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Gas Sensing Studies of Tin Oxide Thin Films Annealed at Different Temperatures

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ABSTRACT

Thin films of Tin oxide (SnO_2) were prepared by physical vapour deposition method. The as-prepared films were further annealed at 300°C, 400°C and 500°C to study the effect of annealing on the physical as well as gas sensing properties of the thin films. Gas sensing performance of annealed SnO_2 thin films was studied for different gases having different concentrations at working temperature of 250°C. Significantly, gas response changes for SnO_2 samples annealed at different temperatures, which is discussed herein.

Keywords: SnO_2 Thin films, PVD technique, XRD, FESEM, EDAX, gas sensing.

Introduction

Monitoring and controlling the toxic gases has become a serious topic not only in industries but also in day to day life. These toxic gases are used either as process gases or generated as byproducts from vehicles, industries, etc. In concern to this, detecting the harmful gases is of prime importance and hence, research in developing gas sensors is at its forefront these days. The semiconductor metal oxide gas sensors like SnO_2, ZnO, WO_3 etc. have been studied due to their range of conducting variability and their strong response to reducing as well as oxidizing gases [1-3].

A variety of thin film preparation methods have been used by the researchers to synthesize metal oxide thin films in various nano-structured forms. Some of the synthesis methods are chemical vapour deposition [4], spray pyrolysis [5], sputtering [6], activated reactive evaporation [7] etc. Physical Vapour Deposition (PVD) method is straight forward and simple one. It has several advantages [8] such as (a) Minimum impurity concentration in the film, (b) sublimation of materials at lower temperature under vacuum, (c) considerably large mean free path of the vapour atoms at lower pressure and hence a sharp pattern of the film is obtained, (d) wide substrate selection. Another advantage of this method is that evaporation yields a large number of films of uniform thickness. The thin film deposition takes place in four stages [9]. In the first stage the formation of the vapor phase from the condensed phase. The transport of atoms or molecules from the source to the substrate constitutes the second stage. The third stage involves the deposition of atoms on the substrate. The final stage involves rearrangement of the atoms on the film to give the desired properties for the product. Moreover, the post treatments like annealing, etc. are important, too in obtaining the desired phases and the stoichiometry. Annealing of films is a necessary step to control grain growth, altering the stoichiometry of the film, introducing dopants and oxidizing the film, inducing compressive stresses in the film are some of the reasons for post-film deposition processing [10-11].

Therefore, in the present paper, we prepared SnO_2 thin films on the thoroughly cleaned glass substrates. The so prepared thin film samples were then heated in muffle furnace for 24 hours to allow maximum oxidation. These samples were further annealed at different temperatures (viz. 300°C, 400°C and 500°C). The gas response of the samples were studied and discussed.

Experimental Details

The pre-cleaned glass substrates were mounted on the specially designed mask arranged at about 15 cm above the evaporator. A tungsten spiral filament used as an evaporator was screwed to two copper electrodes connected to dimmerstat. The 99.999% pure tin (in the form of wire) purchased from Koch-Light Ltd. Haverhill Sulfolk, England was placed in the evaporator. An appropriate amount of current was passed through the tungsten spiral to evaporate tin. Evaporated tin gets deposited on to the substrates [12]. The films formed were then placed in the muffle furnace at 200°C for 24 hours for allowing maximum oxidation of the samples. The samples were then annealed at three different temperatures 300°C, 400°C and 500°C each for 2 hours to get the desired SnO_2 thin films. The thin film samples were examined by XRD (Model-D8 Advance, Make-Bruker AXS GmbH, Berlin, Germany) using CuK_α radiation having wavelength 1.5402 Å. Surface morphology of the samples was analyzed by using FESEM (Model-S4800 Type II, Make-Hitachi HiTechnologies Corporation, Tokyo, Japan) and Elemental composition of the films by energy dispersive spectrophotometer (Model-XFLASH5030 Detector, Make - Bruker Nano GmbH, Berlin, Germany). The film resistance was measured in air and in the target gas atmosphere over the same range of operating temperature (i.e. from 50°C to 375°C).

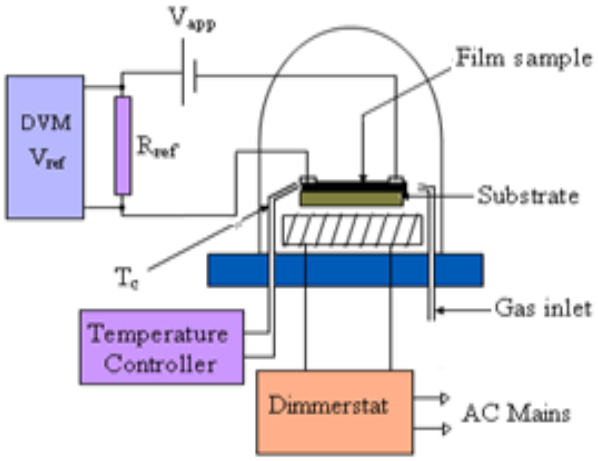

Figure1 Schematics of the Gas sensor unit.

The schematics of experimental arrangement used for this purpose is shown in Fig.1. Gas response was studied for various gases such as acetone, chlorine, CO_2, ethanol, H_2S and NH_3 having different concentrations ranging from 1cc to 15 cc in 15 *l* bell-jar, corresponding to 66.67 to 1000 ppm.

Results and Discussion

Structural, surface morphological and electrical studies were carried out; results were interpreted and systematically tabulated in our previous papers [12, 13]. To avoid repetition, details of only gas sensing studies are given over here.

The gas response studies was carried out by exposing the annealed SnO_2 thin film samples surface to various gases such as acetone, Chlorine, CO_2, ethanol, H_2S and NH_3. For each gas exposure, the change in the resistivity was observed at different operating temperatures i.e., from 50°C to 375°C. Good gas response was obtained for the samples annealed at 400°C, which were further considered for further measurements with these gases at different concentrations.

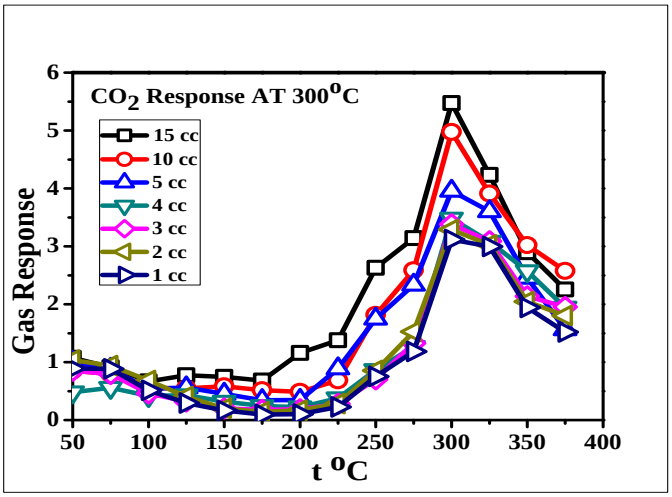

Figure2 (a) Gas response of SnO_2 film samples annealed at 300°C (CO_2 for example).

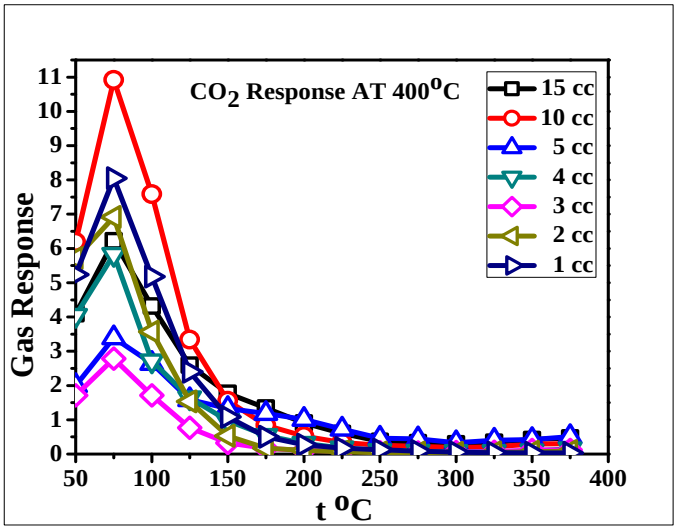

Figure2 (b) Gas response of SnO_2 film samples annealed at 400°C (CO_2 for example).

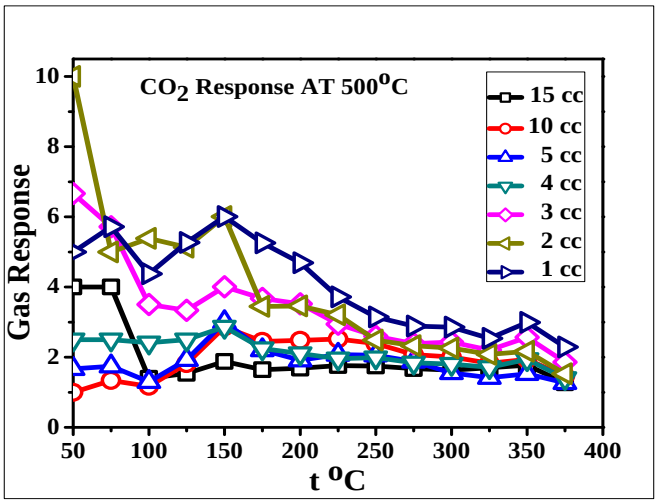

Figure2 (c) Gas response of SnO_2 film samples annealed at 500°C (CO_2 for example).

Fig.2 (a-c) explains relatively smooth gas response curves (e.g for CO_2) of samples annealed at 400°C compared to those annealed at 300 and 500°C.

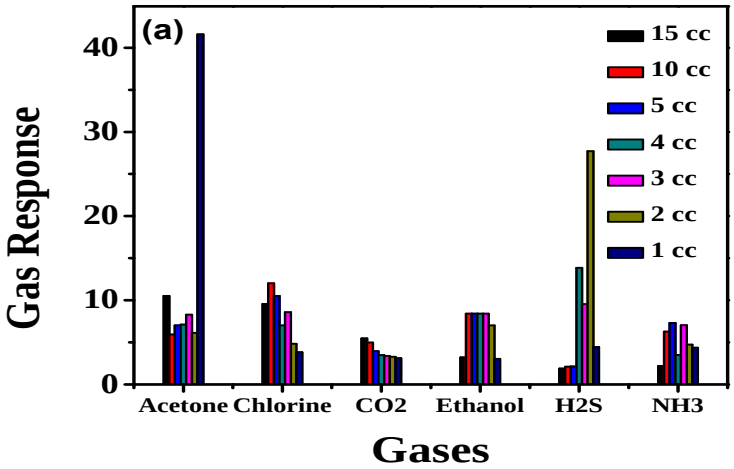

Figure3 (a) Histogram showing selectivity profile of the SnO_2 thin film samples annealed at3 00°C temperature for various gases and their concentrations.

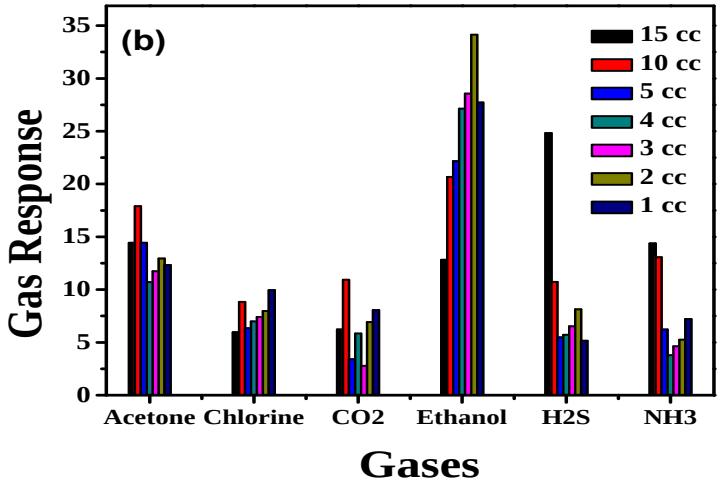

Figure3 (b) Histogram showing selectivity profile of the SnO_2 thin film samples annealed at 400°C temperature for various gases and their concentrations.

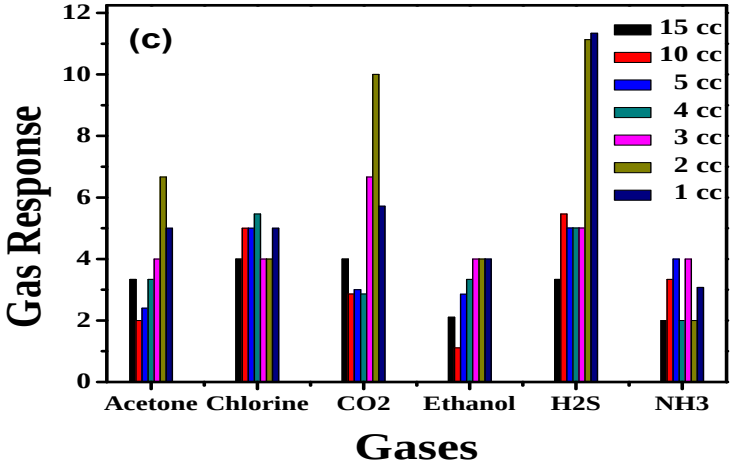

Figure3 (a) Histogram showing selectivity profile of the SnO_2 thin film samples annealed at 500°C temperature for various gases and their concentrations.

Fig.3 (a-c) exhibits the histogram of the annealed SnO_2 samples at 300°C, 400°C and 500°C for different gases and different concentrations, respectively. This indicates that annealing SnO_2 has a strong effect on the gas response properties, which indirectly depends on the oxygen contents, stoichiometry and the morphological evolution of the sample with annealing.

Conclusions

The thin film samples of tin oxide were prepared using the Physical Vapour Deposition technique. The XRD analysis of the samples proved that the film samples were polycrystalline. It is also confirmed from various structural parameters such as crystallite size, degree of crystallinity, texture coefficient etc. obtained for SnO_2 thin films annealed at 400°C. A well compact and irregular shaped grain was also seen for samples annealed at 400°C. Moreover, samples annealed at 400°C indicated rich oxygen contents, thereby increasing the resistivity of the sample. SnO_2 annealed at 400°C showed good gas response towards 2 cc ethanol and moreover good sensor response to all other gases, too. Hence, one can obtain the necessary gas response of a particular gas using a single material and by just manipulating its physical properties with controlled annealing.

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