Nanocomposites thin films as gas sensor application

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Abstract

In present work nanocomposites thin films were prepared by spray pyrolysis technique. As prepared thin films were studied using XRD and FE-SEM and. The film sprayed for composition of WO$_3$-V$_2$O$_5$ (Sample =S2) was observed to be most sensitive (S = 1130) to SO$_2$ at 350 °C. The sensor shows quick response (4 s) and fast recovery (8 s) time. The results are discussed and interpreted.

Keywords: Spray Pyrolysis, WO$_3$-V$_2$O$_5$ nanocomposites, SO$_2$ gas sensing, quick response, fast recovery

1. Introduction

Metal oxide semiconductors are used extensively as a sensing element of different gases and vapors. A depletion region always formed at the surface of metal oxide semiconductor due to adsorption of air oxygen molecules. Then the reaction with the target gas molecule causes reduction of depletion region which results change in conductivity of metal oxide semiconductor. The conductivity may increase or decrease depending on type of semiconductor and type of target gas [1]. The metal oxide-sensing layer (WO$_3$ or V$_2$O$_5$) has been fabricated in different physical forms such as thin film, thick films, and bulk pellets. However, the thin film form is expected to be most effective, because sensing is basically a surface phenomenon of film. Thus, a very few work has been reported for the combination of WO$_3$/V$_2$O$_5$ oxide composite.

In present work efforts was done in the area of SO$_2$ detection using metal oxide thin films. However, not much attention has been given to the fabrication of nanocomposites structure for detection of SO$_2$ gas. There has been intensive research on improving the gas sensitivity and selectivity by controlling the particle size, nanostructures, sensing temperature, surface and structure [2].
2. Experimental details

2.1 Preparation of WO$_3$-V$_2$O$_5$ nanocomposites thin films

The starting material used for the preparation of WO$_3$-V$_2$O$_5$ nanocomposites thin films were tungsten hexachloride (WCl$_6$, Purified Merck) and vanadium (III) chloride (VCl$_3$, Purified Aldrich). Tungsten hexachloride and vanadium (III) chloride were mixed at various volume ratio such as 30:70, 50:50 and 70:30 as indicated in Table 1.

Table 1: Amounts of spraying solutions and reactant

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>WCl$_6$ (cm$^3$)</th>
<th>VCl$_3$ (cm$^3$)</th>
<th>Reactants</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>30</td>
<td>70</td>
<td>WO$_3$:V$_2$O$_5$</td>
</tr>
<tr>
<td>S2</td>
<td>50</td>
<td>50</td>
<td>WO$_3$:V$_2$O$_5$</td>
</tr>
<tr>
<td>S3</td>
<td>70</td>
<td>30</td>
<td>WO$_3$:V$_2$O$_5$</td>
</tr>
</tbody>
</table>

The optimized deposition parameters like substrate temperature (350 °C), spray time (10 mn.), rate of spraying solution (8 ml/min.), nozzle to substrate distance (30 cm), quantity of the solution sprayed (30 ml), pressure of carrier gas, and to and fro movement of the nozzle were kept constant. The temperature of the substrate is maintained at a constant value by using a temperature controlled hot plate. The film formation depends upon the droplet landing, reaction and solvent evaporation, which relates to the droplet size. When the droplet approaches the substrate just before the solvent is completely removed, that is the ideal condition for the preparation of the pure WO$_3$, V$_2$O$_5$, and WO$_3$-V$_2$O$_5$ nanocomposites thin film. The as prepared WO$_3$, V$_2$O$_5$, and WO$_3$-V$_2$O$_5$ nanocomposites thin films samples were annealed at 500 °C for 1 h.

3. Results and discussion

3.1. Structural analysis using X-ray diffractogram

Fig. 1 shows the X-ray diffractogram of thin film sample. The observed peak predominates indicating a preferential growth. This means that the grains have $c$-axis perpendicular to the substrate surface. The calculated average crystallite size was found to be 18 nm.

Fig. 1. X-ray diffractogram of most sensitive nanocomposites thin film (sample S2)
3.2. Field emission scanning electron microscope

![FE-SEM image](image)

*Fig. 2. FE-SEM images of most sensitive nanocomposites thin film (sample S2)*

FE-SEM images of pure WO$_3$, V$_2$O$_5$, and WO$_3$-V$_2$O$_5$ nanocomposites were represented in Fig. 2. Grain size observed to be in the range of 21 - 44 nm.

4. Gas sensing performance of the sensors

4.1. Gas response

![Graph](image)

*Fig. 3. Gas response of nanocomposites thin films with operating temperature*

Fig. 3 represents the response characteristics of the WO$_3$-V$_2$O$_5$ nanocomposite thin films as a function of operating temperature. Among all the films, the sample (S2) film shows the maximum response (1130) at 350 °C to 500 ppm of SO$_2$.

4.2. Response and recovery of the sensor with concentration of gas in ppm

![Graph](image)

*Fig. 4 Response and recovery of the sensor for most sensitive sample (S2).*
The response is quick (4 s) and recovery is fast (8 s). The high oxidizing ability of adsorbed oxygen species on the surface nanoparticles and high volatility of desorbed by-products explain the quick response to H$_2$S and fast recovery [3-5].

5. Conclusion

Nanocomposites thin films were prepared by simple spray pyrolysis technique. The WO$_3$-V$_2$O$_5$ thin film of (Sample S2) was most sensitive to SO$_2$ gas and exhibits the response of S = 1130 to the gas concentration as 500 ppm at the temperature of 350 °C. The WO$_3$-V$_2$O$_5$ nanocomposites thin films exhibit rapid response–recovery which is one of the main features of this sensor.

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Reference