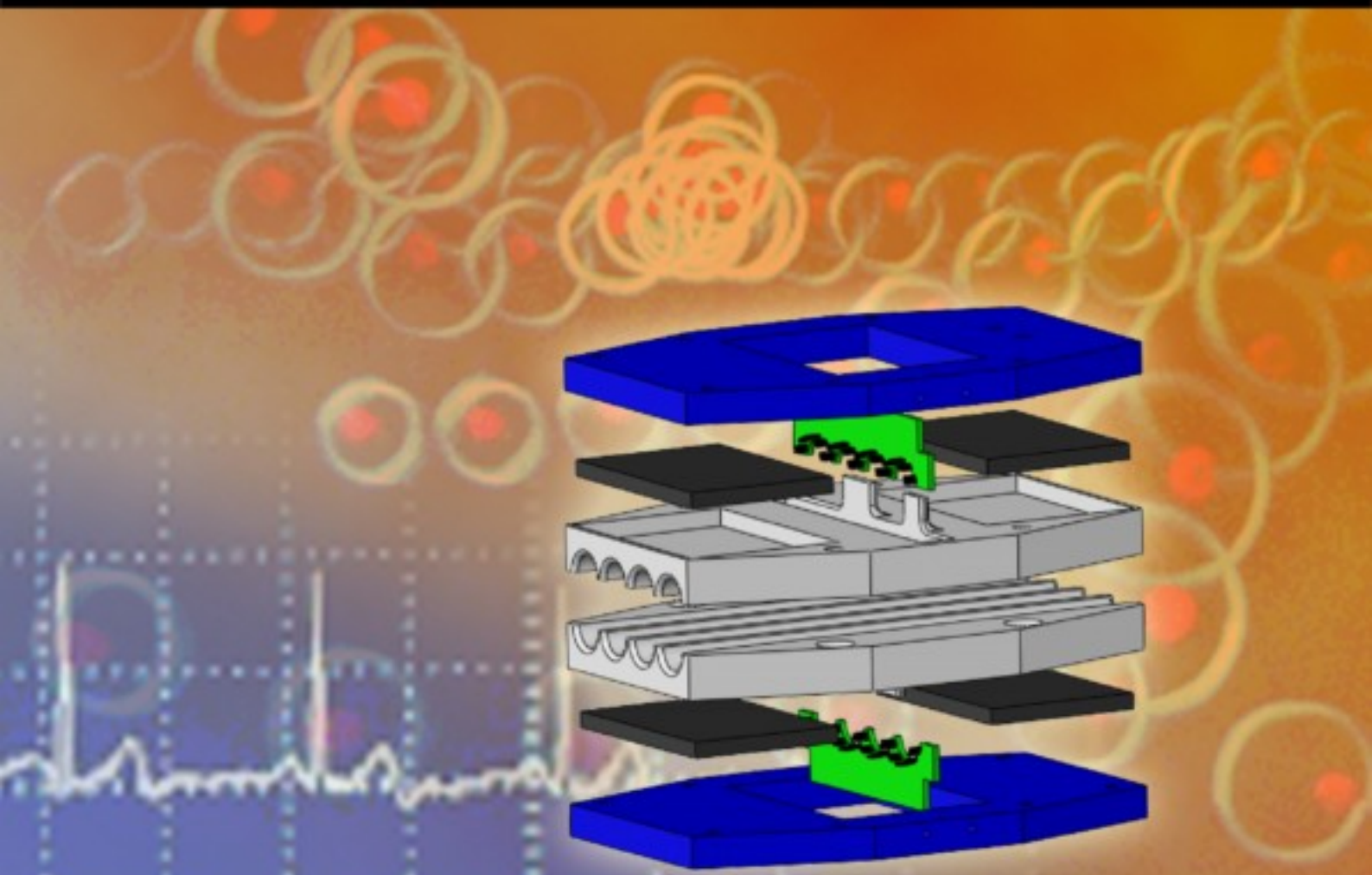


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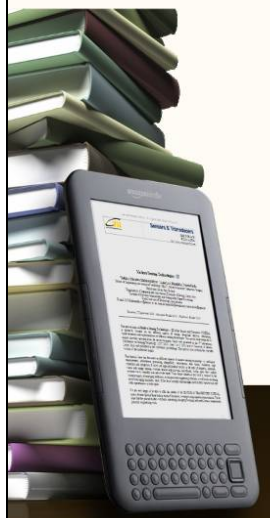
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Al-doped TiO₂ Thick Film Resistors as H₂S Gas Sensor

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Abstract: The characterization and H₂S gas sensing properties of Pure and Al-doped TiO₂ thick films were investigated. Thick films of pure and Al doped titanium dioxide were prepared by the screen printing technique. Pure TiO₂ was almost insensitive to H₂S gas. Thick films of Al (3 Wt. %) doped TiO₂ were observed to be highly sensitive to H₂S gas at 200 °C with fast response and recovery time. The results are discussed and interpreted. *Copyright © 2010 IFSA.*

Keywords: Al doped TiO₂, H₂S Gas sensor, Thick film, Gas response.

1. Introduction

Titanium dioxide (TiO₂) has been extensively studied owing to its wide range of applications which include photo catalysis, heterogeneous catalysis, energy storage, solar cell components, corrosion-protective coatings and optical coatings [1–5]. Titanium dioxide can be synthesized in three crystalline phases: rutile, brookite and anatase [6]. Rutile is more thermodynamically stable than other phases. Anatase phase is stable for TiO₂ at comparatively low temperatures. Titanium dioxide in the anatase crystalline phase is one of the most studied materials for photocatalysis. It has been shown that sensitivity of TiO₂ sensors can be improved by addition of dopants such as Cr, Nb, Sn, Al, Pt, La and Y. Most important effects of dopant addition in TiO₂ are increasing the conductivity, slowing down anatase to rutile transformation and reducing grain growth. [7, 8]. Among the various metal oxides that can be used in gas sensors, only those materials based on stannic oxide and titanium oxide have been widely manufactured and utilized [9]. The aforementioned background justifies the need to improve the properties and features of these oxides in order to obtain a more efficient material for gas sensing

purposes. The advantages of TiO₂ are that it is highly stable material at high temperatures and harsh environments and has thermal expansion coefficient matching with alumina making it suitable for the fabrication as thin or thick film based sensors [10]. TiO₂ doped Cr₂O₃ is reported to be sensitive to NO₂, O₂ and humidity [11-13]. Recently sensors based on Cr_{2-x}TiO₃[X=0.05-0.4] has been commercialized by capteur sensors [12].

Mostly in literatures pure and doped TiO₂ shows sensitivity to high temperatures for O₂, NO₂, Ethanol, H₂, LPG and CO gases rather than H₂S at any temperature [14, 15]. Hydrogen Sulfide is a toxic gas often produced in coal or natural gas manufacturing. The threshold limit value for Hydrogen sulfide is 10 ppm. When the concentration of H₂S is higher than 250 ppm, it is dangerous to human body and may cause death. Reliable, low cost H₂S consumption is in high demand for environmental safety and industrial control purposes. Numerous efforts have been directed to develop H₂S gas sensors by mainly employing solid electrolytes such as alkali metal sulfates [16] by adding hydrophobic silica [17], ZrO₂ [18], CeO₂ [19] to the sensor element. It was also found that addition of as noble metal, Ag makes the material very sensitive to H₂S [20]. Some well known materials for H₂S gas sensing are SnO₂-ZnO [21], SnO₂ [22], and BST films [23], etc. This article deals with preparation procedure of thick films of pure TiO₂ and Al-doped TiO₂ by a screen printing technique and their gas sensing performance. Studies were carried out and results are presented on the variation of sensitivity with different operating conditions, the selectivity of a sensor to identify a target gas and effect of target gas and the effect of catalysts on the sensing performance. The results are interpreted and summarized in the terms of the conclusions.

2. Experimental

2.1. Preparation of Pure and Doped TiO₂ Based Gas Sensors

Analar Reagent (AR) grade TiO₂ powder (99.9 %pure) and Al were mixed mechanochemically to obtain Al-doped TiO₂ powders in various weight percentages such as 1, 3, 5, 7, and 10 wt. %. This prepared powder was then calcined at 400 °C for 2 hours in muffle furnace. The ratio of inorganic to organic part was kept as 70:30. The inorganic part consists of a functional material (TiO₂), doping material (Al) and glass frit (70 wt.% PbO, 18 wt.% Al₂O₃, 9wt.% SiO₂ and 3wt.% B₂O₃) as permanent binder. The organic part consists of 8 % ethyl cellulose (EC) and 92% butyl carbitol acetate (BCA). TiO₂ powder with Al and 5 wt. % of glass frit were mixed thoroughly in an acetone medium with mortar and pestle to ensure sufficiently fine particle size. A solution of EC and BCA in the ratio of 8:92 was added drop wise until proper thixotropic properties of the paste were achieved. TiO₂ doped with Al thick films were prepared on alumina substrate by using a standard screen-printing technique. The printed pattern was allowed to settle for 15 to 20 minutes in air. The films were dried under IR radiation for 45 minutes to remove the organic vehicle and then fired at temperature of 800 °C for 2 hours in muffle furnace. During the firing process glass frit melts and the functional materials and dopants are sintered. The function of glass frit is to bind grains together and also to hold the film firmly to the substrate.

2.2. Characterization of Gas Sensors

The structural properties of TiO₂ films were investigated using X-ray diffraction analysis from 20-80° [Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having CuK α , λ =0.1542 nm radiation] with 0.1°/step (2 θ) at the rate of 2 sec/step. The scanning electron microscopy (SEM- JOEL JED-2300) was employed to characterize the surface morphology. The Composition of TiO₂ thick film samples were analyzed by Energy Dispersive spectrometer (JOEL-JED 6360 LA). The thickness of the TiO₂ thick films was measured by using Taylor-Hobson (Taly-step UK) system. The thickness of the films was

observed uniform in the range of 20 μm to 25 μm . The D.C. Resistance of the films was measured by using half bridge method in an air atmosphere at different temperatures.

2.3. Measurement of Sensing Characteristics

The gas sensing studies were carried out on a static gas sensing system under normal laboratory conditions. The H_2S gas response of thick films was studied in test assembly. The electrical resistances of thick film in air (R_a) and in the presence of H_2S gas (R_g) were measured to evaluate the gas response (S) given by the relation,

$$S (\%) = \frac{R_a - R_g}{R_a} \times 100 \%, \quad (1)$$

where R_a is the resistance of the thick film sample in air and R_g is the resistance of the thick film sample in a gas atmosphere. Response time was defined as the time needed for a sensor to attain the maximum change in resistance, while recovery time as the time taken to get back 90 % of the original resistance. The ability of a sensor to respond to a certain gas in the presence of other gases is known as selectivity [24].

3. Results

3.1. Elemental Analysis

The quantitative elemental compositions of the pure and doped TiO_2 films were analyzed using an energy dispersive spectrometer (EDS). The mass % of Ti and O in each sample was not as per stoichiometric proportion. The entire samples were observed to be oxygen deficient. The film doped with 3 Wt. % Al was observed to be most oxygen deficient, as evidenced from EDS analysis (Table 1). The deficiency or excess of the constituent material particles leads to the semiconducting nature of the material [23, 25].

Table 1. Composition of Al-doped TiO_2 obtained from EDS.

Element (Wt %)	TiO_2	Al-doped TiO_2				
		1wt. %	3wt. %	5wt. %	7wt. %	10wt. %
O	42.37	43.19	38.68	38.75	39.68	39.71
Ti	57.63	55.52	59.38	58.82	57.38	56.97
Al	--	1.59	1.94	2.43	2.94	3.32

3.2. Structural Analysis (X-ray Diffraction Studies)

Fig. 1 depicts XRD pattern (2θ - 80°) of the TiO_2 thick films loaded with x wt.% of Al ($x = 0, 1, 3, 5, 7$ and 10 wt.%) prepared by a screen printing method and fired at 800°C . The XRD patterns shows the film samples are polycrystalline in nature. It was observed that the both anatase and rutile phase coexisted in the TiO_2 -Al composite film samples fired at 800°C [26]. In the 2θ range, the 101 anatase peak, located at 25.8° , is present. This is the most pronounced peak of an anatase structure for film sample fired at 800°C . All values of (hkl) plane are matched with JCPDS data 21-1272 and 21-1276 for anatase and rutile structure respectively. XRD pattern shows that all samples were dominated by anatase phase. The electrical mobility of anatase films is much larger due to the smaller electron effective mass and also the Fermi level is higher by about 0.1 eV compared to the rutile structure. These properties are useful for gas sensing and optoelectronic [27-31]. The peaks of the XRD pattern correspond to TiO_2 and Al observed to be identical and polycrystalline in nature. As seen from XRD of Al-doped TiO_2 no peak was observed for Al, this may be due to Al^{3+} substituted in Ti^{4+} . G. N. Chaudhari et. al also reported similar observation for Al_2O_3 doped TiO_2 [32].

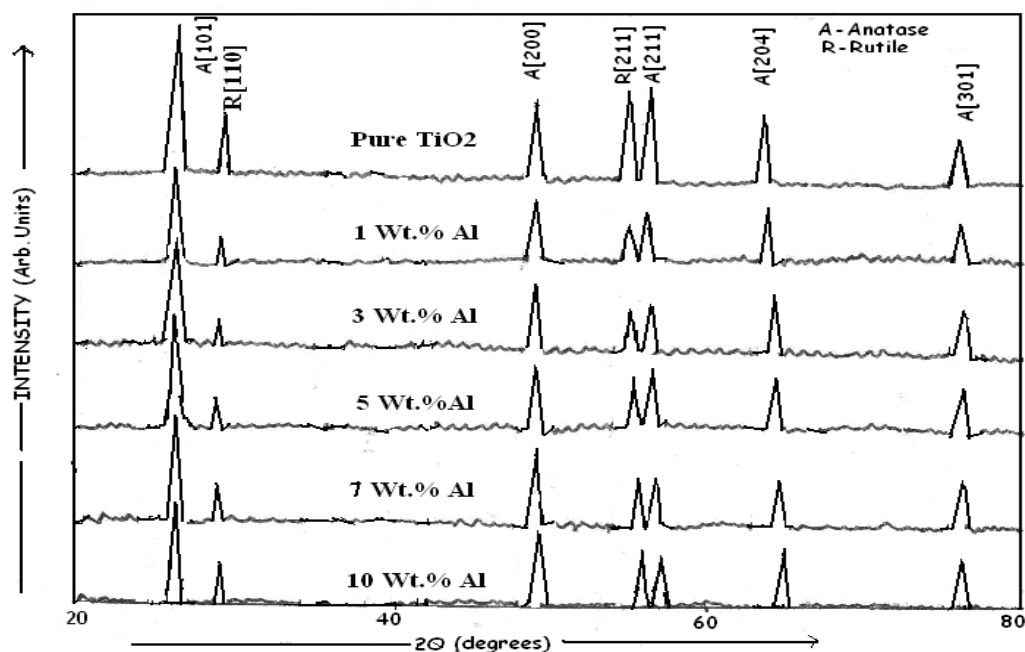


Fig. 1. XRD pattern of pure and Al-doped TiO_2 films.

3.3. Microstructural Analysis

Fig. 2 depicts the SEM images of pure and Al-doped TiO_2 films. Fig. 2 (a) depicts the microstructure of pure TiO_2 thick film. It consists of randomly distributed grains with smaller size and shape distribution. Fig. 2 (b, c, d, e and f) depicts the microstructure of Al-doped TiO_2 thick films. It is clear from these figures that the doping of Al affected the microstructure of TiO_2 . The film consists of voids and grains distributed nonuniformly. Fig. 2(c) depicts the microstructure of Al (3 Wt. %)-doped TiO_2 film which consists of ever smaller grains and comparatively porous with other films. Effective surface to volume ratio would be increased and large number of oxygen ions would be adsorbed as compared to the other films. The increased porosity increases the in-pore adsorption of oxygen and tends to improve the adsorption-desorption mechanism of target gas.

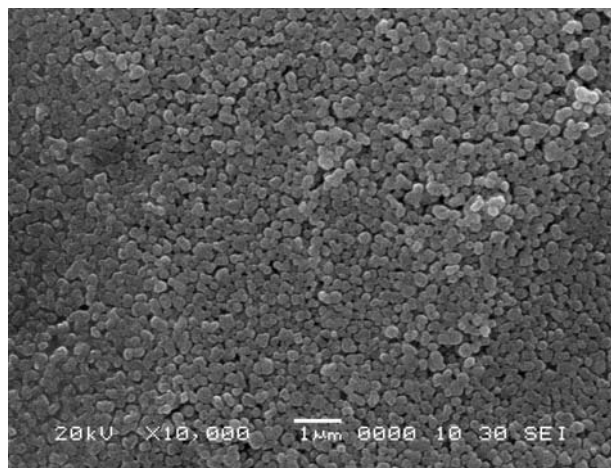


Fig. 2. (a) SEM image of pure TiO₂.

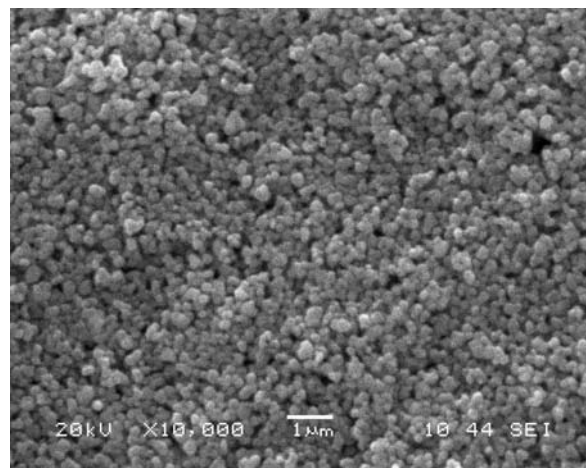


Fig. 2. (b) SEM image of 1 Wt. % Al-doped TiO₂.

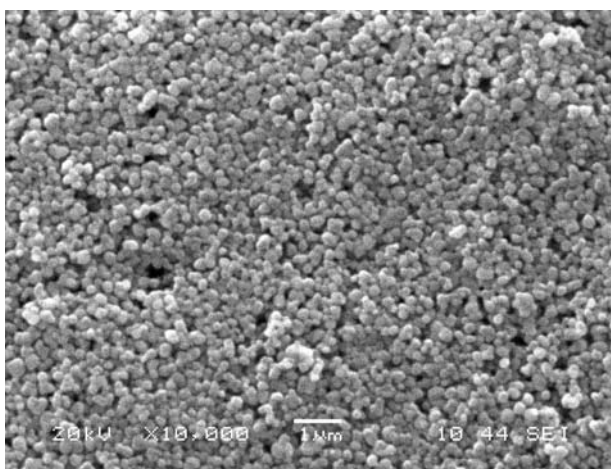


Fig. 2. (c) SEM image of 3 Wt. % Al-doped TiO₂.

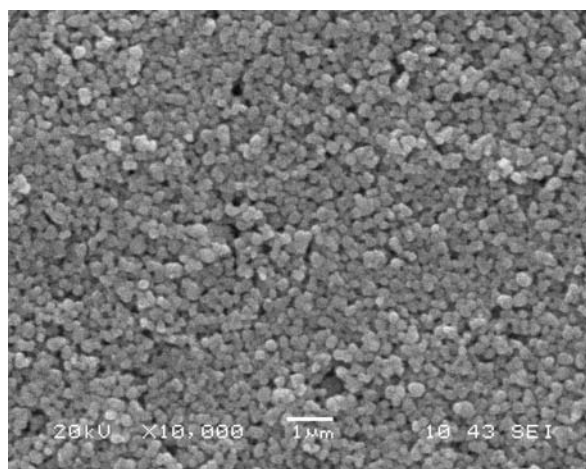


Fig. 2. (d) SEM image of 5 Wt. % Al-doped TiO₂.

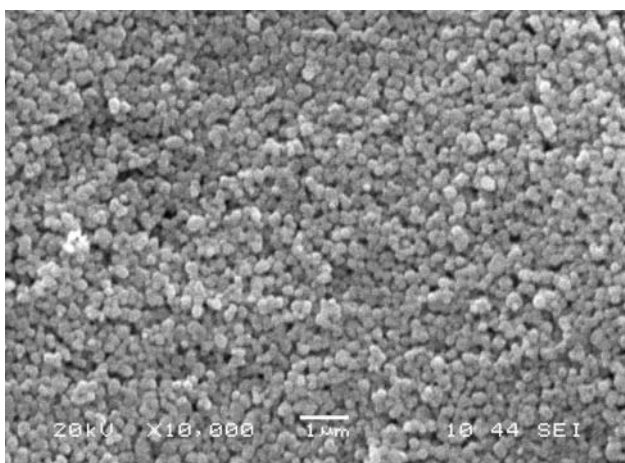


Fig. 2. (e) SEM image of 7 Wt. % Al-doped TiO₂.

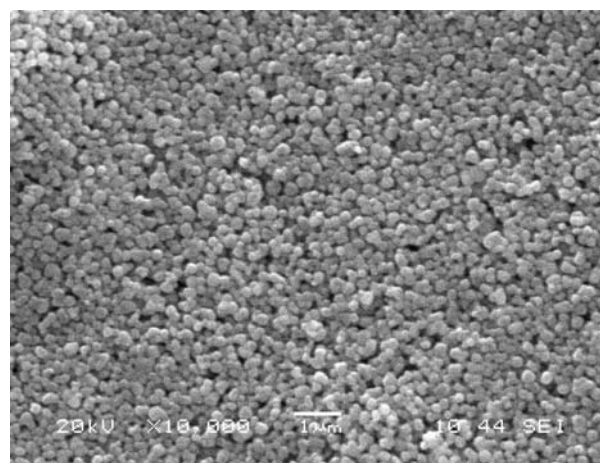


Fig. 2. (f) SEM image of 10 Wt. % Al-doped TiO₂.

3.4. Gas Sensing Characteristics

Electrical characterization of Al-doped TiO₂ composite thick films fabricated by screen printing method and fired at 800 °C has been carried out in a gas test chamber. The resistance of the films was measured by using equation (1). The resistance of the films decreased upon exposure to H₂S

(1000 ppm) diluted in air. Fig. 3 shows gas sensing response to 1000 ppm of diluted H₂S, at different operating temperatures (100–500 °C) for thick films of pure TiO₂ and TiO₂ doped with x wt.% of Al (x=1, 3, 5, 7, and 10wt.%). From the figure it is clear that the response values of every doped sample apparently increased with increasing the operating temperature. Also, it is observed that response for pure TiO₂ is obtained at high operating temperature 450 °C, which is rather high and practically inconvenient in view of commercial standards. Therefore, efforts were made to modify the TiO₂ based sensors by doping with Al, so as to be operated at lower operating temperatures with high sensitivity and selectivity. In this figure it is observed that the 3 wt. % Al doped TiO₂ thick film had the largest sensing response (99.23 %) in the range of the operating temperature studied, exhibiting a slightly marked maximum at 200 °C. The doping higher than 3 Wt. % resulted in the deteriorated H₂S sensing.

Fig. 4 shows histograms indicating the selectivity of 3 % Al-doped TiO₂ films for different gases. It is clear from the histogram that 3 Wt. % Al-doped TiO₂ sample is more selective to H₂S gas at 200 °C against all other tested gases viz: NH₃, LPG, CO₂, NO₂ and Ethanol. As easily seen, the doped TiO₂ was most selective to H₂S among the gases tested, while it was least selective to NO₂.

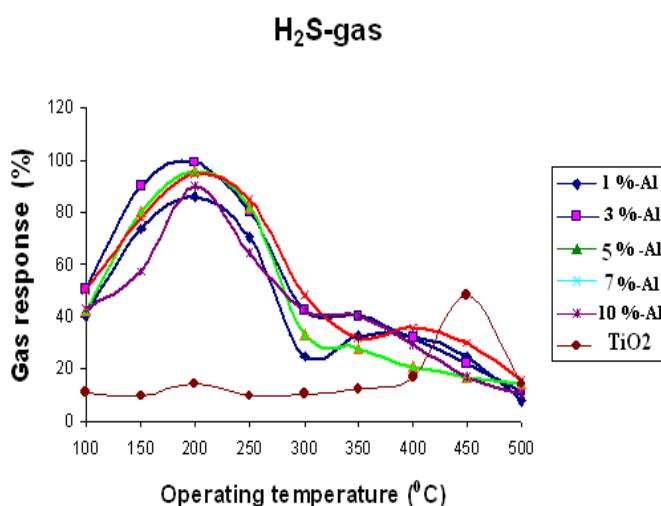


Fig. 3. Variation of H₂S Gas response with operating temperature for 1000 ppm.

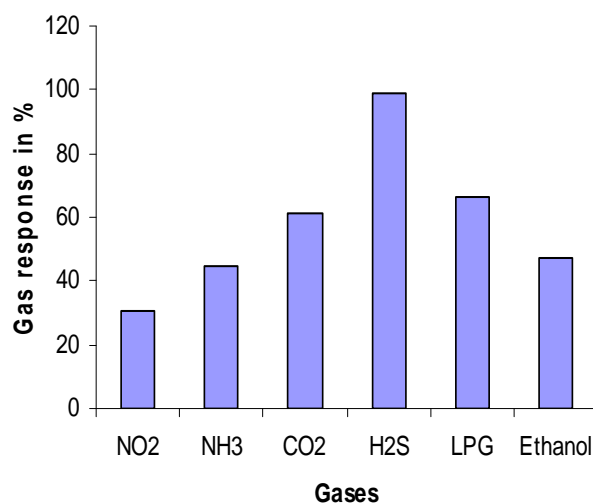


Fig. 4. Selectivity of 3 Wt. % Al-doped TiO₂ films for different Gases.

Fig. 5 depicts the variation of sensitivity of 3 % Al-doped TiO₂ film sample with H₂S gas concentrations at 200 °C temperature. It is clear from the figure that the gas response goes on increasing linearly with gas concentration up to 1000 ppm and saturated beyond it. The rate of increase in gas response was relatively larger up to 1000 ppm. The monolayer of the gas molecules formed on the surface could cover the whole surface of the film. The gas molecules from that layer would reach the surface active sites of the film. The excess gas molecules would remain idle and would not reach surface active sites of the sensor. So, the gas response at higher concentrations of the gas is not expected to increase further in large extent. Thus, the active region of the sensor would be up to 1000 ppm.

The response and recovery times of 3 % Al-doped TiO₂ films are represented in Fig. 6. The response was quick (~ 5 s) to 1000 ppm of H₂S while the recovery was fast (~ 28 s). The quick response may be due to faster oxidation of gas. Its high volatility explains its quick response and fast recovery to its initial chemical status.

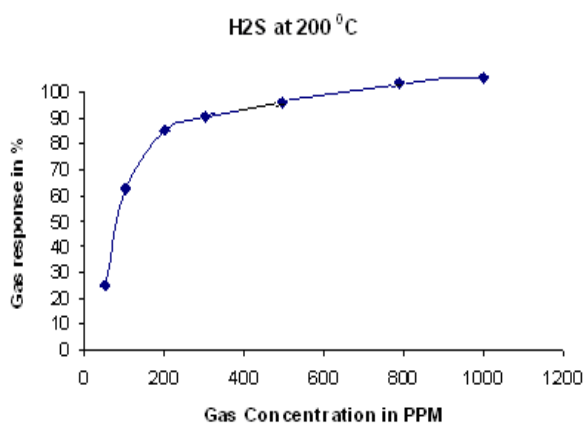


Fig. 5. Variation of gas response with gas concentration.

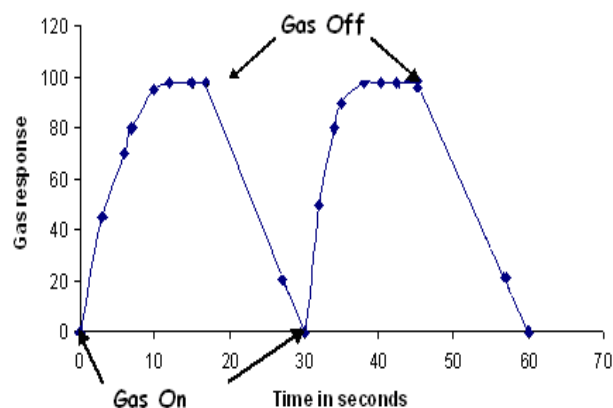


Fig. 6. Response and recovery of 3 Wt. % Al-doped TiO₂ film.

4. Discussion

It is known that atmospheric oxygen molecules are adsorbed on the surface of n-type semiconductor oxides in the forms of O⁻ and O²⁻ thereby decreasing the electronic conduction. Atmospheric oxygen molecules take electron from the conduction band of n-type TiO₂ to be adsorbed as O⁻_{TiO₂}. The reaction is as follows:



The TiO₂ material is oxygen deficient. The excess Ti ions (due to oxygen vacancies) act as electron donors [33]. When reducing gas molecules like H₂S reacts with negatively charged oxygen adsorbates, the trapped electrons are given back to conduction band of TiO₂. The energy released during decomposition of adsorbed H₂S molecules would be sufficient for electrons to jump up into conduction of TiO₂, causing an increase in the conductivity of the sensor. The possible reaction is:



For this reaction to proceed to the right hand side, some amount of activation energy has to be provided thermally. An increase in operating temperature surely increases the thermal energy so as to stimulate the oxidation H₂S (Eq. (3)). The reducing gas (H₂S) donates electrons to TiO₂. Therefore the resistance decreases, or the conductance increases. This is the reason why the gas response increases with operating temperature. The point at which the gas response reaches maximum is the actual thermal energy needed for the reaction to proceed. However, the response decreases at higher operating temperatures, as the oxygen adsorbates are desorbed from the surface of the sensor [34]. Also, at high temperatures the carrier concentration increases due to intrinsic thermal excitation and the Debye length decreases. This may be one of the reasons for the decreased gas response at high temperatures [35]. As the species are desorbed from the surface, oxygen is adsorbed again.

When the optimum amount of Al (3 Wt. %) is incorporated on the surface of TiO₂ film, the Al species would be distributed uniformly throughout the surface of the film. It has been reported that aluminum introduces electronic states at the surface or into the bulk that modifies the base material [36]. Conductivity of Al-doped TiO₂ is higher than pure TiO₂. Since ionic radii for Al and Ti are close to each other (0.74Å⁰ for Ti⁴⁺ and 0.67Å⁰ for Al³⁺). Al can occupy a regular cation position forming a substitutional solid solution. To maintain electrical neutrality such substitutions will create oxygen vacancies and donate electrons and the overall change in the resistance on exposure of H₂S gas leading

to high sensitivity. When the amount of Al is less than the optimum, the surface dispersion may be poor and the sensitivity of the film is observed to be decreased since the amount may not be sufficient to promote the reaction more effectively. On the other hand, as the amount of Al on the surface is more than the optimum, the Al atoms would be distributed more densely. As a result, overall change in the resistance on the exposure of gas would be smaller leading to lower response to the target gas.

Fig. 7 shows the adsorption of oxygen species on the surface of Al-doped TiO_2 oxide, abstracting electrons, and thus, causing an increase in potential barrier at the grain boundaries.

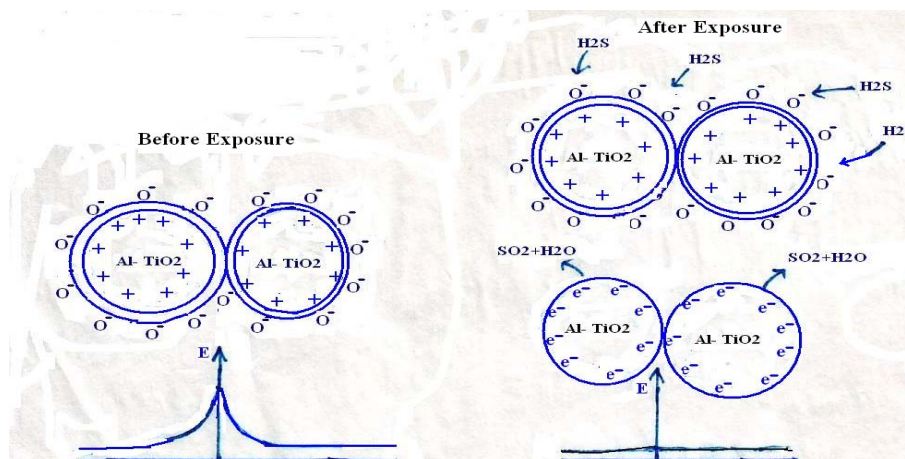


Fig. 7. Gas sensing mechanism of Al-doped TiO_2 Samples: Before and after exposure of H_2S gas.

The reaction occurring on the surface of the Al-doped TiO_2 is catalytic and is rate determining. In case of catalytic reaction, the H_2S gas is first adsorbed on a catalyst, gets split up into ions and then spills over on the surface and reacts with surface oxygen ions of functional material thereby decreasing the resistance of the sensor and enhancing the response. Higher response of Al doped as compared to undoped TiO_2 can be attributed to the distribution of the Al-species on the TiO_2 surface. When the film is doped, all the Al misfits are readily available on the surface and act as a very efficient catalyst for adsorption-desorption reactions of oxygen and H_2S gases.

5. Conclusions

From the results obtained, pure TiO_2 showed low response to H_2S gas. Al doped TiO_2 thick films were found to be sensitive for H_2S gas. Among all other additives 3 wt. % Al-doped TiO_2 thick film was found to be optimum and showed highest response to H_2S gas at 200°C . Response to H_2S gas increases with increase in operating temperature attains 99.23 % at 200°C and then decreases further with an increase the temperature. The gas response increases with the test gas concentration up to 1000 ppm. The 3 wt. % Al- doped TiO_2 thick films sensor has good selectivity to H_2S gas against LPG, NH_3 , Ethanol vapours, CO_2 and NO_2 at 200°C . Also this sensor showed very rapid response and recovery to H_2S gas. Over long exposure it was observed that sensor exhibited a good stability and repeatability as gas sensor with consistent pattern and response magnitude. These studies show that screen printed thick film of TiO_2 (3 wt. % Al- doped) on alumina substrate can be used as H_2S sensor.

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Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In addition, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

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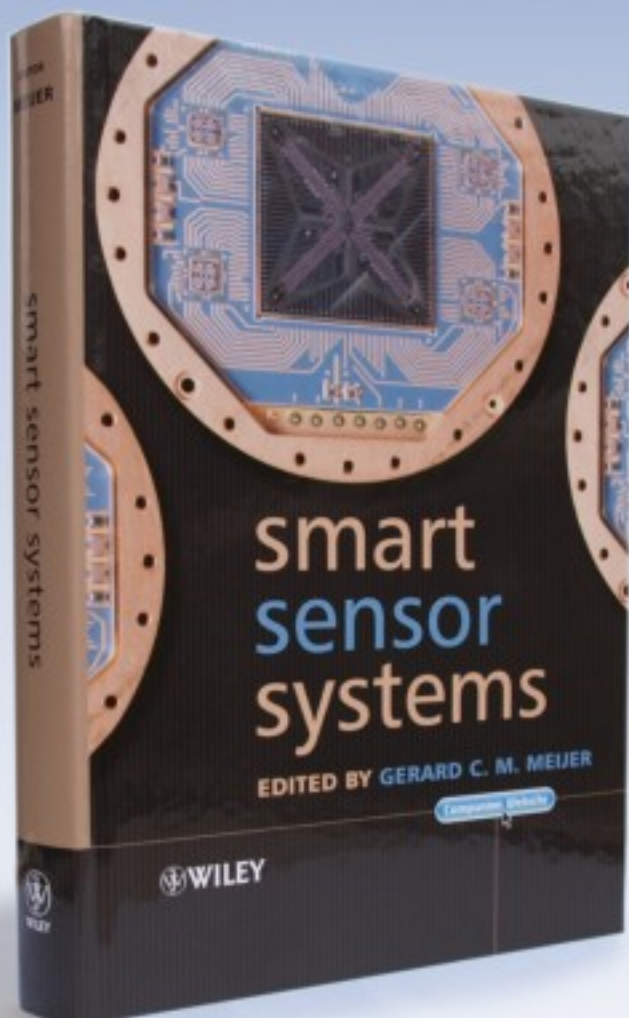
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