ABSTARCT

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Title: **Growth and Characterization of Pure and Doped Metal Oxides Nano-structured Thin Films**

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The preparation and characterization of thin films of pure and doped metal oxide nanostructured thin films has gained importance in the recent years as these oxides have been found useful in technological applications. Metal-oxide-semiconductors have many advantages such as transparency due to their large band gap, high uniformity in large scale fabrication applications, environmental stability and high electron conduction property.

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

The first chapter describes the introduction and the review of the previous work carried out on metal oxide thin films with particular reference to the, tin oxide, In_2O_3 and ITO. The work on the tin oxide, In_2O_3 and ITO 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 tin oxide, In_2O_3 and ITO thin films deposited onto various substrates. These films were further annealed under oxygen atmosphere at different temperatures to grow high quality and single phase films. Thin films deposited onto glass and quartz substrate were used to investigate electrical, optical properties. Thin films synthesized in the laboratory under different conditions have been characterized by different characterization techniques such as transmission electron microscope (TEM), scanning electron microscope (SEM), energy dispersive x-ray spectrometer (EDS), 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, PL spectrometer, UV-Vis spectrometer and AFM have been described.

In chapter three, microstructural characterization and optical performance of nano-structured thin films (thickness ~ 90 to 100nm) of tin oxide synthesized under varied vacuum conditions and annealing temperatures have been described. Synthesis details associated with the preparation of Tin Oxide thin films have been described in detail. In addition to the preparation of Tin Oxide thin films, microstructural characterization of the as grown films for uniformity, homogeneity, particle shape, size and distribution, defects generated during synthesis of the films, phase identification, and orientation of the films using transmission electron microscope (TEM) have also been described. The results of the investigations carried out by using SEM, EDS, XRD, SIMS, AFM, PL and TEM have been described and discussed in details.

Fourth chapter presents the Microstructural and Optical characterization of indium oxide thin films prepared by using thermal evaporation technique under high vacuum conditions. Indium oxide thin films were prepared by thermal evaporation method under vacuum conditions using high purity indium (In 99.999% pure) as the source material. The pure element was procured from M/s Alfa Aesar. The thickness of the thin films was kept around ~100nm. Thin films of indium were deposited at room temperature under vacuum of the order of 10^{-5} mbar. As deposited films, on glass and quartz substrates, were annealed at different temperatures (150^o, 200^o, 250^o, 300^o, 350^o, 500^o and 700^oC) for 2 hr and 4hr under high purity oxygen gas atmosphere in order to synthesize good quality single phase indium oxide films. Structural and optical properties of these films have been investigated by using XRD, SEM, TEM, AFM, UV-Visible, and Photoluminescence spectroscopy respectively. Synthesis parameters such as temperature and time period of annealing of as synthesized indium oxide thin films have been optimized in order to get high purity, homogeneous, transparent and single phase thin films.

Fifth chapter describe the synthesis and characterization of tin doped indium oxide (ITO) thin films. Indium tin oxide is one of the most widely used transparent conducting oxides because of its two chief properties, its electrical conductivity and optical transparency, as well as the ease with which it can be deposited as a thin film. As with all transparent conducting films, a compromise must be made between conductivity and transparency, since increasing the thickness and increasing the concentration of charge carriers will increase the material's conductivity, but decrease its transparency.

This chapter describes the synthesis details of indium tin oxide (ITO) thin films prepared by using thermal evaporation technique under high vacuum conditions and further annealed at different temperature conditions. Initially the thin films of indium of thickness around 50nm were deposited on to single crystals and glass substrates at room temperature in the vacuum of the order of 10^{-5} mbar. After that, a thin layer of tin film of thickness 10nm, 20nm, 30nm and 40nm was deposited on already deposited indium layer. The layered structured is represented as super impose of tin on indium film (Sn/In) having final thickness: 60nm, 70nm, 80nm and 90nm. These films were further characterized by using XRD, SEM, AFM, Four probe, UV-Visible and Photoluminescence spectroscopy in order to understand the structural, electrical and optical properties associated with them. Parameters to synthesize uniform, homogeneous, single phase and transparent ITO thin films have been optimized and to be further used as gas sensors.

Sixth chapter describes the design and development of an indigenous gas sensor set up for thin films for sensing the presence of various gases such as methanol, ethanol and LPG gas. Sensing device is an instrument which reads the response and translates it into quantifiable and an interpretable term. After optimizing the synthesis parameters of In_2O_3 and ITO, these films were examined to be used as various gas sensors. In this chapter metal oxide gas sensing mechanism, working principle of metal oxide semiconductor based sensors, factors influencing the sensor performance and merits and demerits of metal oxide nanostructures as sensing material have also been explained in detail. Comparison of maximum electrical response of indium oxide thin films deposited at 300^oC, 500^oC and 700^oC in the exposure of methanol, ethanol vapors and LPG has also been presented in this chapter.

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