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Comparative H_2S Sensing Characteristics of Fe_2O_3: Thin Film vs. Bulk

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ABSTRACT

Comparative investigations of gas sensing characteristics of Fe_2O_3 in both thin film as well as bulk forms have been performed. Thin film sensors were realized by first depositing Fe films using electron-beam evaporation followed by thermal oxidation. Bulk sensors in the form of pellets were prepared by cold pressing commercial Fe_2O_3 powder with subsequent sintering. Both thin film and bulk Fe_2O_3 sensors exhibited a selective and reversible response characteristics towards H_2S with maximum response at an operating temperature of 250˚C and 200˚C, respectively. A negligible response towards other interfering gases was observed. Thin film sensors exhibited an enhanced response in comparison to that of pellets.

Keywords: Fe_2O_3; Thin Film; Pellets; H_2S Sensor

1. Introduction

H_2S is a colorless, highly flammable and toxic gas, which in low concentration has an offensive odor similar to that of rotten eggs. As the Threshold Limit Value (TLV) for H_2S is 10 ppm, there is a need for its detection in either ppm or sub ppm level [1-3]. α-Fe_2O_3, an intrinsically n-type semiconductor has been widely exploited as a gas sensitive material owing to its good structural and thermodynamical stability along with resistance to photo-corrosion. In the present work, H_2S sensing characteristics of Fe_2O_3 in both thin film and conventional forms have been investigated and compared. Our results indicate that thin film based sensors exhibited better response towards H_2S in comparison to that of pellets.

2. Experimental

Fe_2O_3 thin films were prepared in two steps. In the first step, Fe films with ~100 nm thickness were deposited onto pre-cleaned (0001) Al_2O_3 substrates using electron-beam evaporation under a base vacuum of ~1.3×10^{-4} Pa. A high purity (99.99%) Fe powder was cold pressed in the form of a pellet and used as source material. These films were subsequently subjected to thermal oxidation at 800˚C in an oxygen environment (O_2 flow: 50 sccm) for 2 h. Bulk sensors in the form of pellets were prepared by cold pressing commercial Fe_2O_3 powder followed by sintering at 800˚C for 2 h. The response characteristics were recorded as a function of temperature, gas (H_2S, C_2H_5OH, NH_3, CH_4, CO, CO_2, NO, and Cl_2) and their concentrations using a static setup as described elsewhere [4]. For this purpose, the sensor was mounted on a Pt-100 based heater assembly in a leak-tight glass chamber (net volume: 500 ml). Electrical contacts were made by thermally depositing two Au pads (120 nm thick). A desired concentration of the test gas in the chamber was achieved by injecting a known quantity of the test gas using a micro-syringe. The sensor response was calculated using the relations:

$$R\% = \left(\frac{G_g - G_a}{G_a}\right) \times 100 \quad (1)$$

where G_g and G_a are conductance in the presence of test gas and air, respectively.

3. Results

Figure 1 shows the X-ray diffraction (XRD) pattern ob-

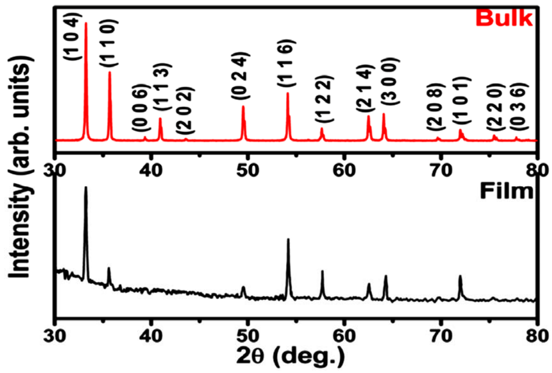

Figure 1. XRD diffraction pattern for Fe_2O_3 in bulk and thin film form.

tained for Fe_2O_3 thin film and bulk samples. All the peaks could be assigned to the single phase of Fe_2O_3 (hematite). The least-square fitting of the pattern indicated hexagonal rhombo-centred cubic unit cell structure with lattice parameters a = b = 5.028 Å, c =13.73 and $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$, which are in agreement with the reported values [JCPDS card no. #79-0007].

Figure 2 is a plot of % response recorded for both the sensor films as a function of operating temperature towards 10 ppm of H_2S. It is clearly evident that thin films and bulk sensors exhibit response maxima at 250˚C and 200˚C, respectively. At all the temperatures the response of thin films is higher than that of bulk samples. For instance, thin film sensors show, response maxima of 262% in comparison to 112%, exhibited by bulk samples towards 10 ppm H_2S.

A Typical response curves for both the sensor films towards 10 ppm H_2S is shown in **Figure 3**. The conductance of the sensors increases on exposure to H_2S indicating an n-type response. Thin film sensors exhibited a maximum response towards H_2S. A baseline drift was observed for bulk sensors.

Figure 4 shows the selectivity histogram recorded upon exposure to 10 ppm of various reducing and oxidizing test gases. It is clearly evident that both the sensors are highly selective towards H_2S. A negligible response towards all the other interfering gases was observed.

4. Conclusion

Comparative gas sensing properties of Fe_2O_3 in thin film and bulk form have been investigated. Both the sensor types exhibited an n-type response characteristic with highly selective response towards H_2S. An operating temperature for maximum response towards H_2S was found to be 250˚C and 200˚C for thin film and bulk sensors, respectively. For H_2S detection, thin film is a better sensor in comparison to bulk owing to the enhanced response and less baseline drift.

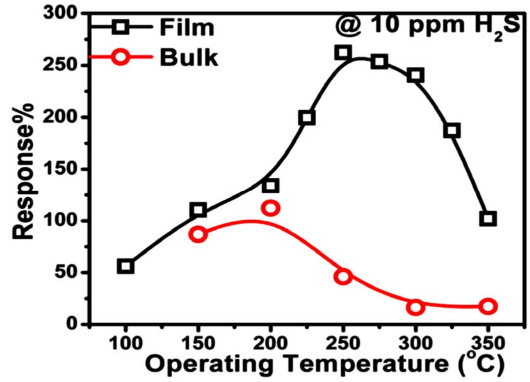

Figure 2. Sensor responses as a function of operating temperature.

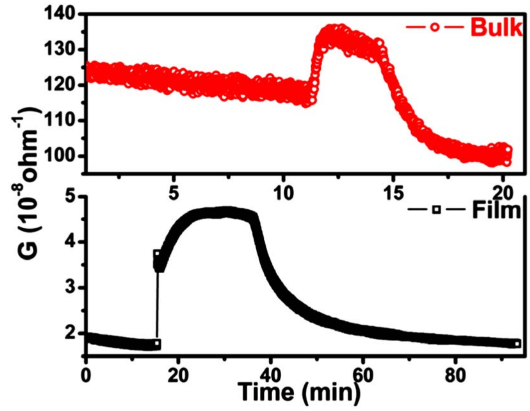

Figure 3. Sensor responses @ 10 ppm H_2S for bulk and thin film sensor.

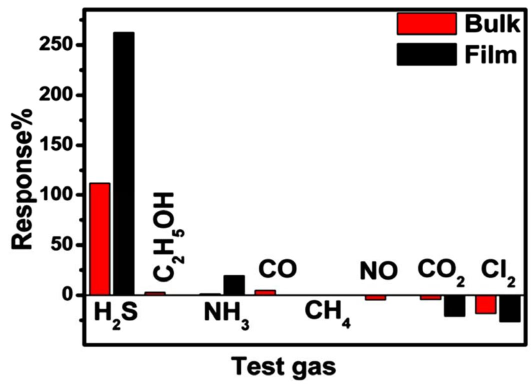

Figure 4. Selectivity data for bulk and thin films.

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