PREPARATION AND CHARACTERIZATION OF THIN FILMS OF COMPOUND SEMICONDUCTORS

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The preparation and characterization of thin films of compound semiconductors of III-V group elements has gained importance in the recent years as these compounds have been found useful as magnetic sensors, high speed electronic and galvanometric devices, infrared detectors in different wave length ranges. The electrical and optical properties of these semiconductors are widely investigated to understand their behaviour. Some of the III-V compound such as InSb, InP, GaAs and GaSb have high electron mobility which makes them useful for many optoelectronic applications. Although group IV semiconductors such as silicon and germanium are most widely used in device fabrication, their high melting points requires stringent conditions for their growth in bulk crystalline as well as crystalline thin film form. On the other hand compound semiconductors with similar energy gap can be produced at relatively lower temperatures. This is particularly important because low melting point of these semiconductors simplifies the conditions of preparations and hence the processing parameters for device purposes. Electrical, electronic and optical properties of compound semiconductors have been studied in detail by several workers, but little work has been done on the microstructural investigations of these compounds.

In the present study it is proposed to grow thin films of compound semiconductor such as indium antimonide (InSb) onto various substrates under different deposition conditions by thermal evaporation technique under high vacuum conditions. Efforts have been made to prepare the starting basic material indium antimonide semiconducting compound in bulk form by vertical directional solidification (VDS) method using high purity indium and antimony in the stoichiometric proportions.

X-ray diffractrometry (XRD) measurement of the as grown InSb compound were carried out by X-ray diffractometer using CuKa line. The surface morphology and the composition of as grown InSb compound were investigated using scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) system attached with SEM.

The as grown bulk InSb compound has been used as the source material to deposit thin films on to glass substrates and freshly cleaved single crystals of KCl, NaCl, KBr at different temperatures by thermal evaporation technique under high vacuum conditions. The thickness of the films have been varied from 500 to 1000A^{o}. Thin films of InSb deposited onto single crystals at different deposition temperatures have been used to investigate the microstructural features using transmission electron microscopy (TEM) technique. These films on investigation with TEM have provided very important and interesting information about the changes taken place in the films in respect of grain size, defects, orientation, different phases, nature of films (whether amorphous, polycrystalline or crystalline).

InSb thin films deposited on to glass substrate at different temperatures were used for the measurement of electrical resistivity with varying temperature. The variation in electrical resistivity with temperature was studied in detail for different deposition temperatures and the band gap determined using the slope of the resistivity versus temperature plot. The changes in microstructural features of the films and its effects on electrical resistivity has been investigated and discussed in detail.

Infrared transmittance of bulk compound and its thin films was measured using fourier transform infrared (FTIR) spectrometer in the wave number range 400 to 4000 cm^{-1}. The transmittance results of bulk compound and thin films were compared and the energy band was determined using transmittance data. An attempt has been made to correlate the IR- transmittance data with microstructural features associated with the thin films.

A systematic study of the electrical, optical and structural properties of InSb thin films deposited on to various substrates at different deposition temperatures by thermal evaporation technique under high vacuum conditions have been carried out and it is revealed that electrical and optical behaviour of these films depends on the corresponding structural properties. It is envisaged that such a study would be useful and would play an important role in deciding the use of these materials for different applications in the field of optoelectronics.

The total work presented in this thesis has been divided into six chapters as given below:

The first chapter describes the introduction and the review of the previous work carried out on III-V semiconductors with particular reference to the InSb. The work on the InSb thin films as investigated by various workers has been described. The motivation, objective and the scope of the present work have been briefly described.

The second chapter carries the details of experimental techniques used in the present work for the preparation and characterization of indium antimonide in the bulk form and its thin films deposited onto various substrates at different temperatures. The details of the preparation of InSb compound in bulk form as well as the thin films grown using bulk indium antimonide as source material by thermal evaporation technique have been given. The experimental arrangement of the dc four probe technique to measure the resistance versus temperature characterstics of the thin films has been described. Energy band gap of the as deposited as well as annealed films have been estimated from the slope of the resistivity versus temperature curve. The carrier type of the as grown bulk compound and the thin films have been measured by the hot probe method. The specimen preparation techniques for transmission electron microscope (TEM), scanning electron microscope (SEM), energy dispersive x-ray spectrometer (EDS), fourier transfom infrared spectrometer (FTIR) and x-ray diffractometer (XRD) investigations have been briefly mentioned. The characterization techniques used for the determination of surface morphology and structure, elemental analysis, identification of phases, determination of lattice parameters, microstructure and transmittance or absorbance spectra etc. have been given. The major instruments and their brief working principles used in the present work particularly SEM, EDS, XRD, TEM and FTIR have been described.

Conditions for epitaxial growth and microstructural investigations of the indium antimonide thin films have been described in chapter III of the thesis. Formation of high purity bulk InSb compound by VDS method and its elemental analysis and stoichiometry of In and Sb in the compound using EDS has been discussed in detail. Results of XRD measurement of the powder of the as grown InSb compound has been discussed in this chapter. Surface morphology and structure of the compound using scanning electron microscope have also been described. The microstructural features associated with the thin films of InSb deposited onto various substrates at different temperatures using bulk InSb compound have been characterized using SEM, TEM and EDS system and the results thus obtained are described in detail.

Thin film deposition of a cubic InSb compound was carried out onto KCl, NaCl and KBr substrates at 373K using thermal evaporation technique under high vacuum conditions. The details of interesting microstructural investigations of InSb thin films deposited at 373K have been presented in chapter IV. Fine grained microstructure, presence of moire fringes with varied spacings and the variety of electron diffraction patterns were the noteworthy observations of the films. The present analysis has brought out certain unusual microstructural characterstics, which are not in general obtained at other process conditions. Accordingly some of these features are explained by postulating the suitable mechanism to explain the intricacies involved during evolution of fascinating microstructures.

In chapter V , the effect of substrate temperature on electrical resistivity, transmittance and microstructural features of thermally evaporated InSb thin films under high vacuum conditions have been reported. Indium antimonide thin films deposited onto corning glass slides at different temperatures were characterized for the measurement of resistance with temperature using dc four probe technique. The variation of electrical resistance with temperature was investigated in detail for different deposition temperature conditions and the band gap was determined using the slope of the resistivity versus temperature curve. The effect of annealing of the films at high temperatures for two hours under high vacuum conditions were also studied for their electrical behaviour in detail. The changes in microstructural features of the InSb thin films and its effects on electrical resistivity have been discussed in this chapter. Thin films of InSb deposited onto freshly cleaved KCl and NaCl crystals were characterized for their optical properties using fourier transform infrared spectrometer (FTIR) in view of the usefulness of these films as an infrared detectors. Transmittance of the thin films deposited at different temperatures recorded with FTIR spectrometer was analyzed and correlated with structural properties, have also been described.

The conclusion of the present investigations and the challenges faced during the study have been summarized in chapter VI

Future scope of the work

The processing and characterization of group III-V class of materials has led significant applications in semiconducting industries. The present work has contributed a considerable degree of scientific input to the field. The structural and microstructural studies of InSb compound grown by vertical directional solidification technique and subsequent preparation of thin films by thermal evaporation has cited to explore many new findings. The growth optimization of process parameters during thermal evaporation, leading to the development of epitaxial thin films, has enriched the knowledge that how to prepare the thin films of desired microstructure and therefore to other properties.

The present work is exciting and the epitaxial InSb films can be utilized for making hall sensors and other magnetoresistive applications. The InSb films doped with other group III elements van provide the films with varying band gap and microcrystallinity under specific conditions. These films can also open up possibility of being used as various types of sensors and also the other similar applications.