

CuO- SnO₂ nanocomposites thin films for the fast detection of H₂S gas

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Abstract

CuO-SnO₂ nanocomposites thin films were prepared by spray pyrolysis method onto the heated glass substrate at 250 °C. The films were fired at 500 °C. As prepared thin films were studied using XRD and SEM. The gas sensing performance of different composition of CuO-SnO₂ was studied on exposure of different gases for 10 ppm. sensor shows quick response (3 s) and fast recovery (6 s) time. The results are discussed and interpreted.

Keywords: Spray Pyrolysis, CuO-SnO₂ nanocomposites, H₂S gas sensing, quick response, fast recovery

1. Introduction

The semiconductor-based chemical sensors own their popularity to their small size, simple operation, high sensitivity, selectivity, and relatively simple associated electronics. The metal oxide-sensing layer (SnO₂ or CuO) 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 [1].

Monitoring of H₂S is crucial in laboratories and industrial areas. Hence a sensor with fast detection and high gas response is desired. It was demonstrated that the cations with a low electro- negativity value are best suited for SnO₂ doping with regard to hydrogen sulfide detection. However, it was found by Yamazoe and co-workers, that doping of tin dioxide with copper that is intermediate in electro negativity, gives rise to the outstanding sensitivity [2].

This paper deals with the preparation of composite thin films of SnO₂ and CuO. These films were studied using different analytical techniques. These CuO-SnO₂ nanocomposites thin films were tested for sensing different gases and was observed to be most sensitive to H₂S at 250 °C.

2. Experimental details

2.1. Preparation of pure CuO - SnO₂ nanocomposites thin films

The starting material used for the preparation of CuO-SnO₂ nanocomposites thin films were copper chloride dehydrate (CuCl₂.2H₂O Purified Loba Chemie) and tin chloride dehydrate (SnCl₂.2H₂O Purified Merk). Copper chloride dehydrate and tin chloride dehydrate were mixed at various volume ratio such as 30:70, 50:50 and 70:30 as indicated in Table 1. To stabilize the starting solution, a few droplets of hydrochloric acid (HCl) were added. The temperature of the substrate is maintained at a constant value by using a temperature controlled hot plate. The deposition parameters like substrate temperature (250 °C), rate of spraying solution (7 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 as prepared CuO- SnO₂ nanocomposites thin films samples (S1, S2, and S3) were annealed at 500 °C for 1 h.

Table 1 Amounts of spraying solutions and reactant

Sample No.	CuCl ₂ .2H ₂ O (cm ³)	SnCl ₂ .2H ₂ O (cm ³)	Reactants
S1	30	70	CuO:SnO ₂
S2	50	50	CuO:SnO ₂
S3	70	30	CuO:SnO ₂

3. Results and discussion

3.1. Structural analysis

Fig. 1 shows the X-ray diffractogram of CuO-SnO₂ nanocomposites thin film. The structural properties of the films were investigated using XRD. The 2θ values were varied from 20 to 80. The calculated average crystallite size was found to be 14 nm respectively.

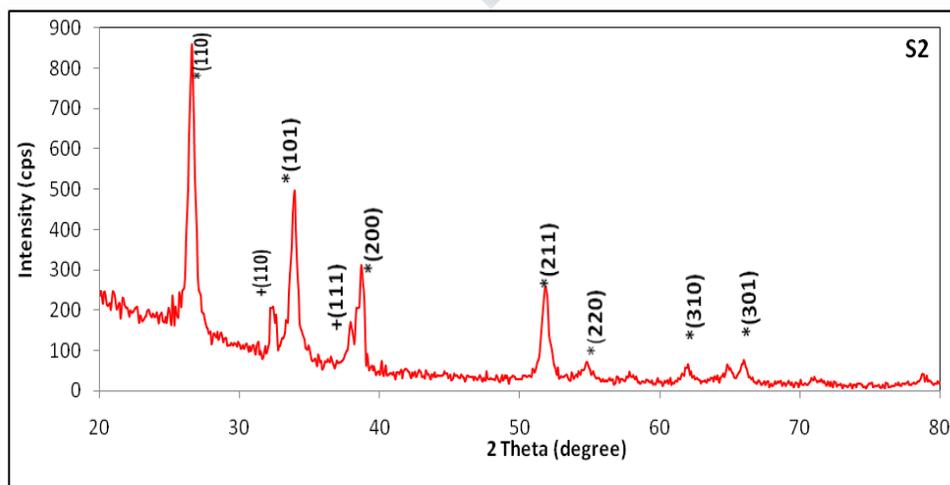


Fig. 1. X-ray diffractogram of CuO-SnO₂ nanocomposites thin film (most sensitive sample (S2))

3.2. Surface Morphology

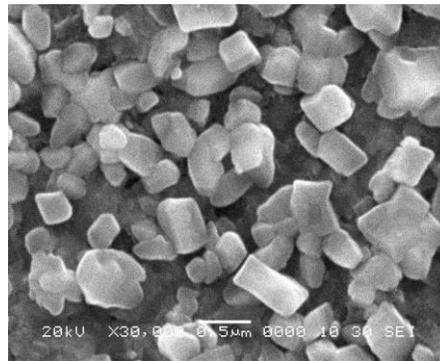


Fig.2. SEM image CuO-SnO₂ nanocomposites thin film (most sensitive sample (S2))

SEM imaged of CuO-SnO₂ nanocomposites was represented in Fig.2. Grain size observed to be in the range of 23 - 33 nm.

3.3. Elemental composition using (EDAX)

Stoichiometrically expected at % of Sn, Cu and O is : 20, 20 and 60, Observed at % pure CuO - SnO₂ nanocomposites thin films were given in Table 2. It is clear from table 2, that as prepared CuO - SnO₂ nanocomposites thin films were observed to be nonstoichiometric in nature.

Table 2 Quantitative elemental analysis as prepared CuO-SnO₂ nanocomposites thin film

Element	Observed					
	S1		S2		S3	
	mass %	at %	mass %	at %	at %	mass %
O	27.19	72.36	67.96	27.19	72.36	67.96
Cu	4.85	3.26	19.61	4.85	3.26	19.61
Sn	67.96	24.38	60.03	67.96	24.38	60.03
Total	100.00	100.00	100.00	100.00	100.00	100.00

4 Gas sensing performance of the sensors

4.1. Gas response:

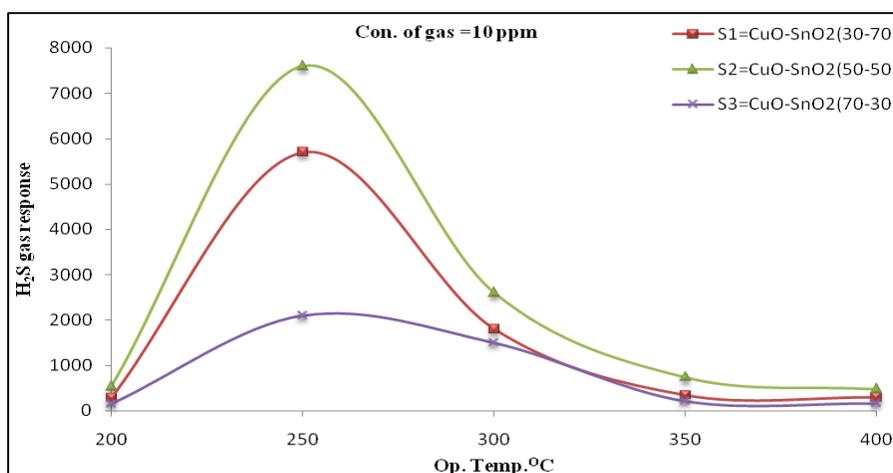


Fig. 3 represents the response characteristics of the CuO-SnO₂ nanocomposite thin films as a function of operating temperature. Among all the films, the sample (S2) film shows the maximum response (7599) at 250 °C to 10 ppm of H₂S.

4.2. Response and recovery of the sensor

The response and recovery of the CuO-SnO₂ nanocomposites thin film (S2) sensor on exposure of 10 ppm of H₂S at 250 °C are represented in Fig. 4. The response is quick (3 s) and recovery is fast (6 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 H₂S and fast recovery.

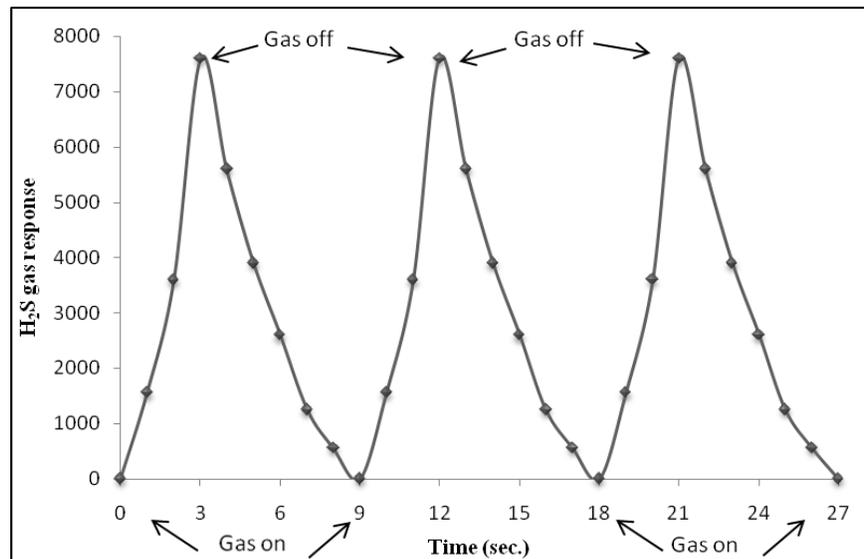


Fig. 4. Response and recovery of the sensor for most sensitive sample (S2).

Conclusion:

CuO-SnO₂ nanocomposites thin films were prepared by simple spray pyrolysis technique. The structural and microstructural properties confirm that the as-prepared CuO-SnO₂ nanocomposites thin films are nanostructured in nature. The CuO-SnO₂ thin film of (Sample S4) was most sensitive to H₂S gas and exhibit the response of $S = 7499$ to the gas concentration as 100 ppm at the temperature of 250 °C. The CuO-SnO₂ nanocomposites thin films exhibit rapid response–recovery which is one of the main features of this sensor.

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