Studies on gas sensing performance of tin oxide thick film

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Abstract: Thick films of SnO_2 were prepared using screen printing technique. These films have a potential application in fabrication of gas sensors. These films were dried at 60-80°C and fired at 550°C for 30 min. I-V characteristics of thick films were measured in air. The SnO_2 thick films exhibit semiconducting behavior. The gas sensing performance of thick films were tested on home-built static gas system for various gases such as: H_2S, CO, CO_2, H_2, NH_3, Ethanol, LPG, O_2 and Cl_2 etc. with operating temperature ranging from 50°C to 450°C. The SnO_2 thick film resistor showed the highest sensitivity to H_2S gas at 250°C. The film was structurally and morphologically characterized by using X-ray diffraction and SEM techniques respectively. The parameters such as sensitivity, selectivity, response and recovery time were studied and discussed.

Keywords: Screen printing technique; SnO_2 thick film; gas sensing system; sensitivity.

I. INTRODUCTION

The recent concern over environmental pollution and increased awareness of the need to monitor hazardous gases has stimulated substantial interest in sensors for the gases CO, CO_2, NO_x, H_2S, etc [1-4]. The requirement to detect and monitor these gases has led to the development of a wide variety of devices for solid state gas sensors.

H_2S monitoring is increasing in importance in industrial areas. Small amounts of H_2S can also have drastic effects. Detection of the volatile sulfide H_2S with SnO_2 sensors has so far been attempted by different preparation techniques.

The exact fundamental gas sensing mechanism of SnO_2 is not fully understood. However the principal of operation of SnO_2 based sensor lies on detecting the conductivity changes experienced by an n-type material when surface chemisorbed oxygen reacts with reducing gases, such as H_2S, CO, CO_2 or H_2. In the simpler detection mechanism, in clean dry air the conductivity of SnO2 is low because the conduction electrons are bound to surface oxygen, whereas in the presence of reducing gas, electrons are no longer bound to the surface states and the conductivity increases. Therefore the adsorption of gaseous species controls the surface resistance of SnO_2 as well as the grain boundaries. As gas adsorption is related to the surface of a material, it is preferred that the sensing material is in the polycrystalline and porous form.

We have tried to improve the H_2S sensitivity by screen printing technique and hence, further studies on gas sensing performance of SnO_2 thick film is investigated.

II. EXPERIMENTAL

The SnO_2 fine powder was calcined at 1000°C for 30 min. The thixotropic paste was formulated by mixing the resulting SnO_2 fine powder with a solution of ethyl cellulose (a ternary binder) in a mixture of inorganic solvents such as butyl cellulose, butyl carbitol acetate and terpineol. The paste was screen printed on a glass substrate in a desired patterns. The films prepared were fired at 550^0C for 30 min.

III. CHARACTERIZATION

X-ray diffraction pattern was recorded on diffractometer (Miniflex Model, Rigaku, Japan) using CuK_α radiation with a wavelength λ =1.5418 A° at 2θ values between 20° and 80°. The average crystallite size (D) was estimated using the Scherrer equation [5] as follows:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

where λ, β and θ are the X-ray wavelength, the full width at half maximum (FWHM) of the diffraction peak, and Braggs diffraction angle, respectively.

A JEOL 2300 model (Japan) was used to examine the surface morphology of the sample by scanning electron microscopy (SEM) and the percentage of constituent elements was evaluated by the energy dispersive X-rays analysis (EDAX) technique. The thickness of the thick films was measured by using the Taylor-Hobson (Talystep, UK) system. The optical absorbances of the films were measured using UV-visible-2450 spectrophotometer (Shimadzu) in the wavelength range 200-700 nm at room temperature. The static gas sensing system had been employed for testing of the films to gases, which is explained elsewhere [6]. The sensitivity (S) is defined as,

$$S = \frac{(Ra - Rg)}{Ra} = \frac{\Delta R}{Ra}$$

where R_g is the resistance in presence of test gas and R_a is the film resistance in dry air, measured at respective temperatures.

IV. RESULTS AND DISCUSSION

4.1 Structural analysis by XRD

A typical X-ray diffraction pattern recorded for SnO_2 thick film shown in fig 1. It is evident that SnO_2 thick film exhibits sharp peaks are observed for the polycrystalline thick film and crystalline structure of the film corresponds to the cassiterite tetragonal phases of the SnO_2. The diffraction peaks and their positions are associated with the reflections reported in JCPDS data file no. 77-0452. The XRD spectra of SnO_2 are characteristics of textured polycrystalline films with predominant reflections from the crystallographic (110), (200) and (101) planes parallel to the substrate [7]. The (110) surface of SnO_2 is energetically the most stable and the predominant crystal face found in polycrystalline samples. Hence, the sensing mechanism of SnO_2 gas sensors have focused on the SnO_2 (110) surface [8]. The crystallite size was esteemed to be 46 nm.

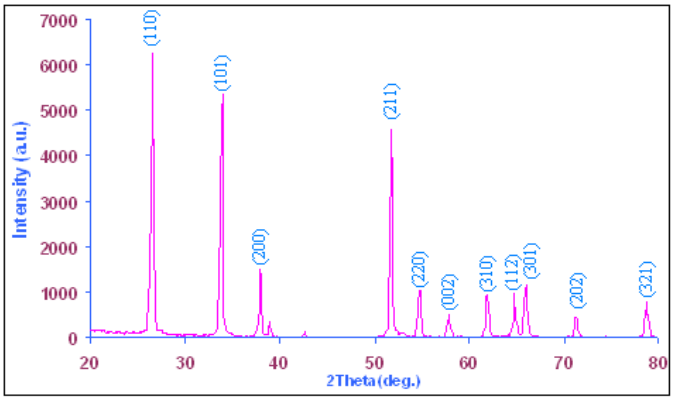

Fig. 1: XRD pattern of polycrystalline SnO_2 thick film.

4.2 Surface morphological analysis by SEM

Figure 2 shows SEM photograph of SnO_2 tick film sample, it is clear that the particle sizes are in the range of 0.1 µm. A random distribution and large agglomeration of particles are observed for the sample. This may be due to the smallest particle size as it offers higher surface area and enhances the sensitivity very effectively. The surface area plays an important role. Higher surface area leads to the higher concentration of chemisorbed air species which further increases the change in the conductance during the adsorption of reducing gases.

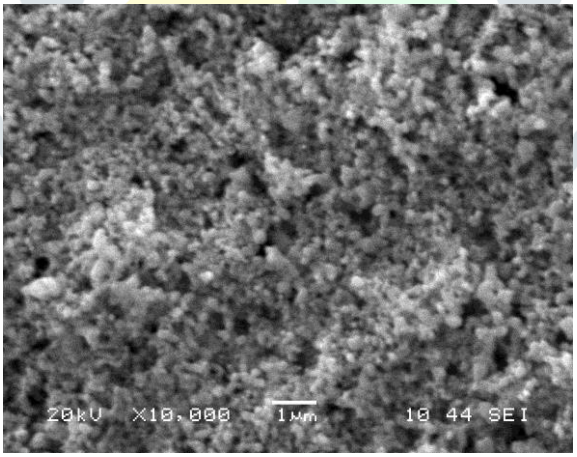

Fig. 2: Scanning electron micrograph of SnO_2 tick film.

4.3 Gas sensing performance of SnO_2 thick film

Figure 3 shows the response sensitivity of SnO_2 thick-film sensor sample toward H_2S gas at different temperatures. It can be seen that both of the two different samples showed maximum response at about 150 ^oC. The sensitivity curve of the SnO_2 thick film sample toward 8 ppm of H_2S in air when operated at 250 ^oC showed higher response.

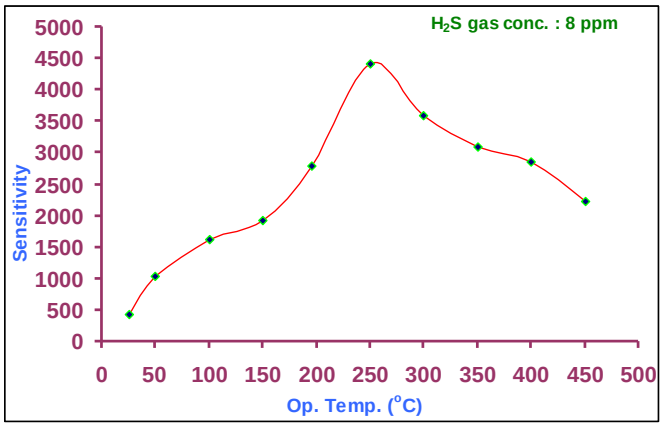

Fig. 3: Sensitivity of SnO_2 thick film towards H_2S gas

Selectivity or specificity is defined as the ability of a sensor respond to a certain (target) gas in the presence of other gases [9]. Fig. 4 the shows a histogram of SnO_2 thick film sensor selectivity towards 8 ppm of H_2S and 80 ppm of other test gases such as H_2, CO_2, CO, liquid petroleum gas, Cl_2, NH_3 and ethanol vapors at 250 °C. The sensor shows the high selectivity for H_2S and could distinguish a small amount of H_2S among other gases.

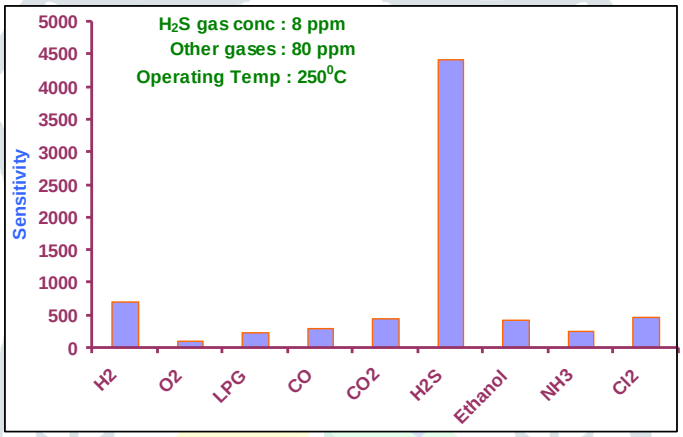

Fig. 4: Selectivity of SnO_2 thick films.

Figure 5 shows the sensitivity of SnO_2 thick film sensor at different gas concentration The sensitivity values observed increasing continuously with increasing the gas conc to 8 ppm of H_2S .Furthermore, we observed saturation of the sensor after exposing them to more than 8 ppm.

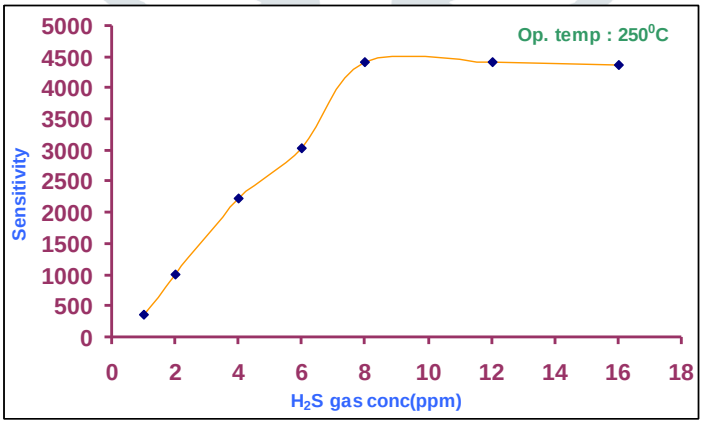

Fig. 5: Sensitivity at different gas concentration.

V. RESULTS AND DISCUSSION

The SnO_2 thick films were prepared by screen printing technique. The resulting SnO_2 thick film material was confirmed by XRD measurement and SEM shows the porous film. Particularly noteworthy results were obtained with H_2S, for which the most sensitive SnO_2 thick film -reached a maximum sensitivity for the exposure to 8 ppm of H_2S. The results of the H_2S sensing studies reveal that the SnO_2 film is a suitable material for the fabrication of the H_2S sensor.

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