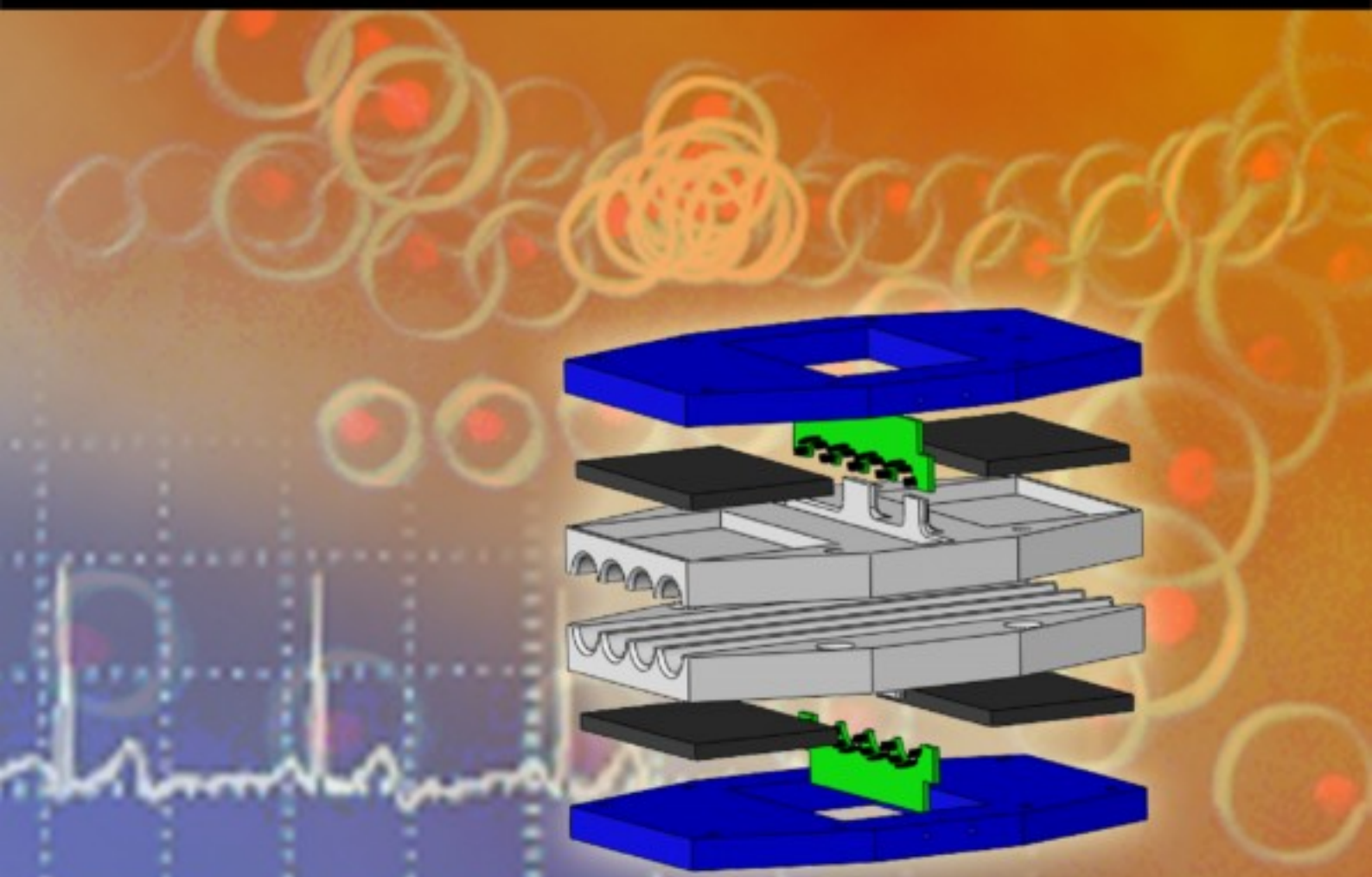


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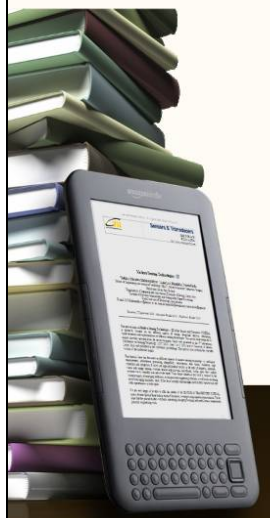
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- Biodevices
- Biomedical technologies
- Biological technologies
- Biomanufacturing

Important deadlines:

Submission (full paper)	January 10, 2011
Notification	February 20, 2011
Registration	March 5, 2011
Camera ready	March 20, 2011

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Notification	February 20, 2011
Registration	March 5, 2011
Camera ready	March 20, 2011

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- EDNA: Emergency Services and Disaster Recovery of Networks and Applications
- IPv6DFI: Deploying the Future Infrastructure
- IPDy: Internet Packet Dynamics
- GOBS: GRID over Optical Burst Switching Networks



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- Safety in industrial systems
- Complex Systems

Studies on Gas Sensing Performance of Pure and Surface Chrominated Indium Oxide Thick Film Resistors

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Abstract: The thick films of AR grade In₂O₃ were prepared by standard screen-printing technique. The gas sensing performance of thick film was tested for various gases. It showed maximum gas response to ethanol vapor at 350 °C for 80 ppm. To improve the gas response and selectivity of the film towards a particular gas, In₂O₃ thick films were modified by dipping them in an aqueous solution of 0.1 M CrO₃ for different intervals of time. The surface chrominated (20 min) In₂O₃ thick film showed maximum response to H₂S gas (40 ppm) than pure In₂O₃ thick film at 250 °C. Chromium oxide on the surface of the film shifts the gas response from ethanol vapor to H₂S gas. A systematic study of sensing performance of the sensor indicates the key role played by chromium oxide on the surface of thick film. The selectivity, gas response and recovery time of the sensor were measured and presented. *Copyright © 2010 IFSA.*

Keywords: Ethanol vapor sensor, Surface chromination, H₂S gas sensor, Gas response, Selectivity.

1. Introduction

Hydrogen sulphide (H₂S) is a toxic and inflammable gas produced in sewage plants, coalmines and oil and natural gas industries. It is used in a large amount in various chemical industries, research laboratories and as a process gas in the production of heavy water. Although the occupational 'exposure-limit' for the gas is 10 ppm for 8 h. exposure and the acceptable ambient levels of H₂S (recommended by the scientific Advisory Board On Toxic Air Pollutants, USA) are in the range

of 20-100 ppb [1]. Xu et al. have reported H₂S gas sensing by thick film of nano crystalline In₂O₃ at 250 °C [2]. However, so far there are no reports on sensing of low concentration or room temperature detection of H₂S gas by In₂O₃. M. Kaur et al. showed that the single crystal whiskers were selective and could be used to sense very low concentration (200 ppb) of hydrogen sulphide gas [3]. Even at low concentration, H₂S gas produces severe effect on the nervous system. This gas can be very perilous for human bodies when its concentration is greater than 250 ppm. Therefore a lot of attention has been paid to search for H₂S gas sensitive materials [4-7]. An efficient gas sensor must have high sensitivity and good selectivity at low operating temperature [8]. Jiao et al. found that an In₂O₃ film had high sensitivity to concentration of NO₂ but low sensitivity to deoxidizing gases [9]. The sensors for toxic gases have attracted much attention due to the growing concern of environmental protection and safety. A number of semiconductor oxides such as ZnO, SnO₂ and In₂O₃ are used for different gas sensors [10-13]. Most of these sensors are based on the resistance variation when the semiconductor oxide films are exposed to the target gases. Normally, the conventional resistance type sensors are operated at an elevated temperature usually within the range of 250–300°C [14-17]. The operating temperature of all the Ag doped sensors is lower than that of the reported H₂S sensors [18-21]. The amount of oxygen adsorbed on sensor surface depends on the operating temperature, the particle size and specific surface area of the sensor [22].

B. Shouli et al. explain the enhancement of gas-sensing properties of the semiconducting CH₄ sensor could be attributed to the much smaller crystalline size of SnO₂. The adsorption behavior and chemical reaction of CH₄ and O₂ on the composite surface and introduction of the additives into the basic material, the sensing behavior was straight forwardly related to the catalytic activity for CH₄ oxidation [23-24]. Among all the possible oxides, indium-based metal oxides have been very effectively resulted as material for gas sensor making [25-28]. The Sn-doped In₂O₃-based sensor showed higher conductance than In₂O₃, due to n-doping of Sn cations in the In₂O₃ lattice, and higher defectiveness than pure oxide [29].

In this article, we reported that pure In₂O₃ thick film showed maximum gas response to ethanol vapor at 350 °C. After the surface chromination (20 min) by CrO₃ precursor, the thick film of material showed maximum gas response to H₂S gas at 250 °C by suppressing the response to ethanol vapor.

2. Experimental

2.1. Preparation of In₂O₃ Thick Films

The AR grade powder of In₂O₃ was calcined at 1000 °C for 6 h. Then In₂O₃ powder was milled for 2 h. using planetary ball mill to obtain fine-grained powder. The thixotropic paste was formulated by mixing the fine powder of In₂O₃ with a solution of ethyl cellulose (a temporary binder) in a mixture of organic solvents such as butyl cellulose, butyl barbital acetate and terpineol etc. The weight ratio of the inorganic to organic part was kept at 75:25 in formulating the paste. This paste was screen printed on a glass substrate in a desired pattern [30, 31]. The sensitivity of the films increases with the film porosity as well as with thickness of the film [32]. The films were fired at 550°C for 30 min. Silver contacts are made for electrical measurements.

2.2. Preparation of Surface Chrominated In₂O₃ Thick Films

The surface chrominated In₂O₃ thick films were obtained by dipping them in a 0.1 M aqueous solution of chromium trioxide (CrO₃) for different intervals of dipping time of 5, 10, 15, 20 and 30 min. These films were dried at 80 °C, followed by firing at 550 °C for 30 min. These prepared films are termed as 'surface chrominated' films [33-37]. The CrO₃ dispersed on the films; the surface was reduced to

Cr_2O_3 in firing process. CrO_3 is not thermally stable above its melting temperature (197°C) losing oxygen to give Cr_2O_3 . Cr_2O_3 is a stable compound.

2.3. Thickness Measurements

The thickness of the films was measured by using the Taylor-Hobson (Talystep, UK) system and was observed in the range from 65 to 70 μm . The reproducibility of the films' thickness was achieved by maintaining the proper rheology and thixotropy of the paste

2.4. Details of Gas Sensing System

The sensing performance of the sensors was examined using a 'static gas sensing system' [7]. There were electrical feeds through the base plate. The heater was fixed on the base plate to heat the sample under test up to required operating temperatures. A sample under test can be mounted on the heater. The Cr-Al thermocouple was mounted to measure the operating temperature. The output of the thermocouple was connected to a digital temperature indicator. A gas inlet valve was fitted at one of the ports of the base plate. The required gas concentration inside the static system was achieved by injecting a known volume of a test gas with a gas-injecting syringe. A constant voltage was applied to the sensor and the current was measured by digital Picoammeter. Air was allowed to pass into the glass chamber after every gas exposure cycle.

3. Characterization Results

3.1. Micro Structural Analysis

Scanning electron microscopic (SEM) studies were carried out by using JEOL JSM 6360 (LA) Germany. Fig. 1 (a) depicts the SEM image of pure In_2O_3 thick film fired at 550°C . The film consists of voids and a wide range of grains with size distribution ranging from 0.25 μm to 0.5 μm , distributed non-uniformly.

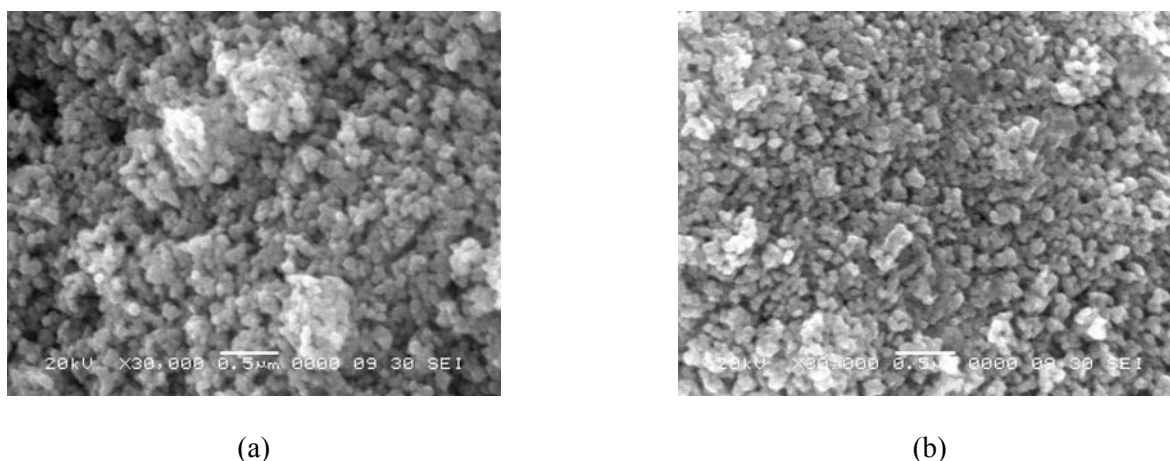


Fig. 1. SEM images of (a) Pure and (b) Surface chrominated (20 min) In_2O_3 thick films.

Fig. 1 (b) is the SEM image of the surface chrominated (20 min) In_2O_3 thick film. The Cr_2O_3 grains associated with the In_2O_3 grains. Cr_2O_3 grains may reside in the intergranular regions of the In_2O_3 .

Thus the effective surface area was expected to increase largely. This may be the reason of giving maximum gas response of the film. The smaller particles may be attributed to the Cr₂O₃ grains.

3.2. Quantitative Elemental Analysis-EDAX

The quantitative elemental analysis of the pure and surface chrominated In₂O₃ thick films was carried out by using an energy dispersive spectrophotometer (EDS) JEOL – JED – 2300 LA Germany. The constituent elements such as In, O and Cr associated with various films are represented in Table 1.

Table 1. Quantitative elemental analysis of pure and surface chrominated In₂O₃ thick films.

Film	In (wt%)	O (wt%)	Cr (wt%)
Pure In ₂ O ₃	41.33	58.67	-
Surface Chrominated, Dipping time: 5 min	39.03	60.30	0.94
Dipping time: 10 min	39.24	59.67	1.10
Dipping time: 15 min	39.37	59.00	1.62
Dipping time: 20 min	38.46	58.96	2.58
Dipping time: 30 min	35.97	60.50	3.53

It is clear from the Table 1 that the weight percentage of chromium increased with dipping time reached to a maximum. The film with the dipping time of 20 min was observed to be more oxygen deficient (58.96 wt%). This oxygen deficiency may make the sample possible to adsorb a large amount of oxygen species.

3.3. Thermal Stability of Pure and Surface Chrominated (20 min) In₂O₃ Samples

Thermo gravimetric analysis (TGA) of the films was conducted in air using Mettler Toledo Star System 851 at a heating rate of 10 °C min⁻¹ in the temperature range from 30 °C to 900 °C. Fig. 2 shows the TGA profiles of pure and surface chrominated (20 min) In₂O₃ samples. Table 2 lists ‘loss’ or ‘gain’ in weight of these films observed during TGA in the different temperature range. It can be concluded from the profiles that the weight of pure sample continuously decreased up to temperature 680 °C and then remained constant in the temperature range 680 °C to 820 °C. It shows smallest weight gain in the temperature range 820 °C to 900 °C.

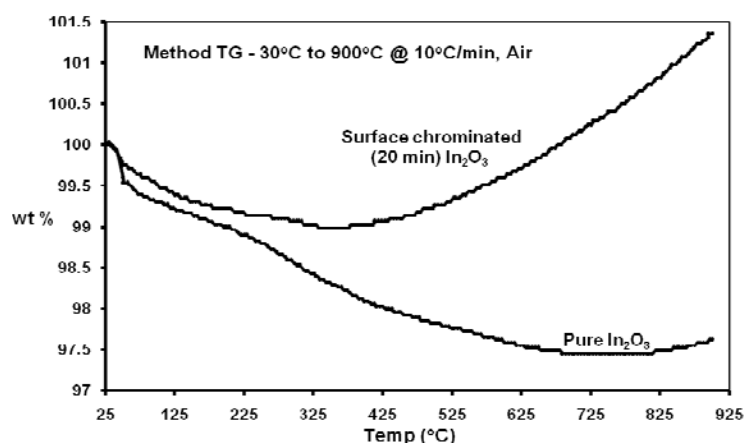


Fig. 2. TGA profile of: pure and surface chrominated (20 min) In₂O₃ samples.

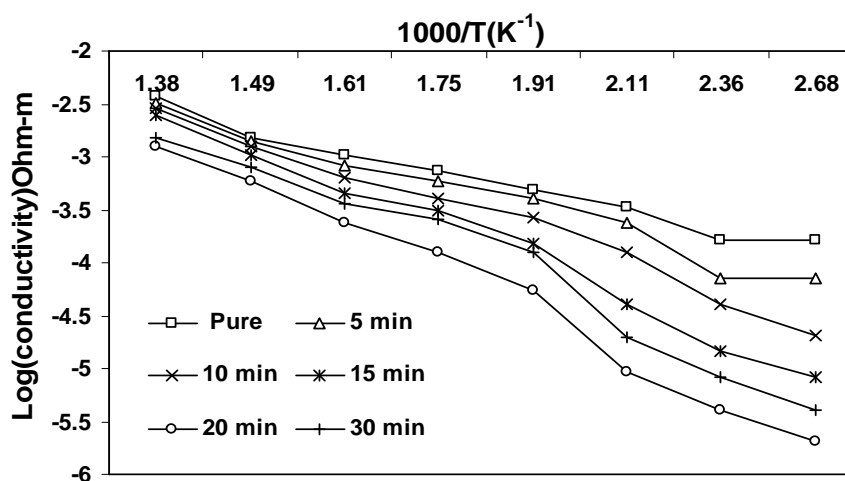
Table 2. Thermal analysis of pure In₂O₃ and surface chrominated (20 min) In₂O₃ samples.

Temperature °C	Pure In ₂ O ₃ sample		Temperature °C	Surface chrominated (20 min) In ₂ O ₃ sample	
	Loss (wt%)	Gain (wt%)		Loss (wt%)	Gain (wt%)
25 - 680	2.57	--	25 - 330	1.0	--
680 - 820	---	---	330 - 375	---	---
820 - 900	--	0.11	375 - 900	--	2.29

Surface chrominated (20 min) In₂O₃ sample was more stable than the pure In₂O₃ sample. The variation in weight with temperature was comparatively a less weight loss and a more weight gain in the surface chrominated (20 min) sample can be attributed to the larger adsorbed oxygen content. The film with the high amount content of Cr (2.58 wt%) was observed to contain the small amount of oxygen (58.96 wt%, Table 1), which could be attributed to the largest deficiency of oxygen in the film. It is therefore quite possible that the material would adsorb the largest possible amount of oxygen, showing relatively larger gain in weight (2.29 wt%) in the temperature range of 375 °C to 900 °C. The small weight loss of surface chrominated (20 min) In₂O₃ sample may be due to its larger stability. The chromium oxide on the surface of surface chrominated (20 min) sample would form misfit (Cr₂O₃) regions between the grains of In₂O₃ as compared to pure In₂O₃ sample and could act as an efficient catalyst for oxygenation.

3.4. Dependence of Electrical Conductivity with Temperature

Fig. 3 represents the variation of conductivity with temperature for the pure and surface chrominated samples. The conductivity varied nonlinearly with temperature for all samples, showing negative temperature coefficient. The surface chromination makes the sample more and more resistive, which in turn beneficial for improvement in response and selectivity.

**Fig. 3.** Electrical profile of pure and surface chrominated In₂O₃ thick films.

3.5. Measurement of Gas Response

In the field of gas sensors, it is well known that ethanol vapor is one of the most exhaustively studied gases, particularly due to the great demand in the biomedical, chemical, and food industries, especially in wine-quality monitoring and breath analysis.

Gas response of a sensor was defined as the ratio of the change in conductance of a sample on exposure to the test gas to the conductance in air [38].

$$\text{Gas response} = \left| \frac{G_g - G_a}{G_a} \right| = \left| \frac{\Delta G}{G_a} \right|$$

where G_g & G_a are conductance of a sample in the presence and absence of a test gas respectively & ΔG is the change in conductance.

The pure In_2O_3 thick film was tested to various gases such as CO_2 , CO , H_2S , Cl_2 , H_2 , NH_3 , O_2 , LPG, ethanol vapor etc. It showed maximum response to ethanol vapor at 350°C . Fig. 4 shows the variation of gas response of the pure In_2O_3 thick films (fired at 550°C) with operating temperature ranging from 100°C to 400°C to ethanol vapor. The gas response goes on increasing with operating temperature and attains maximum gas response (936) at 350°C and decreases with a further increase in temperature.

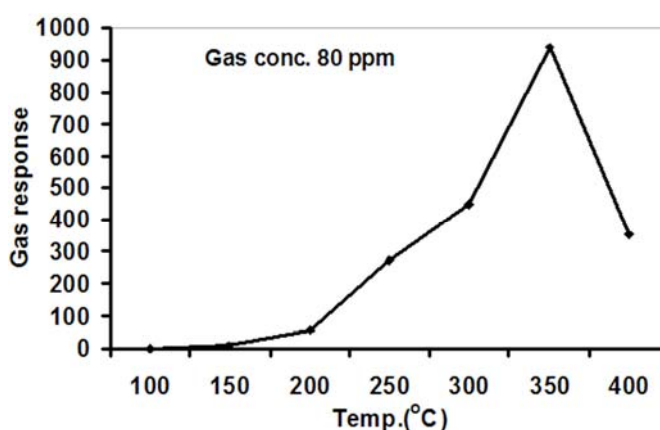


Fig. 4. Variation of ethanol vapor response of pure In_2O_3 thick film with operating temperature.

3.6. Selectivity of Pure In_2O_3 Thick Film

Selectivity or specificity is defined as the ability of a sensor to respond to certain gas in the presence of other gases. Fig. 5 shows the selectivity profile of the pure In_2O_3 thick film at 350°C . The film shows a maximum selectivity to ethanol vapor against the other gases.

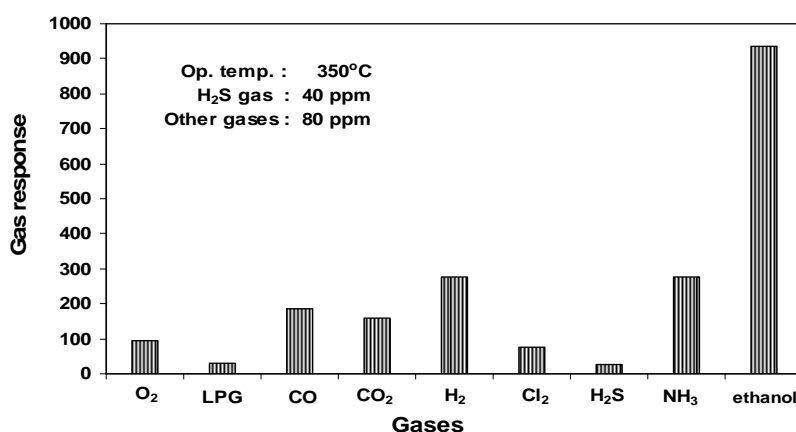


Fig. 5. Selectivity of pure In_2O_3 thick film.

3.7. Variation of Gas Response with Dipping Time

Fig. 6 shows variation of response to H₂S (40 ppm) with operating temperature of the pure and surface chrominated In₂O₃ thick films fired at 550 °C. The response goes on increasing with the operating temperature, attains maximum gas response (at 250 °C) and then decreases with a further increase in operating temperature. It is clear from Fig. 6 that the optimum operating temperature is 250 °C.

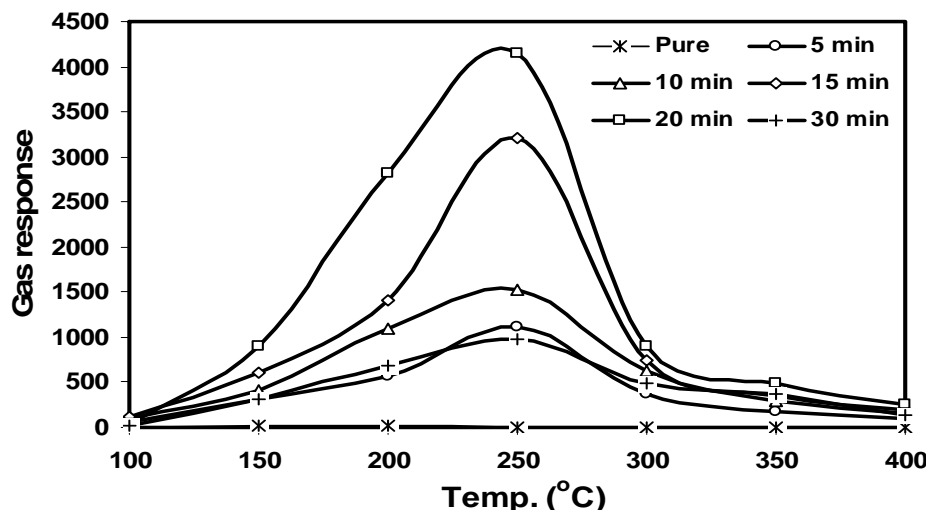


Fig. 6. Variation of H₂S gas response to pure and surface chrominated In₂O₃ thick films with operating temperature.

The H₂S response of pure In₂O₃ thick film fired at 550 °C was 2.73 at 250 °C while that of surface chrominated (20 min) In₂O₃ thick film was 4142 at the same operating temperature. Therefore, the surface chromination of In₂O₃ was observed to be more effective for H₂S gas sensing than the pure In₂O₃.

The surface chrominated (20 min) In₂O₃ thick film showed maximum gas response to H₂S gas. The amount of Cr (2.58 wt%) incorporated onto the In₂O₃ thick film surface would be optimum to cover the surface nonuniformly, leading to the enhanced adsorption mechanism. The gas response to H₂S gas goes on increasing with increasing the amount of Cr up to a certain limit attains maximum gas response and decreases with increasing the amount of Cr. The largest gas response in case of the sample with 2.58 wt% of chromium (for 20 min) may be because of more available sites (chromium misfits). The surface chromium misfit regions enhance the oxygen adsorption on the surface. Thus the number of oxygen species adsorbed on the activated surface would be larger. The larger the number of oxygen species adsorbed, the faster would be oxidation of H₂S gas. This would increase the conductance of the film crucially, enhancing gas response. At lower concentration, the decrease in gas response may be due to the insufficient number of Cr- misfits available on the film surface, which would adsorb less oxygen species on the film surface. However, at higher concentrations, the chromium oxide would mask the entire base material and would resist the gas to reach to the surface active sites, so gas response would decrease further [39].

3.8. Selectivity of Surface Chrominated (20 min) In₂O₃ Thick Film to Various Gases

Fig. 7 shows the bar diagram indicating the selectivity of the surface chrominated (20 min) In₂O₃ sensor operated at 250 °C to H₂S gas against CO, LPG, NH₃, CO₂, Cl₂, O₂, H₂ and ethanol vapor. It is

evident from the figure that the sensor was highly selective to H₂S against other gases. The high selectivity to H₂S can be attributed to the surface chromination of In₂O₃ thick film.

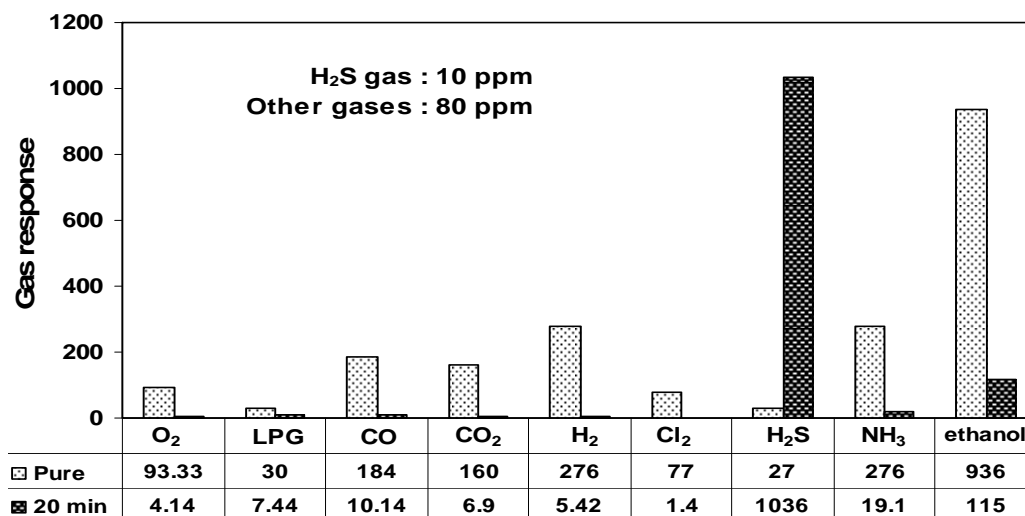


Fig. 7. Selectivity of surface chrominated (20 min) In₂O₃ thick film to H₂S gas with other gases at 250 °C.

3.9. ppm-Level Gas Sensing

Fig. 8 is the histogram which shows the gas response of a surface chrominated (20 min) In₂O₃ sensor to H₂S gas up to 500 ppm gas concentration at 250 °C. At lower concentration, a monolayer of the gas molecules would be expected to be formed on the surface, which could interact with the surface more actively, giving larger response. There would be multilayer of gas molecules on the sensor surface at the higher gas concentration, resulting in saturation in response.

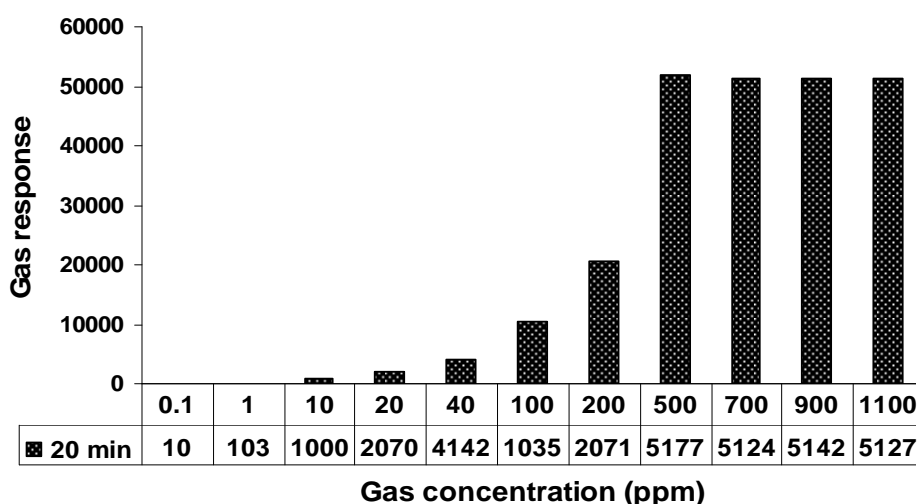


Fig. 8. ppm-level H₂S gas response of surface chrominated (20 min) In₂O₃ thick film at 250 °C.

3.10. Shifting of Gas Response

Fig. 9 shows shifting of gas response of pure In₂O₃ thick film to the surface chrominated (20 min) In₂O₃ thick film. The thick film of pure In₂O₃ was sensitive to ethanol vapor (936) at 350 °C. The

surface chrominated (20 min) In_2O_3 thick film suppresses the response to ethanol vapor and enhances to H_2S gas at lower temperature. The chromination changes the reactivity of film surface to oxidize the adsorbed gas more effectively.

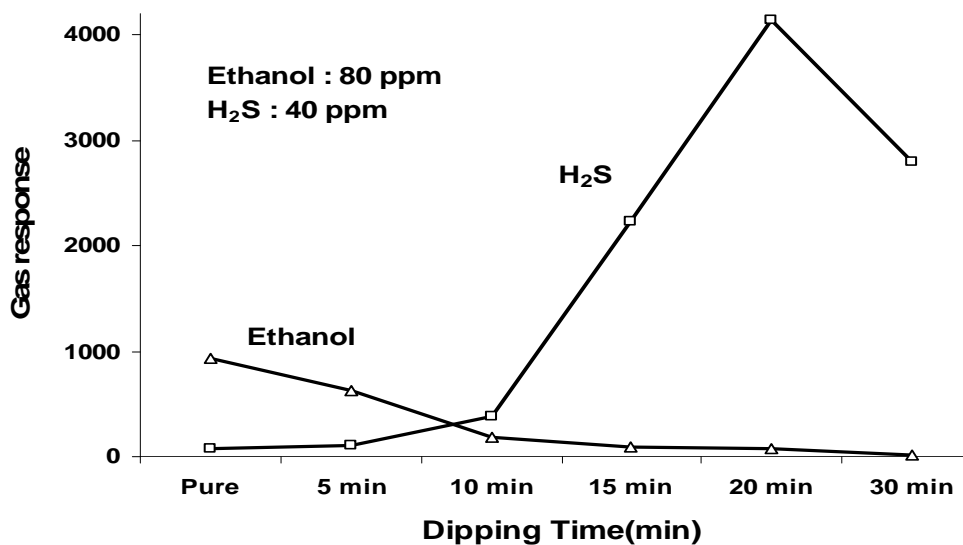


Fig. 9. Shifting of gas response with dipping time.

3.11. Response and Recovery Time

Response and recovery time are basic parameters of the gas sensors. The response time has been defined as the time taken to attain 90 % of final value and the recovery time as time taken to recover 90 % of the original value. The Fig. 10 shows the response time and recovery time of surface chrominated (20 min) In_2O_3 thick film at 250 °C for 1 ppm H_2S gas concentration. The 90 % response and recovery time were attained within 4 s and 12 s respectively. The very short response and recovery time are the important features of the surface chrominated (20 min) In_2O_3 sensor. The sensor also showed good repeatability as shown in Fig. 10.

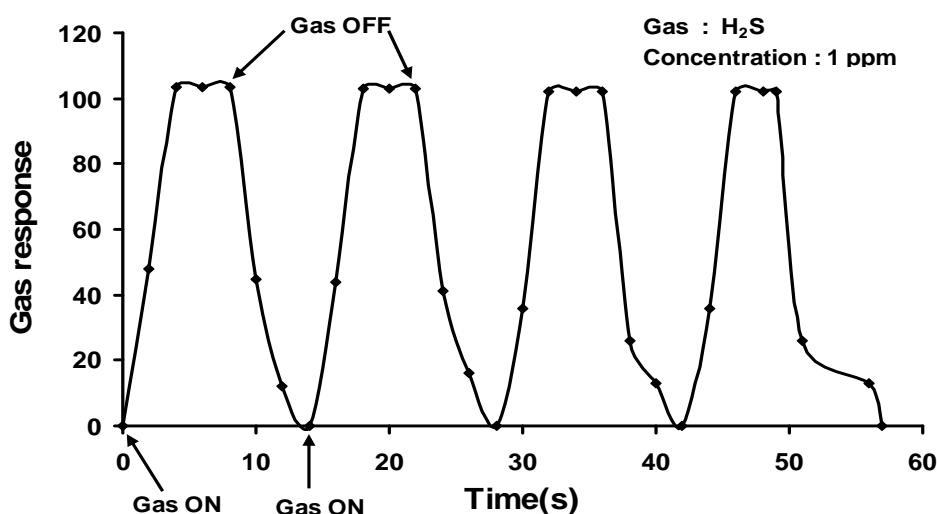


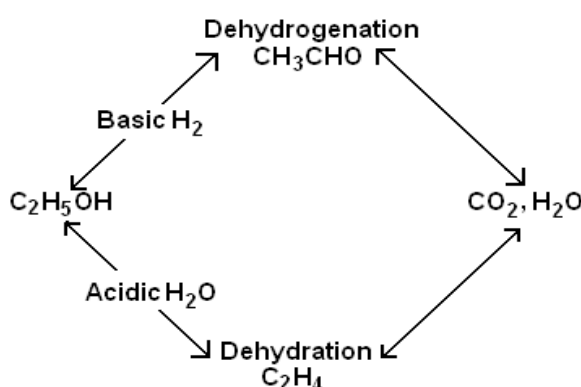
Fig. 10. Repeatability of surface chrominated (20 min) In_2O_3 thick film.

4. Discussions

4.1. Pure In₂O₃ Thick Film as a Ethanol Sensor

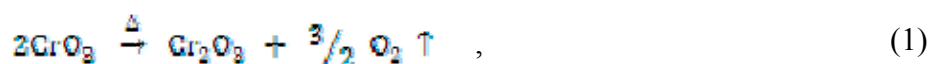
Pure In₂O₃ is a basic oxide. It is well known that the response to ethanol vapor is greatly promoted by basic oxides [40]. Being specific to ethanol vapor, the promotion would be related to the oxidation of ethanol vapor. It is known in catalytic chemistry that ethanol vapor is oxidized via two reaction routes, i.e. dehydrogenation to CH₃CHO on the basic surface and dehydration to C₂H₄ on the acid surface. These intermediates are consecutively oxidized to CO₂ and H₂O:

Out of these intermediates, CH₃CHO is known to have much higher molecular sensitivity of a semiconductor gas sensor than C₂H₄ [41]. Due to the basic nature of In₂O₃, the dehydrogenation route is more favored than the dehydration route, giving rise to the maximum gas response to ethanol vapor.



4.2. Surface Chrominated (20 min) In₂O₃ Thick Film as a H₂S Sensor

The surface chrominated (20 min) In₂O₃ thick film can be looked upon as the small particles of chromium oxide distributed along the grain boundaries of In₂O₃. CrO₃ on the surface of In₂O₃ thick film is not thermally stable above its melting temperature (197 °C) losing oxygen to give, after series of intermediate stages, Cr₂O₃ [42]. It is usual procedure, in this investigation, to test the sensors by heating them at the operating temperatures ranging from 50 to 450°C. The first time heating of sensor, above 200 °C, would convert CrO₃ into Cr₂O₃. Some amount of CrO₃ may also be reduced into Cr₂O₃ by trapping electrons from the oxygen vacancies in In₂O₃ thick film material. Local oxygen deficiency may trigger the occurrence of the reaction:



On exposure of H₂S gas, Cr₂O₃ would be converted into Cr₂S₃ or CrS, which are known to be metallic in nature and more conducting than Cr₂O₃ [43]. On exposure of H₂S gas on the surface chrominated (20 min) In₂O₃ thick film, the sensor resistance decreases suddenly giving higher sensitivity. This can be represented as:



or



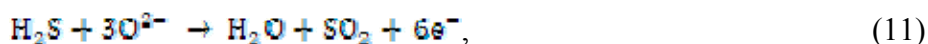
On subsequent exposure of sensor to O₂ at elevated temperatures, sulphides can be recovered back to oxides as:



When oxygen is adsorbed on the chromium zones of strong localization at elevated temperatures, the potential between the surface chrominated (20 min) In₂O₃ thick film, grains may be raised further and as a result the total resistance increases in comparison with the sample without any chromium. It is known that abstraction of electrons from bulk of surface chrominated (20 min) In₂O₃ thick film by the adsorbed oxygen results in the formation of surface states. The semiconductivity in Cr₂O₃-activated In₂O₃ must be due to large oxygen deficiency in it the material would then adsorb the oxygen species at higher operating temperatures [44].



Upon exposure to H₂S gas on the surface chrominated (20 min) In₂O₃ thick film, H₂S adsorb between the chrominated grains of In₂O₃, the potential barrier would be decreased as a result of oxidative conversion of the H₂S and desorption of oxygen. The reaction of H₂S gas with the adsorbed oxygen ions can be represented as [45].



The amount of oxygen adsorbed on the surface of chrominated (20 min) In₂O₃ thick film is more since chromium oxide form misfit regions between the grains of surface of chrominated (20 min) In₂O₃ thick film and act as efficient catalysts for oxygenation. The Cr₂O₃ can create artificial surface states in the midgap region, leading to unusual physical and chemical properties. For example, the adsorption energy can be higher for the misfit regions, and the discontinuity in the adsorption potential can give rise to unusual selectivity effects for surface of chrominated (20 min) In₂O₃ thick film based semiconducting oxide sensors. More specifically, the electron-electron interaction in the presence of periodically enhanced disorders in these two-dimensional systems can affect the adsorbate adsorbent interaction and the range of adsorption potentials leading to additional sensitivity improvement.

When the optimum amount of chromium oxide is incorporated on the surface of the surface of chrominated (20 min) In₂O₃ thick film, chromium species would be distributed uniformly throughout the surface (Fig. 2 (b)). Due to this not only the initial resistance of the film is high but this amount would also be sufficient to promote the catalytic reaction effectively and the overall change in resistance on exposure of the test gas larger leading to high gas response.

When the amount of chromium oxide on the surface of the film is less than the optimum, the surface dispersion would be poor and amount may not be sufficient to promote the reaction more effectively leading to decreased sensitivity.

The observations from thermal analysis indicate that surface chrominated (20 min) In₂O₃ thick film is more stable than the pure In₂O₃ thick film. Therefore, reproducibility of chrominated samples is

expected to be more than pure In_2O_3 thick film. The oxygen adsorption mechanism of surface chrominated (20 min) In_2O_3 thick film is observed to be more effective as compared to the pure In_2O_3 thick film leading to higher gas response.

The nonlinearity of 'Electrical conductivity-temperature' profiles of the samples reveals the semiconductor nature of pure and surface chrominated (20 min) In_2O_3 thick film. The semiconducting nature may be attributed to oxygen deficiencies in surface chrominated (20 min) In_2O_3 thick film. The chromination of In_2O_3 thick film enhances the electrical conductivity. This may be due to the conducting nature, though small, of small particles of Cr_2O_3 segregated around the grain boundaries of surface chrominated (20 min) In_2O_3 thick film.

Fast recovery and response to H_2S gas may be due to faster adsorption-desorption reactions on the surface chrominated (20 min) In_2O_3 thick film. The large number of oxygen ions could adsorb on the misfit regions of chromium in absence of H_2S gas. These adsorbed oxygen ions could oxidize the H_2S gas immediately on its exposure leading to fast response and recovery of the sensor.

5. Conclusions

From the results, following statements can be made for the sensing performance of the pure and surface chrominated (20 min) In_2O_3 sensors.

- (1) The In_2O_3 was found to be a non stoichiometric and oxygen deficient material. It showed negative temperature coefficient in nature.
- (2) The pure In_2O_3 thick film showed highest gas response to ethanol vapor at 350 °C and gas concentration was at (80 ppm).
- (3) Surface chromination by dipping process are one of the most suitable methods of modifying the surface of the films.
- (4) The surface chromination changes the gas response of the film to H_2S at 250 °C temperature.
- (5) The Cr incorporated onto the film surface for 20 min is (2.58 wt%). This amount would be an amount to form sufficient number of misfits, which are responsible for the change in the adsorption capabilities of the film.
- (6) The gas response of the surface chrominated (20 min) In_2O_3 thick film was observed to increase suddenly upon exposure to H_2S at optimum operating temperature. The sensing mechanism of the surface chrominated (20 min) In_2O_3 sample was the surface controlled mechanism (adsorption/desorption of oxygen) the oxidation of sulphides (Cr_2S_3 , CrS) and the reduction of oxides (Cr_2O_3 , CrO) have also boosted the gas response and selectivity.
- (7) TGA data shows that, the surface chrominated (20 min) In_2O_3 sample is more stable than pure In_2O_3 sample. Therefore, reproducibility of surface chrominated (20 min) In_2O_3 samples is expected to be better than pure In_2O_3 sample. The adsorption of oxygen on the surface chrominated (20 min) In_2O_3 sample would be more effective as compared to pure In_2O_3 sample leading to higher gas response.
- (8) The repeatability and reproducibility of the sensor were observed improved through the surface chromination process.

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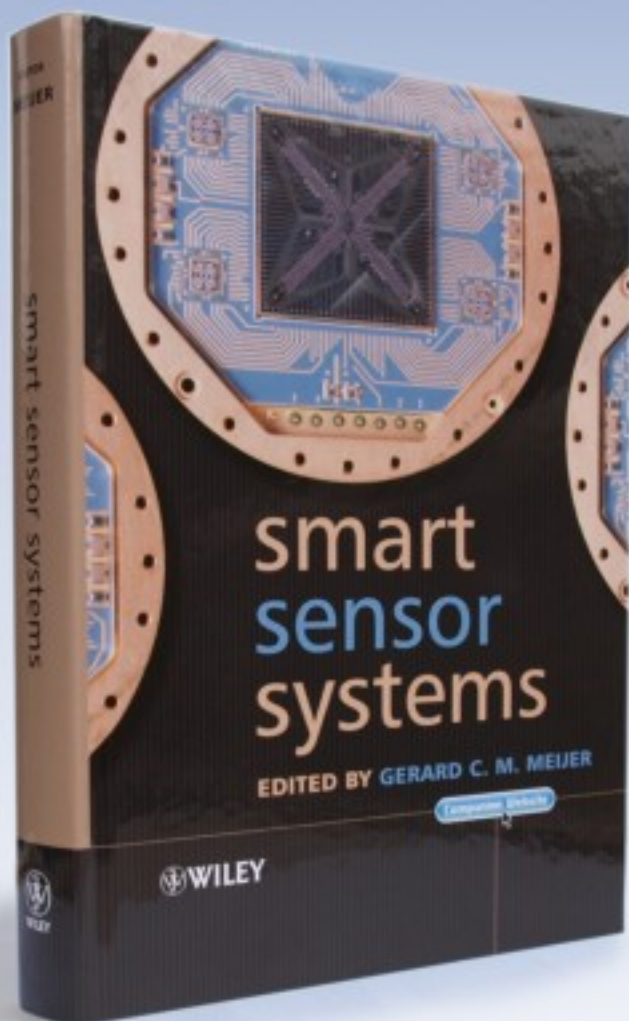
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