Growth and characterization of n-type indium antimonide thin films structure deposited by electron beam evaporation technique

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ABSTRACT

The indium antimonide having small band gap is an important material for IR detectors and sources. The indium antimonide thin film was deposited on glass substrate in the high vacuum chamber at pressure $10^{-5}$ torr. To prepare the starting material for fabrication of n-type indium antimonide thin films, it was grown under vacuum $10^{-5}$ torr in vacuum coating unit using Indium (99.999 %) and Antimony (99.999 %) metal powder as a source materials with various composition using formula $\text{In}_{1-x}\text{Sb}_x$ (where $x$ is variable having value 0.2 to 0.4) for preparation of different composition. X-ray diffraction studies of the samples and its thin films confirmed the polycrystallinity nature and show preferential orientation along the (111), (220) planes. The particle size (D), dislocation density ($\delta$) and strain ($\varepsilon$) were evaluated with XRD data of starting materials and its thin films. The particle size increased with increasing of boat temperature & composition ratio (In/Sb) while dislocation density and strain were decreased with increase of boat temperature and composition ratio (In/Sb).

INTRODUCTION

Among the compound semiconductors, Indium antimonide is a polycrystalline compound has melting point 525°C. It is a narrow band gap n-type and p-type semiconductor with an energy band gap of 0.17 eV at 300 K, 0.23 eV at 80 K. In n-type Indium Antimonide semiconductor the electron has high electron mobility (80,000.00 cm²/V.sec) due to their smallest effective mass. Similarly in p-type InSb semiconductor the hole has mobility 1250 cm²/V.sec and thus it is the best material available for magnetic-field sensing devices such as Hall sensor and magnetoresistors[1], speed-sensitive sensors[2] and magnetic sensors[3].Many reports are available on the growth of InSb thin films using techniques such as Molecular beam epitaxy(MBE), metal organic chemical vapour deposition and vacuum evaporation[4].

Of all the methods used to prepare InSb thin film, the electron beam evaporation technique is the very simple and inexpensive technique and can be used for large area deposition. Thin films of InSb prepared by electron beam evaporation techniques have difficulties in maintaining the stoichiometry because of large differ-
ences in vapour pressure of In and Sb.  

The indium antimonide infrared detectors are sensitive between 1-5 µm wavelengths. This material can also used as bio-sensor to detect the bacteria. The present article explains the effect of composition ratio (In/Sb) on the structural properties of thin films deposited by electron beam evaporation technique.

**EXPERIMENTAL**

**Growth of InSb compound**

The high purity Indium (99.999%) and Antimony (99.999%) metal powder have purchased from Alfa-Aesar Ltd. USA. To prepare the starting material for fabrication of n-type indium antimonide thin films, first we take different amount of In (Indium) and Sb (Antimony) metal powder using formula In$_x$Sb$_{1-x}$ (where x is variable from 0.2 to 0.4). For each composition/sample, the Indium and Antimony powder grinded with each other by mortar rod and then heated in vacuum coating unit (Hind Hivac Co.Ltd, India) using molybdenum boat under a vacuum of $2 \times 10^{-5}$ torr for ten hours and cooled at room temperature in vacuum. The cooled sample again grinded with mortar rod and again heated in vacuum at same pressure. This process is repeated five times with different temperature for each sample. The X-ray diffractograph of starting materials have taken by PC based X-Ray Diffractometer (model no. Rigaku D/max-2200).

**Preparation of InSb thin films**

Bulk InSb compound grown by above process and it was used to deposited thin film on glass substrate by electron beam evaporation technique under vacuum $10^{-5}$ torr. In this experiments bulk materials was taken in the graphite crucible and evaporated in vacuum ($\sim 10^{-5}$ torr) system equipped with liquid nitrogen trap. The distance between source material and substrate was kept...
125 mm. The source temperature and thus the deposition rate (0.3-18 nm/s) were adjusted by changing the electrical current flow and flow time. In this study the deposition rate was evaluated by thickness monitor using quartz crystal sensor (6 MHz) set up near the substrate. During deposition, the film thickness was always measured by the thickness monitor, and the film of 300 nm was prepared for characterization.

The structural analysis was made using X-ray diffractometer with CuK$_{\alpha1}$ radiation at 40 kV/20 mA using with wavelength of 1.5404 Å in angle region from 20° to 90° for starting materials and thin films both.

TABLE 1: Standard and experimental ‘d’ value of InSb

<table>
<thead>
<tr>
<th>S.No</th>
<th>(h k l) plane</th>
<th>Observed ‘d’ value (Å)</th>
<th>Standard ‘d’ value (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>(111)</td>
<td>3.751</td>
<td>3.74</td>
</tr>
<tr>
<td>2.</td>
<td>(220)</td>
<td>2.287</td>
<td>2.290</td>
</tr>
<tr>
<td>3.</td>
<td>(400)</td>
<td>1.619</td>
<td>1.620</td>
</tr>
<tr>
<td>4.</td>
<td>(311)</td>
<td>1.952</td>
<td>1.953</td>
</tr>
<tr>
<td>5.</td>
<td>(511)</td>
<td>1.247</td>
<td>1.247</td>
</tr>
<tr>
<td>6.</td>
<td>(531)</td>
<td>1.095</td>
<td>1.095</td>
</tr>
<tr>
<td>7.</td>
<td>(331)</td>
<td>1.486</td>
<td>1.486</td>
</tr>
<tr>
<td>8.</td>
<td>(422)</td>
<td>1.322</td>
<td>1.323</td>
</tr>
</tbody>
</table>

RESULTS AND DISCUSSION

Structural properties

Characterization of InSb compound

The X-Ray Diffractograph of starting materials obtained by X-ray Diffractometer operated at 40 kV/20 mA using CuK$_{\alpha1}$ radiation with wavelength of 1.5404 Å in angle region from 20° to 90° are given figure 1 to

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

5. The experimental ‘d’ value recorded by PC based diffractometer for each composition and there corresponding (h k l) planes obtained with the help of Joint Committee on Powder Diffraction standard (JCPDS) data are given in TABLE 1. The X-ray diffractographs of starting material show the formation of polycrystalline Indium Antimonide compound.

Characterization of InSb thin films

The X-Ray Diffractograph of thin films obtained by X-Ray Diffractometer operated at 35 kV/30 mA using CuK$_{\alpha1}$ radiation with wavelength of 1.54056 Å in angle...
region from 20º to 65º. The spectra confirm the polycrystalline nature of the prepared thin films. The crystallites in a polycrystalline material normally have a crystallitic orientation different from that of its neighbours. The orientation of the crystallites called the preferential orientation, may be randomly distributed with respect to some selected frame of reference. From the XRD patterns of InSb thin films, it has been observed that the film structure are found to cubic with predominant orientation along the (111) direction. The XRD result of thin films were compared with the XRD data of starting materials. As increases the ratio of In/ Sb in different composition show that the peaks of indium, indium antimonide increases while decreased the antimony peaks. The results are comparable with films prepared by other techniques[5]. Hence it is concluded that the polycrystalline InSb film can be grown highly oriented along (111), (220) planes by Electron beam evaporation technique.

From the XRD profiles, the inter-planner spacing \(d_{hkl}\) was calculated for the (111), (220) planes using the Bragg’s relation,

\[d_{hkl} = \frac{n\lambda}{2\sin\theta}\]

where \(\lambda\) is the wavelength of the X-ray used, \(d\) is the lattice spacing, \(n\) is the order and \(\theta\) is the Bragg’s angle.

The factor ‘d’ is related to \((h\ k\ l)\) indices of the planes and the dimension of the unit cells. The crystalline size \((D)\) of the starting materials & thin films were calculated with help of Debye Scherrer’s formula using the full-width at half-maximum (FWHM) \(\beta\) of the peaks expressed in degree[5].

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

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

The strain value \((\varepsilon)\) can be evaluated by using the following relation,

\[\varepsilon = \frac{(D/\cos\theta) - \varepsilon}{\tan\theta}\]

The dislocation density \((\delta)\), defined as the length of dislocation lines per unit volume of the crystal and has calculated by using the formula.

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

where \(D\) is crystalline size.

The lattice parameter ‘a’ calculated from the equation as,

\[d = a/(h^2+k^2+l^2)^{1/2}\]

where- \(h, k, l\) represent the reciprocal lattice plane and ‘d’ Interplaner- spacing.

The values of particle size, strain and dislocation density for the starting materials and thin films of different composition are shown in TABLE 2 & 3. It has been observed that the crystalline size increased with

<table>
<thead>
<tr>
<th>TABLE 2 : Structural parameters of starting material InSb have different composition.</th>
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<tbody>
<tr>
<td>Composition Ratio(In/Sb)</td>
</tr>
<tr>
<td>--------------------------</td>
</tr>
<tr>
<td>In0.62Sb0.38</td>
</tr>
<tr>
<td>In0.64Sb0.36</td>
</tr>
<tr>
<td>In0.66Sb0.34</td>
</tr>
<tr>
<td>In0.68Sb0.32</td>
</tr>
<tr>
<td>In0.70Sb0.30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TABLE 3 : Structural parameters of InSb thin film have different composition</th>
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<tbody>
<tr>
<td>Composition Ratio(In/Sb)</td>
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</tbody>
</table>
increases of composition ratio (In/Sb) which may be due to decrease in strain in strain value. The increase in particle size with composition ratio may be due to the coalescence of small crystallites. The dislocation density decreases with the increases of composition ratio. Since dislocation density and strain are the manifestation of dislocation network in the films, the decrease in dislocation density indicates the formation of high-quality films at higher composition ratio[6].

The observed ‘d’ value and their correspondence standard ‘d’ value of starting material InSb and its thin film with (h k l) plane are depicts in TABLE 1.

The values of crystalline size (D), dislocation density (δ), lattice parameter (a) of the starting materials of InSb (Indium antimonide) have different composition are given in TABLE 2.

The values of crystalline size (D), dislocation density (δ), strain, lattice parameter (a) of the thin film of InSb (Indium antimonide) have different composition are given in TABLE 3.

CONCLUSIONS

Stoichiometric InSb compound metal powder with cubic crystal was grown using molybdenum boat. The as grown InSb metal powder was used to deposited thin films on glass substrate and glass substrate maintained at the temperature 373 K. All the films deposited have shown the cubic structure with nearly maintain stoichiometry and indium, antimony and Indium antimonide peaks were obtained. A systematic grain growth was noticed with increase in composition ratio of indium and antimony in thin films. X-ray diffraction studies of the samples and thin films confirmed the polycrystallinity nature and show preferential orientation along the (111), (220) planes. The particle size (D), Dislocation density (δ) and strain were evaluated with XRD data of starting materials and its thin films. The particle size increased with increasing of boat temperature & composition ratio(In/Sb) while Dislocation density, strain were decreased with increase of boat temperature and composition ratio(In/Sb).

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REFERENCES