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H₂S Gas Sensor at Room Temperature For SnO₂-CuO Thick Film

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Abstract: The SnO₂ and CuO powder mixed with different ratio and heated at 800°C and then this powder is used to prepared thick films by a screen – printing technique on glass substrate. The H₂S gas sensing properties, particularly the rate of response of CuO-SnO₂ sensors are influenced by the CuO loadings at room temperature. XRD analysis showed that crystallite size is small (97.3nm) for 50SnO₂-50CuO composition. The Sensitivity increases drastically as the expose of H₂S gas for 50SnO₂-50CuO sample. This sample is found to be better sensing material as regards to other.

Keywords: Hydrogen sulphide; SnO₂-CuO; Sensors; X-Ray Diffraction

I. INTRODUCTION

The detection of H₂S, a bad-smelling and toxic gas, is important in environmental monitoring. Therefore, much attention has been paid to the search for H₂S gas sensitive materials. The sensing properties of various semiconductor oxides, especially the SnO₂ - based materials, have been extensively studied. The p-n junction concept is used in the present investigation, instead of adsorption-desorption mechanism, which was introduced in 1979 and has been applied as humidity sensors, liquid sensors and gas sensors [1-4].

II. EXPERIMENTAL

A. Construction of sensor

SnO₂ and CuO is mixed in different ratio i.e SnO₂-CuO (SA1:100-00, SA2:80-20, SA3:70-30, SA4:60-40, SA5:50-50, SA6:40-60, SA7:30-70, SA8:20-80, SA9:00-100) thoroughly in an acetone medium by using a mortal and pestle and then heated at 800°C in a furnace. The paste used in Screen-printing was prepared by maintaining inorganic to organic material ratio at 70:30. The paste was screen printed on an glass substrate [5], of size was 75mm X 25 mm .The films were dried at 150 °C for about 20 min to remove the organic material. The film was aged for 4 weeks in open air [6] for drying. For the electrical characterization purpose form the electrode on two side of thick film by using silver paste.

B. Measurement of gas sensing characteristics

The gas sensing properties of these samples (thick film) were studied in a home –built static gas characterization system. The system consist of DC power supply, resistor (Rs), DC Millivoltmeter (Systronics type, Model No.412, ±1μV) and chamber(2 liters and size 12 cm diameter, 27 cm height, cylindrical shape) with inlet ,outlet and open/close window. Using silver paste between two sides of thick films forms the electrodes. The DC power supply (V) in series with resistor (Rs=1MΩ) is connected to sensor. The voltage drop (Vs) across the Rs is measured by the microvoltmeter. The required gas concentration inside the system is achieved by passing gas through flow meter with flow rate 200 ml/min in the airtight chamber at ambient condition. The samples were tested for a wide range of concentration of H₂S. The sensitivity is calculated by using the formula

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

Where R_a & R_g are the electrical resistance of the sensor in presence air and gas+air respectively.

The structure was of sensor material was examined by X-ray diffractometer (PANalytical PW: 3040/60 made in Netherland) using CuKα radiation (1.5418 Å) in the 2θ range of 5 -100°. The 2θ step and step acquisition time were 0.0170° and 7.7523 s respectively.

III. RESULTS

The thrust for the present work was to study the gas sensing characteristics of SnO₂: CuO thick films. The results obtained are analysed, discussed and presented in the following part of this section.

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A. Characterization of film by XRD

In Fig. 1, shows XRD spectra of pure SnO₂, pure CuO and composite of SnO₂ and CuO. The crystallite size for all the samples is calculated by using the Scherrer equation [7]

$$D = 0.9\lambda / \beta \cos\theta$$

where D is the crystallite size, k is the constant ($= 0.9$ assuming that the particles are spherical), λ is the wavelength of X-ray radiation, β is the line width (obtained after correction for the instrumental broadening) and θ is the angle of diffraction. The minimum crystallite size of the sample is found for 50SnO₂:50CuO system i.e. 97.37nm

1) *Effect of Crystallite size:* If the crystallite size is small so that the space-charge region extends through a large fraction of the grain or if the whole grain is included completely in this region, the gas sensor sensitivity could be high. From calculated value seen that the crystallite size is small for 50SnO₂-50CuO sample in a SnO₂-CuO system and hence it gives higher sensitivity than the other sample.

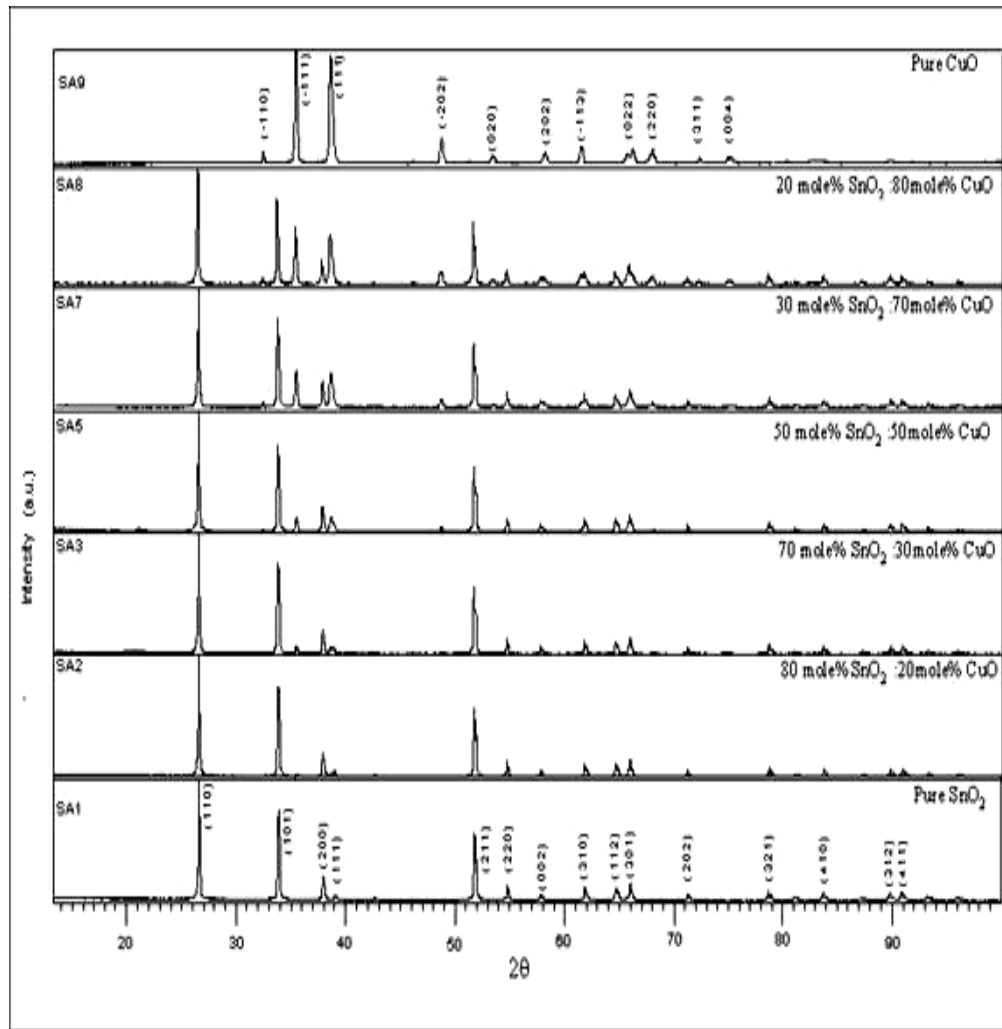


Figure 1-XRD patterns of SnO₂:CuO powder

B. Sensitivity Curve

Figs. 2 (a) and (b) shows the H₂S response of SnO₂ - CuO sensors. Fig. 2 (a) shows variation of sensitivity with H₂S gas concentration for different compositions of SnO₂-CuO at room temperature. It is seen that change of resistance in air and after exposed to the H₂S gas are very drastic and gives higher sensitivity. It is observed that the sensitivity is highest for 50 SnO₂ – 50 CuO sample and for all other sample the sensitivity is lower.

Fig. 2 (b) shows variation of sensitivity of sensor with composition of SnO₂ in mol % at different concentration of H₂S gas at room temperature. It is observed that the sensitivity is high for 50 SnO₂ – 50 CuO sample at different ppm of H₂S.

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Figure 2(a) Variation of sensitivity with H₂S gas concentration of different sample of SnO₂-CuO system

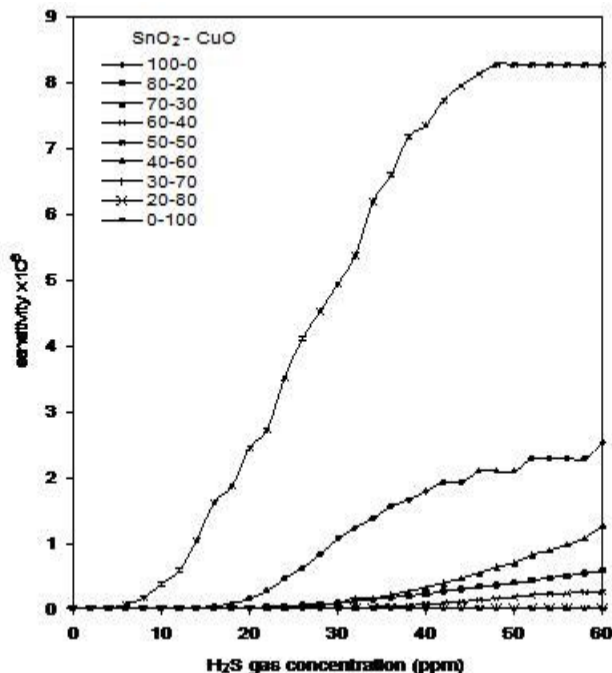


Figure 2(a) Variation of sensitivity with H₂S gas concentration of different sample of SnO₂-CuO system

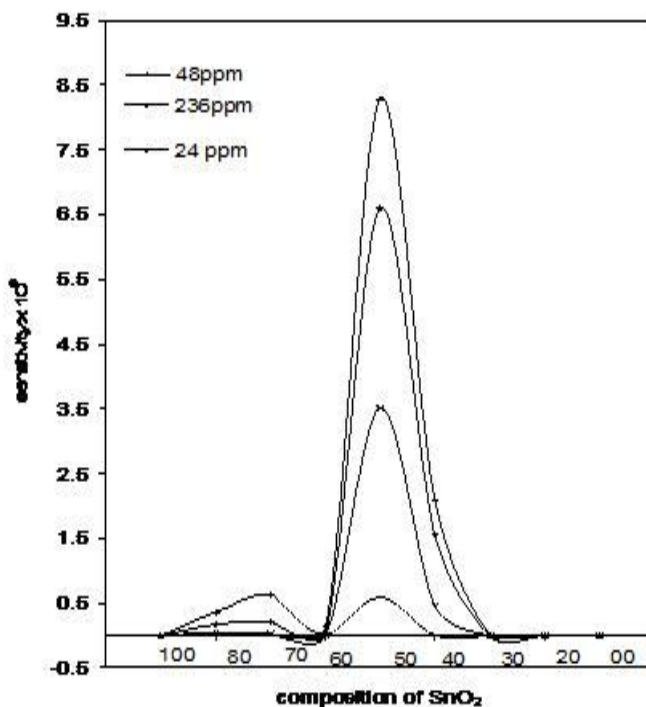


Figure 2 (b) Variation of sensitivity with composition of sample at different ppm of H₂S gas

C. Sensing Mechanism

The mechanism to explain the observed excellent behavior of increase in sensitivity of the CuO- SnO₂ film exposed to H₂S gas can be explained as follows. We consider that sensing

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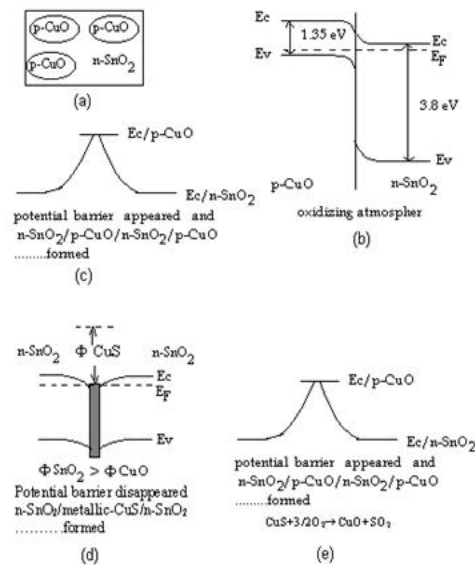
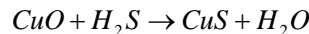


Figure 3: Schematic diagram of CuO-SnO₂ sensing mechanism

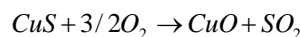
- (a) SnO₂ particles surrounded by CuO
- (b) band structure for CuO-SnO₂ contact
- (c) potential barrier diagram of n-SnO₂/p-CuO/n-SnO₂/p-CuO
- (d) potential barrier diagram of n-SnO₂/metallic-CuS/n-SnO₂
- (e) potential barrier appeared again

reaction of hydrogen sulfide (H₂S) occurs only on the surface of the film and diffusion of chemical species into the inner part of the film is negligible, and p- n junctions is formed only at the interface between CuO and SnO₂ while oxygen deficient SnO₂ shows n type conductivity by electrons, oxygen- excess CuO shows p- type conductivity by holes. Sintered gas sensors are composed of a mixture of CuO and SnO₂ and thus, CuO is dispensed here and there is a sensor. In this case, in an oxidizing atmosphere a thick charge depletion layer is formed near the grain surface of SnO₂ as a p- n junction as shown in fig. 3 (a). The electrons associated with these charged species are drawn from the conduction band of the bulk material, leading to an increase in resistance. As a result a potential barrier appears at the boundary forming a continuous chain of n- p- n- p junctions.

The schematic band diagram of the typical n-p-n-p is a SnO₂ – CuO – SnO₂ – CuO and the band structure in the vicinity of the CuO-SnO₂ interface can be drawn as shows in fig. 3 (b)&(c). However if the above CuO layer is exposed to H₂S or other sulfur compound gases, the CuO is converted to CuS[7], which is a good electronic conductor in the following equation



This CuS is known to be metallic in nature and thus the n- p- n heterostructure as well as charge depletion layer will be destroyed and transformed to a metal -n- type semiconductor configuration. A typical metal -n- semiconductor band picture can be drawn in fig 3 (d). Since the work function of CuS is lower than that of SnO₂ the band bending is as shown. This particular situation arises because at equilibrium there is a flow of electrons from the lower work function species i.e. CuS, to higher work function species, i.e., SnO₂. This results in the band bending downwards which facilitates the easy flow of electrons from CuS to SnO₂ and vice versa, since there is no barrier between them as shown in fig. 3 (d). This flow of electrons results in a decrease in electrical resistance. This configuration is similar to a metal- semiconductor contact for an ohmic junction where there is an unimpeded conduction of electrons in either direction. This theory explains quite convincingly the decrease in resistance on exposure of CuO- SnO₂ sensor element to reducing gases like H₂S and the increase in resistance back to the original value once this H₂S atmosphere ceases to exist. This is because CuS converts back to CuO when placed in air and return to its normal state, which is shown as follows.



So, the potential barrier appears again, as seen in fig 3 (e).

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IV. CONCLUSION

In response of H₂S gas for the SnO₂-CuO system, it is observed that sensitivity is higher i.e 82.83x10⁵ at 56 ppm of H₂S gas concentration for 50 SnO₂-50 CuO sample. For other compositions of samples sensitivity decreases. We have discussed sensing mechanism of H₂S on the surface of SnO₂-CuO, it reveals that the breaking of pn heterojunction and formation of CuS from CuO reduces the resistance of the sensor because of removal of the oxygen due to spill-over of hydrogen quickly interacts with the SnO₂ surface between the dispersed CuO islands, leaves behind excess free electrons available for conduction. Consequently, reduces the resistance of the sample to great extent.

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