Conference Paper

Effect of MgO and V$_2$O$_5$ Catalyst on the Sensing Behaviour of Tin Oxide Thin Film for SO$_2$ Gas

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The present work shows the SO$_2$ gas sensing property of SnO$_2$ thin film based sensor prepared by using RF sputtering technique. Different catalysts (MgO and V$_2$O$_5$) in form of nanoclusters having diameter of 600 $\mu$m have been loaded on SnO$_2$ surface to detect SO$_2$ gas. The sensing response of all these films towards SO$_2$ is monitored. Microstructural studies have been carried out using XRD and UV-Visible Spectrophotometer and a good correlation has been found between the microstructural and gas sensing properties of these deposited samples. Both catalysts when incorporated with SnO$_2$ film show high selectivity towards SO$_2$ gas at lower operating temperature. MgO gives a sensitivity of 317% at an operating temperature of 280°C towards 500 ppm of SO$_2$ gas whereas V$_2$O$_5$ catalyst gives a sensitivity of 166% at 280°C for the same amount of gas.

1. Introduction

Recently, substantial interest has arisen in terms of protecting the environment from various air pollutants generated by combustion exhausts. SO$_2$ is one of the most hazardous atmospheric pollutants because it directly contributes to acid rain. Several studies have shown that repeated exposure to low levels of SO$_2$ (<5 ppm) can cause permanent pulmonary impairment [1]. The long-term and short-term exposure limits for SO$_2$ gas are 2 ppm and 5 ppm, respectively. Therefore, the development of an efficient SO$_2$ gas sensor for environmental monitoring has become a necessary task.

There are various techniques for determining the SO$_2$ gas concentration in the atmosphere such as ion chromatography [2], Fourier transform infrared spectrometry [3], optical fiber sensors [4], SAW gas sensors [5], conductometry [6], and electrochemical methods [7]. Amongst all the techniques, conductometric sensors are fast, highly sensitive, reproducible, of low cost, and convenient.

Semiconducting tin oxide (SnO$_2$) based conductometric gas sensors have received much attention for more than four decades due to their suitable physical-chemical properties and possibility to detect wide variety of gases with high response [8–11]. SnO$_2$ is naturally nonstoichiometric having a rutile phase that eases the adsorption of oxygen on its surface and thus it is highly sensitive towards many toxic and harmful gases [10, 11]. The main problem with the pure SnO$_2$ thin film based gas sensors is their poor selectivity and high operating temperatures that can be improved to a great extent by using specific catalysts [12, 13].

The aim of this study is to develop a SnO$_2$ thin film based SO$_2$ gas sensor with MgO and V$_2$O$_5$ catalysts having efficient response characteristics.

2. Experimental

SnO$_2$ thin films of 90 nm thickness were deposited using RF diode sputtering technique using a metal Sn target (99.999% pure) in a reactive ambient of Ar and O$_2$ gas mixture as optimized in our previous work [14]. The sensing response characteristics of SnO$_2$ thin films were studied using Interdigital Electrodes (IDEs) of platinum (Pt) as shown in Figure 1.
The sensor fabrication details are given in our previous report [14]. For the enhanced and selective gas sensing response characteristics, nanoclusters of MgO and V$_2$O$_5$ of 10 nm thickness and 600 $\mu$m diameter were dispersed uniformly over the surface of SnO$_2$ thin film by pulsed laser deposition technique (PLD) using a fourth harmonic ($\lambda = 266$ nm) of Nd:YAG laser with fluence $= 1.5$ J/cm$^2$ using a shadow mask. Ceramic targets of MgO and V$_2$O$_5$ of 1” diameter were used for deposition using PLD. The as-grown sensing elements (MgO/SnO$_2$ and V$_2$O$_5$/SnO$_2$) were annealed at 300 $^\circ$C in air for 3 h to stabilize the sensor.

Thickness and surface roughness of deposited thin films were measured using a (Veeco Dektak 150) surface profiler. Crystalline structure of SnO$_2$ thin films was studied using Bragg-Brentano ($\theta-2\theta$) scan of an X-ray diffractometer (Bruker D8 Discover) using the CuK$_\alpha_1$ source ($\lambda = 0.154$ nm). A double beam UV-Visible Spectrophotometer (Perkin Elmer, Lambda 35) was used to study the optical properties of SnO$_2$ thin films.

A specially designed gas sensor test rig (GSTR) has been used to study the gas sensing response characteristics of the sensing layer for SO$_2$ gas. The sensor was placed on a temperature controlled heating block inside the glass test chamber to measure the sensor response as a function of temperature (100 to 300 $^\circ$C). At each temperature, the sensor was first stabilized in air to obtain a stable resistance value. Target gas (SO$_2$) of specific concentration (500 ppm) was introduced into the test chamber and changes in the sensor resistance were recorded after every second using a data acquisition system consisting of a digital multimeter (model: Keithley 2700) interfaced with a computer. SO$_2$ is a reducing gas and the sensor response is defined as

$$ S = \frac{R_a - R_g}{R_g}, $$

where $R_a$ and $R_g$ are the resistance of the sensor element in the presence of atmospheric air and resistance of target SO$_2$ gas, respectively.

### 3. Results and Discussions

Figure 2 shows the X-ray diffraction (XRD) pattern of the SnO$_2$ sensor structure. The diffraction peaks observed at 2$\theta = 26.6^\circ$, 33.8$^\circ$, and 51.8$^\circ$ can be assigned to (110), (101), and (211) planes of the rutile structure of SnO$_2$, respectively [15]. The observed XRD peaks are found to be broad indicating that the particles are of small size. No peaks corresponding to any secondary phase due to MgO and V$_2$O$_5$ have been observed in the XRD spectra which may be attributed to the small thickness of the nanoclusters of the catalysts. The crystallite size was estimated by fitting the width of dominant (101) diffraction peak using Scherrer’s formula and is found to be $\sim$8 nm.

Figure 3 shows the transmittance spectra of the SnO$_2$ thin film. The films exhibit a high transmission ($>$80%) in the visible region and show a sharp fundamental absorption edge at around 340 nm. The value of optical band gap was estimated from the Tauc plot and is found to be 3.9 eV which matches very well the reported value [16].

Figure 4 shows the sensing response of bare SnO$_2$ film and SnO$_2$ film having MgO and V$_2$O$_5$ as catalysts. Bare SnO$_2$ thin film based sensor showed no response whereas sensors having MgO and V$_2$O$_5$ catalysts are showing a response of 3.17 and 1.66, respectively. Table 1 shows the sensor response (%), response time, and recovery time of the prepared samples at their operating temperature (280 $^\circ$C). It can be seen from Table 1 that the sensor response (%) is low for SnO$_2$ thin film and gets improved with V$_2$O$_5$ as a catalyst and the maximum value of response (%) is found for the SnO$_2$ with MgO thin film as catalyst with a response time and recovery time of 59 s and 52 s, respectively. MgO and V$_2$O$_5$ doped SnO$_2$ sensors for sensing SO$_2$ gas have already been reported by Lee et al. [13]. However, the sensing response is reported to be poor (44%) at a high operating temperature (400 $^\circ$C) towards 1 ppm of SO$_2$ gas. Since gas sensing is a surface phenomenon, the incorporation of catalyst/modifier
### Table 1: Sensing response characteristics of the prepared sensors at an operating temperature of 280°C.

<table>
<thead>
<tr>
<th>Serial number</th>
<th>Sample</th>
<th>Response (%)</th>
<th>Response time (s)</th>
<th>Recovery time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SnO₂</td>
<td>107</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>SnO₂ + V₂O₅</td>
<td>166</td>
<td>90</td>
<td>43</td>
</tr>
<tr>
<td>3</td>
<td>SnO₂ + MgO</td>
<td>317</td>
<td>59</td>
<td>52</td>
</tr>
</tbody>
</table>

Figure 3: Transmittance spectra of SnO₂ film deposited on glass substrate. Inset: Tauc plot for the SnO₂ thin film.

Figure 4: Sensing response of SnO₂ thin film, SnO₂ thin film having V₂O₅ catalyst, and SnO₂ thin film having MgO catalyst as a function of temperature.

in the form of nanoclusters onto the surface of SnO₂ thin film is proved to give enhanced sensing response of 317% and 166% at 280°C towards 500 ppm SO₂ gas.

### 4. Conclusion

SnO₂ thin films of desired morphology and higher electrical resistance have been deposited by RF sputtering for efficient detection of SO₂ gas. SnO₂ film with MgO catalyst exhibits enhanced sensor response of 317% with moderate response time of 59 s and recovery time of 52 s at an operating temperature of 280°C. The sensor response and recovery time are governed by the adsorption and desorption of SO₂ gas molecules at the sensor surface. The formation of SnO₂ thin film with desired surface morphology having moderate porosity and appropriate catalysts results in an enhanced sensing response towards SO₂ gas (500 ppm) at a low operating temperature of 280°C.

### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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