$_{0.66}$Sb$_{0.34}$

Fig. 20 — SEM of $n$-type InSb thin films prepared from starting materials of composition-In$_{0.66}$Sb$_{0.32}$
materials at room temperature is shown in Fig. 21. Extrapolation of straight-line portion to \((\theta h v)^2 = 0\) axis gives the value of direct band gap. It is found that the direct band gap 0.22 eV and band gaps observed in present investigation are found to be similar with observed by other investigators.\(^{1,3,15}\)

4 Conclusions
Non-stoichiometric InSb compound (anion vacancy) as starting materials of polycrystalline nature with zincblende crystal structure with (111) and (220) plane of orientation have been prepared using molybdenum boat in vacuum for deposition of \(n\)-type InSb thin films. The \(n\)-type InSb thin films have been deposited on glass substrate at room temperature of 300 nm thickness by electron beam evaporation technique with use of starting materials. These deposited films are polycrystalline and have zinc blende structure with same plane of orientation but (220) plane is preferred. A grain growth noticed with increase in composition ratio of indium/antimony in starting materials and thin films. Scanning electron microscopic study confirmed the smooth surface of films. The particle size (\(D\)), dislocation density (\(\delta\)) and strain (\(\varepsilon\)) change with increase of composition ratio (In/Sb) in starting material and thin films. The composition In\(_{0.60}\)Sb\(_{0.34}\) is better for deposition of \(n\)-type InSb thin films because the films have better structural parameters, low direct band gap and minimum deviation of composition during deposition of thin films.

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Reference