PREFACE

Growing industrialization and ever increasing pollutants from vehicular exhaust have resulted into increased air pollution. Further, use of cooking gas through pipe lines in modern houses, may become a severe fire hazard due to lack of proper gas-leak alarms. The problems related to air quality monitoring are important issues of the current research activity. In fact, a key component in many process controls, product development, environmental monitoring etc. is the measurement of concentration of one or the other gaseous component of the ambient. In such situations suitable sensors can provide the necessary interface between the ambient and the backup electronic instrumentation to detect the target gas. To replace the heavy, expensive and time consuming analytical systems used to detect pollutant gases, different kind of sensors have been developed, i.e., electrochemical sensors, polymer sensors, surface acoustic waves (SAW) sensors and metal oxide semiconductor (MOS) sensors etc. The last ones are mainly employed because of their simplicity, small dimensions, good performances and low cost. Unfortunately, they present the disadvantage of a cross-sensitivity for several gases and in addition they work at elevated temperatures. To overcome these problems we have deposited thin films of tin oxide, for gas sensor detection. The layers were deposited using a simple and cost effective deposition technique, i.e., Spray pyrolysis technique (SPT) compared with other techniques, which are more or less sophisticated and expensive.

The SPT was selected for the deposition of the films taking into account the advantages such as: simple and cost-effective set-up, high deposition efficiency, large choice of precursors, and ambient atmosphere operation. Besides, the technique provides an easy control of the surface morphology, by varying the deposition parameters such as: substrate temperature, time, flow rate, precursor concentration etc. The control of the surface morphology is of particular interest for gas sensor applications where a porous morphology is desired to increase the adsorption and implicitly the sensor response to a specified gas. The sensor materials are selected according with the literature in order to be sensitive for at least one of the studied gases (H_2S, ethanol, and hydrogen). Some of the films are doped with different materials in order to enhance their response to a specified gas. For example, SnO_2 is doped with copper oxide in order to increase the H_2S sensitivity while with chromium oxide to improve the ethanol response.

The surface morphology and microstructure are evaluated with different techniques, i.e., Scanning and Transmission electron microscopy (SEM and TEM), Energy dispersive analysis by X-ray (EDAX), X-ray diffraction (XRD), Atomic force microscopy (AFM) and UV-visible spectroscopy. The gas sensing performance in the detection of H_2S, ethanol, and hydrogen are studied as function of the operating temperature and the gas concentration.

A brief summary of the comprehensive report of the thesis and its distribution in different chapters is presented as follows.

The thesis consists of nine chapters. **Chapter 1** is 'General introduction and literature survey'. This chapter deals with a brief description of the chemical sensors and their classification. Furthermore, some general aspects concerning the gas sensors based on metal oxide semiconductors are more intensively discussed. Hence, the Metal Oxide Semiconductor (MOS) types, the conduction mechanisms and principal factors, which affect the MOS performance, are presented. The structure of the tin oxide used in this work is also highlighted.

Chapter 2 deals with the 'Experimental techniques for characterizations'. This chapter consists of details of various characterization techniques such as X-ray diffractometry (XRD), Scanning electron microscopy (SEM), Field emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM), Atomic force microscopy (AFM), Energy dispersive analysis by X-rays (EDAX), UV-visible spectroscopy and thickness measurements. This chapter also contains details of static gas sensing system.

Chapter 3 is devoted to the 'Synthesis, characterization and gas sensing performance of SnO_2 thin films: Effect of annealing temperature'. This chapter consists of details of experimental set up of spray pyrolysis technique for the preparation of SnO_2 thin films. The as-prepared thin films were annealed at different temperature and characterized further by XRD, SEM and UV-visible spectroscopy. The gas sensing performance of SnO_2 thin films to various gases were carried out in this chapter. The structural properties carried out by XRD, surface morphological studies carried out by SEM and optical properties carried out by UV-visible spectroscopy. The effects of annealing temperature on these properties were discussed in this chapter.

Chapter 4 deals with ‘SnO_2 thin film gas sensor: Effect of variation of applied voltage’. This chapter consists of preparation, characterization of SnO_2 thin film and the gas sensing characteristics were obtained by measuring the sensor response as a function of various controlling factors like operating temperature, operating voltages and concentration of gases. The results are discussed and interpreted with the help of XRD, SEM, TEM and EDAX.

Chapter 5 presents ‘Effect of precursor concentration and quantity of precursor solution on gas sensing performance of SnO_2 thin films’. This chapter discusses spray pyrolysis technique to prepare SnO_2 thin films with different conditions such as: change in precursor concentration and change in quantity of precursor solution. The as prepared films were characterized by XRD, AFM, TEM and UV-visible spectroscopy. The structural properties carried out by XRD, surface topographical properties by AFM, microstructural studies carried out by TEM and optical properties carried out by UV-Visible spectroscopy. The effect of precursor concentration and change in quantity of precursor solution on these properties as well as gas sensing performance was discussed in this chapter. The results are interpreted and discussed.

Chapter 6 is devoted to ‘Preparation, characterization and gas sensing performance of nanocrystalline $CdSnO_3$ thin films’. This chapter consists of details regarding preparation of nanocrystalline $CdSnO_3$ thin films by spray pyrolysis technique at a substrate temperature of 350 ^{o}C. The structural, surface morphological and microstructural properties were investigated using different techniques such as X-ray diffraction, FESEM, AFM and TEM. A gas sensor based on thin film was applied to ethanol sensing test as well as some other gases. Obtained results show that the sensitivity of nanocrystalline $CdSnO_3$ thin film sensor reaches 576.2 to 100 ppm ethanol with a response time less than 12 s. The results are discussed and interpreted in the light of the results.

Chapter 7 deals with ‘Preparation, characterization and gas sensing performance of Cr-doped SnO_2 thin films. This chapter explains Cr-doped tin oxide thin films prepared by spray pyrolysis technique (SPT). The hollow microspheres with a high Cr incorporation (10 at. %) was found to posses excellent sensitivity, selectivity and rapid response to the presence of ethanol vapors at low temperature. X-ray diffraction, UV, scanning electron microscopy, FESEM and energy dispersive analysis by X-ray were carried out to investigate the gas sensing mechanism. The optical study showed that the films have a band gap of 3.43 eV. The results are discussed and interpreted.

Chapter 8 deals with 'Preparation, characterization and gas sensing performance of pure and Cu-doped SnO_2 thin films'. Nanocrystalline pure and Cu-doped SnO_2 thin films were prepared using spray pyrolysis technique. These films were characterized by XRD and FESEM with EDAX to observe structural and surface morphological properties respectively. It is observed that FESEM images showed pyramid type structures. The sensing performances of pure and Cu-doped SnO_2 thin films were tested. The maximum sensitivity of was found for 3 wt % Cu-doped SnO_2 thin film.

Chapter 9 devoted to 'Scope for the future work'. This chapter explains the thin film gas sensors for chemical warfare agents (CWAs). CWAs are highly toxic and their use is restricted. Research on the environmental fate of CWAs is often conducted using simulant compounds. An ideal chemical agent simulant would mimic all relevant chemical and physical properties of the agent without its associated toxicological properties. Although a number of compounds have been used as CWA simulants, no individual compound is ideal because a single simulant cannot satisfactorily represent all environmental fate properties of a given CWA. Thus, a number of different chemicals will be used as CWA simulants depending on the physical and chemical properties of interest. A brief discussion regarding the preparation of nanostructured thin films by ultrasonic spray pyrolysis (USP) technique and possible applications of thin film gas sensor for CWA sensing is discussed in this chapter.

Annexures give the information of the publications and paper presented in national/international conferences on this work.