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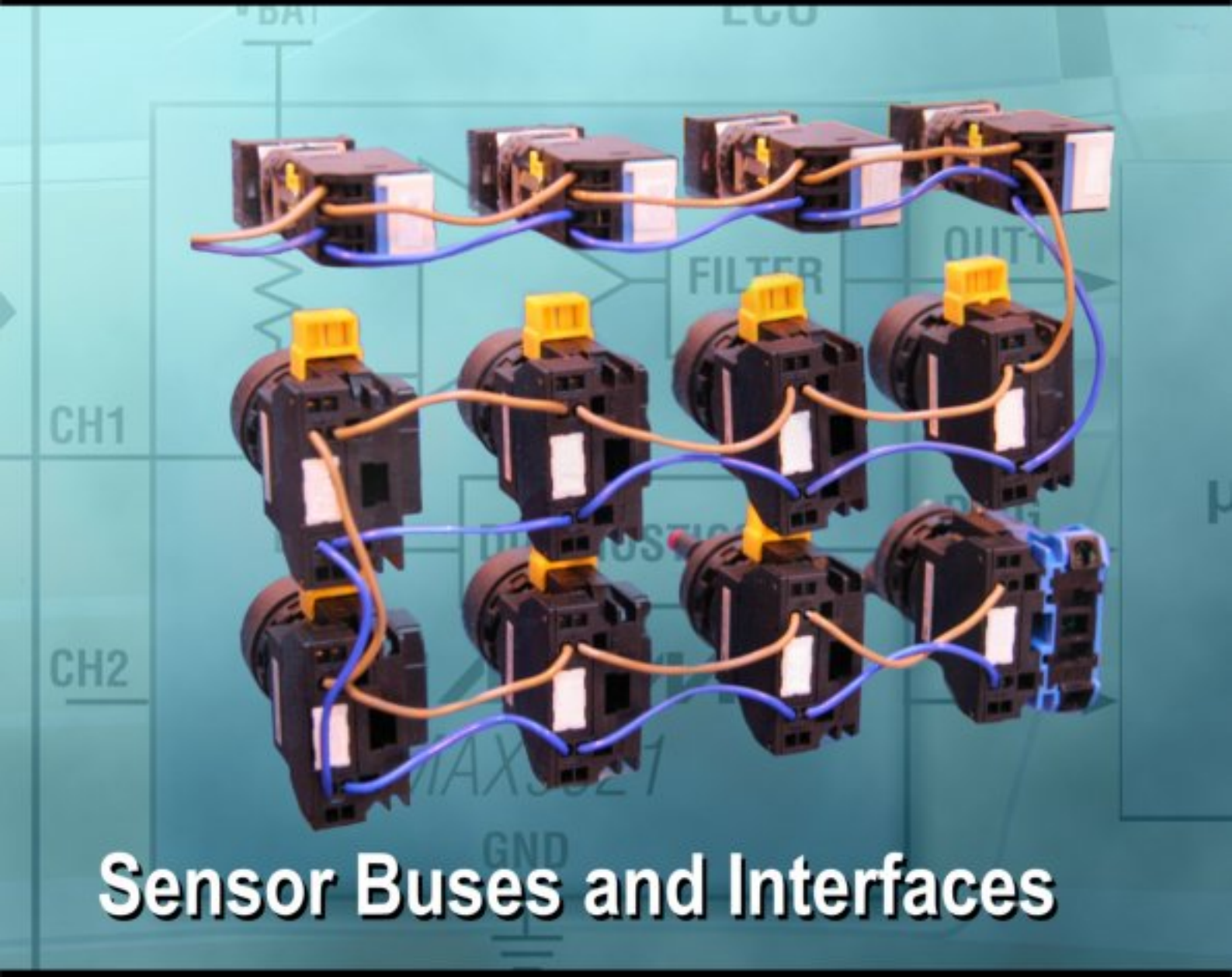

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Effect on Ethanol Gas Sensing Performance of Cu Addition to TiO_2 Thick Films

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Abstract: The preparation, characterization and gas sensing properties of Cu loaded thick films with TiO_2 semiconductor oxides have been investigated. These films of oxides were obtained by loading various concentrations (1 wt. %, 3 wt. %, 5 wt. %, 7 wt. % and 10 wt. %) of Cu in TiO_2 on alumina substrate by using screen printing technique. Pure TiO_2 was observed to be insensitive to ethanol gas. However, TiO_2 film loaded with 10 wt. % Cu was observed to be highly sensitive to ethanol gas. The sample was observed to be oxygen deficient. The Ethanol gas sensing studies were carried out on a static gas sensing system under normal laboratory conditions. D.C. Resistance of TiO_2 films were measured by half-bridge method in an atmosphere at different temperatures. The crucial gas response was found to ethanol gas at 300 ^{0}C and no cross response was observed to other hazardous and polluting gases. The effects of loading concentration on the gas sensitivity, selectivity, response and recovery of the sensor in the presence of ethanol gas were studied and discussed.

Keywords: TiO_2, Cu, Ehick films, Screen printing, Ethanol

1. Introduction

With the recent considerations of environmental pollution, there is need to develop a reliable and inexpensive semiconductor gas sensors. Metal oxide semiconductor gas sensors have been used extensively to detect toxic, pollutant hazardous and combustion gases [1, 2]. The semiconducting metal oxides such as TiO_2, SnO_2, ZnO, Fe_2O_3, and WO_3 etc. offer the potential for developing portable and inexpensive gas sensing devices, which have advantages of simplicity, high sensitivity and fast response. The working principle of these semiconductor gas sensors is based on change in conductivity

when exposed to the target gases [3]. Thick film technology (Screen printing technique) was introduced in the later part of 1950's to produce compact, robust and relatively inexpensive hybrid circuit for many purposes. Later on thick film technique has attracted by the sensor field [4]. Screen printing is viable and economical method to produce thick films of various materials [5-10]. Titanium dioxide (TiO_2) 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, optical coatings and gas sensing [11–15]. Titanium dioxide can be synthesized in three crystalline phases: rutile, brookite and anatase [16]. Rutile is more thermodynamically stable than other phases. Anatase phase is stable for TiO_2 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 TiO_2 sensors can be improved by addition of dopants such as Cr, Nb, Sn, Al, Pt, La, Cu and Y. Most important effects of dopant addition in TiO_2 are increasing the conductivity, slowing down anatase to rutile transformation and reducing grain growth. [17, 18]. 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 [19]. 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 TiO_2 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 [18].

Ethanol is a hypnotic (sleep producer) gas having toxic nature. Heavy exposure and/or consumption of alcoholic beverages, particularly by smokers, increase the risk of cancer of the upper respiratory and digestive tracks. Alcoholic cirrhosis leads to liver cancer. Amongst the women, the chances of breast cancer increase with alcoholic consumption or exposure. Those working on ethanol synthesis have great chances of being victims of respiratory and digestive track cancer. So there is a great demand and emerging challenges for monitoring ethanol gas at trace level. So by considering these points, the efforts are made to develop the thick films of TiO_2 with Cu as dopant on alumina substrate by a screen printing technique and to investigate their sensing properties for ethanol vapour. 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 TiO_2 based Gas Sensors

Analar Reagent (AR) grade TiO_2 powder (99.9 % pure) and Cu were mixed mechanochemically to obtain Cu-doped TiO_2 powders in various weight percentages such as 1, 3, 5, 7, and 10 wt. %. This prepared powder was then calcined at 400^0C 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 (TiO_2), doping material (Cu) 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). TiO_2 powder with Cu 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. TiO_2 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 ^{o}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 TiO_2 films were investigated using X-ray diffraction analysis from 20-80^0 [Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having CuKα, λ=0.1542 nm radiation] with 0.1^o/step (2θ) at the rate of 2 sec/step. The scanning electron microscopy (SEM- JEOL JED-2300) was employed to characterize the surface morphology. The Composition of TiO_2 thick film samples were analyzed by Energy Dispersive spectrometer (JEOL-JED 6360 LA). The thickness of the TiO_2 thick films was measured by using Taylor-Hobson (Taly-step UK) system. The thickness of the films was observed uniform in the range of 18 μm to 25μm. 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 Ethanol response of thick films was studied in test assembly. The electrical resistances of thick film in air (Ra) and in the presence of Ethanol gas (Rg) were measured to evaluate the gas response (S) given by the relation,

$$S\ (\%) = \frac{Ra - Rg}{Ra} \times 100\ \% \qquad (1)$$

where Ra is the resistance of the thick film sample in air and Rg 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 [20].

3. Results

3.1. Elemental Analysis

The elemental compositions of the pure and doped TiO_2 films were analyzed using an energy dispersive spectrometer. The mass% of Ti and O in each sample was not as per stoichiometric proportion. All the samples were observed to be oxygen deficient. Excess or deficiency of the constituent material particles leads to the semiconducting behavior of the material [21]. The film doped with 10 Wt. % Cu was observed to be most oxygen deficient, as evidenced from EDS analysis (Table 1).

3.2. Structural Analysis (X-ray Diffraction Studies)

Fig. 1 depicts XRD pattern (20 - 80^0) of the TiO_2 thick films loaded with x wt.% of Cu (x = 0,1,3,5,7 and 10 wt.%) prepared by a screen printing method and fired at 800 ^{0}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-Cu composite film samples fired at 800 ^{0}C [22]. In the 2θ range, the 101 anatase peak, located at 25.8^O, is present. This is the most pronounced peak of an anatase structure for film sample fired at 800 ^{0}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 anatase phase decreases

and rutile phases increases as Cu concentration increases. Being even all samples was 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 [15-17, 23, 24].The peaks of the XRD pattern correspond to TiO_2 and Cu observed to be identical and polycrystalline in nature. As seen from XRD of Cu-doped TiO_2 , small peak of Cu was found at $2\theta = 35.54^0$ and no peak was observed for lower doping concentrations, this may be due to Cu^{2+} substituted in Ti^{4+} G.N. Chaudhari et. al also reported similar observation for Al_2O_3 doped TiO_2 [25].

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

Element (Wt. %)	TiO2	Cu-doped TiO2				
		1 wt.	3 wt.	5wt.	7wt.	10wt.
O	42.37	41.73	40.39	42.47	42.46	40.30
Ti	57.63	58.03	58.45	56.12	54.58	55.44
Cu	--	0.24	1.16	1.41	2.97	4.26

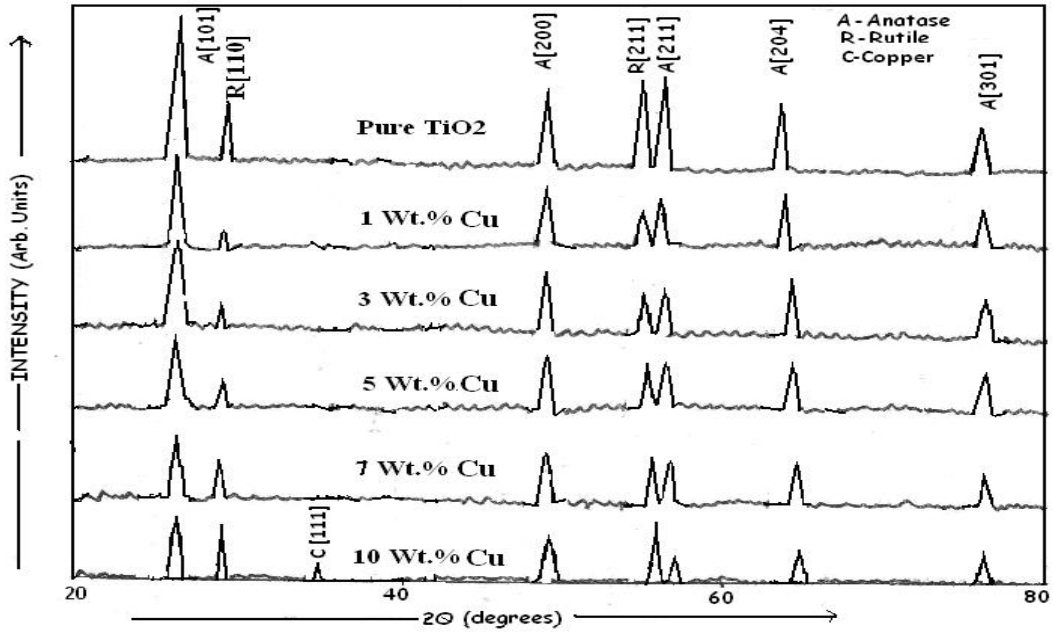
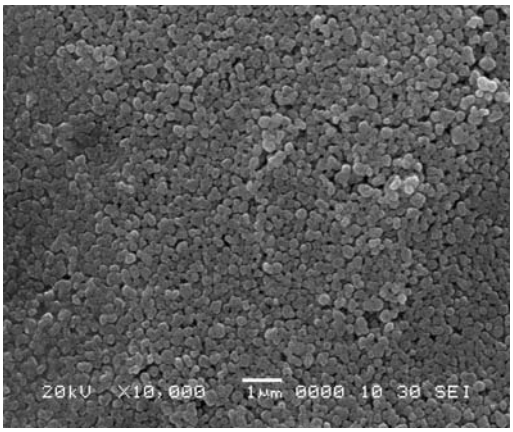


Fig. 1. XRD pattern of pure and Cu-doped TiO_2 films.

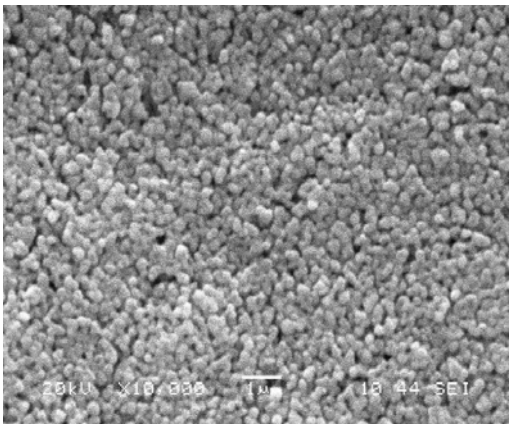
3.3. Microstructural Analysis

Fig. 2 depicts the SEM images of pure and Cu-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-f) depict the microstructure of Cu-doped TiO_2 thick films. It is clear from these figures that the doping of Cu affected the microstructure of TiO_2. The film consists of voids and grains

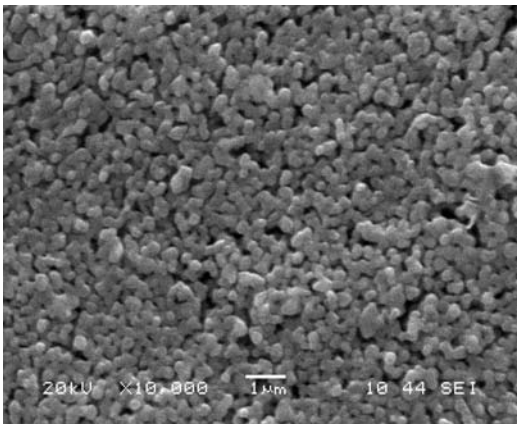
distributed nonuniformly. The increased porosity increases the in-pore adsorption of oxygen and tends to improve the adsorption-desorption mechanism of target gas. The inclusion of Cu in samples promoted the phase transformation yielding an increase of the grain size. In this case, it has been published that transition metals can diffuse into the titania lattice increasing the defect concentration and breaking bonds that yields atomic rearrangements that promote the phase transition [26-27].



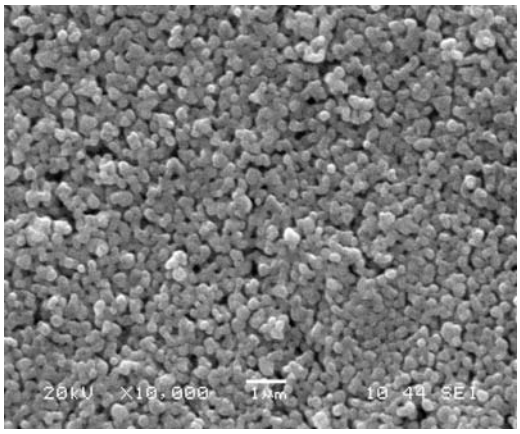
(a) SEM image of pure TiO_2;



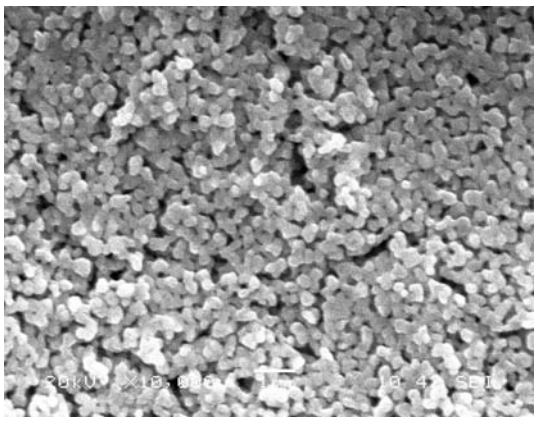
(b) SEM image of 1 Wt. % Cu-doped TiO_2;



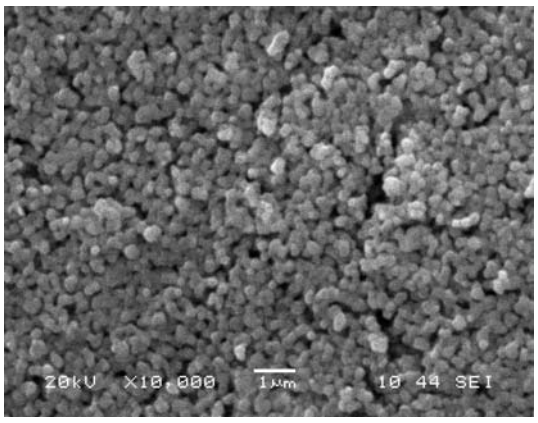
(c) SEM image of 3 Wt. %Cu-doped TiO_2;



(d) SEM image of 5 Wt. % Cu-doped TiO_2;



(e) SEM image of 7 Wt. % Cu-doped TiO_2;



(f) SEM image of 10 Wt. % Cu-doped TiO_2;

Fig. 2. Microstructure of pure TiO_2 thick film.

3.4. Gas Sensing Characteristics

Electrical characterization of Cu-doped TiO_2 composite thick films fabricated by screen printing method and fired at 800 ^oC 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 Ethanol (1000 ppm) diluted in air. Fig. 3 shows gas sensing response to 1000 ppm of Ethanol, at different operating temperatures (100–550 ^oC) for thick films of pure TiO_2 and TiO_2 doped with x wt.% of Cu (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 TiO_2 is obtained at high operating temperature 450 ^0C, which is rather high and practically inconvenient in view of commercial standards. Therefore, efforts were made to modify the TiO_2 based sensors by doping with Cu, so as to be operated at lower operating temperatures with high sensitivity and selectivity. In this figure it is observed that the 10 wt. % Cu doped TiO_2 thick film had the largest sensing response (84.16 %) in the range of the operating temperature studied, exhibiting a slightly marked maximum at 300 ^oC. The doping higher than 10 Wt. % resulted in the deteriorated Ethanol sensing.

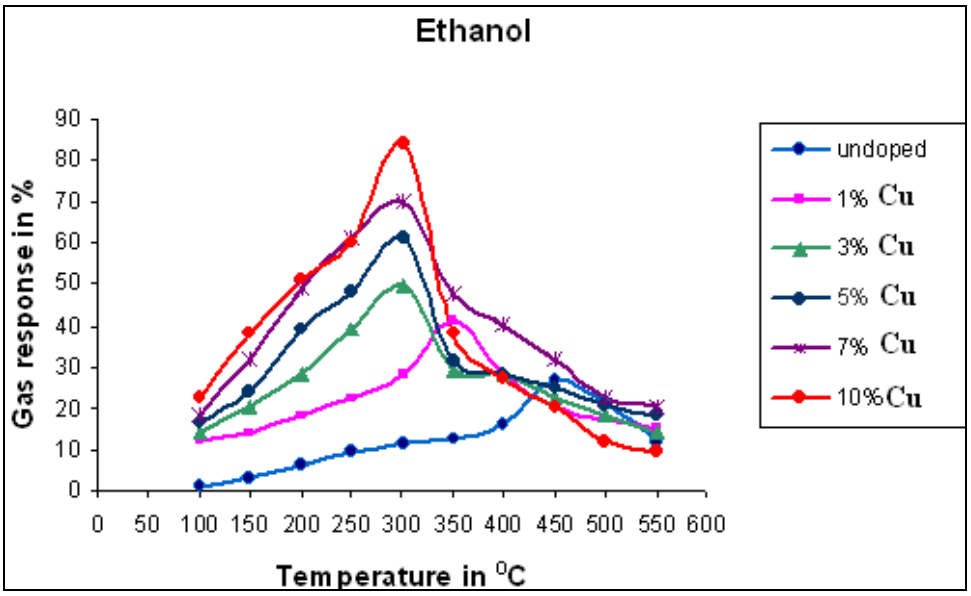


Fig. 3. Variation of Ethanol Gas response with operating temperature for 1000 ppm.

Fig. 4 shows histograms indicating the selectivity of 10 % Cu-doped TiO_2 films for different gases. It is clear from the histogram that 10 Wt. % Cu-doped TiO_2 sample is most selective to Ethanol at 300 ^0C against all other tested gases viz: NH3, LPG, CO2, NO2 and H2S. As easily seen, the doped TiO_2 was most selective to Ethanol among the gases tested, while it was least selective to H2S.

Fig. 5 depicts the variation of sensitivity of 10 % Cu-doped TiO_2 film sample with Ethanol concentrations at 300^oC 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.

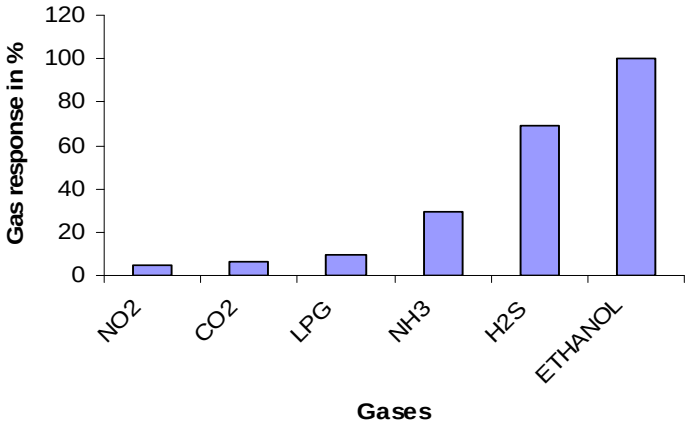


Fig. 4. Selectivity of 10 Wt. % Cu-doped TiO_2 films for temperature for 1000 ppm.

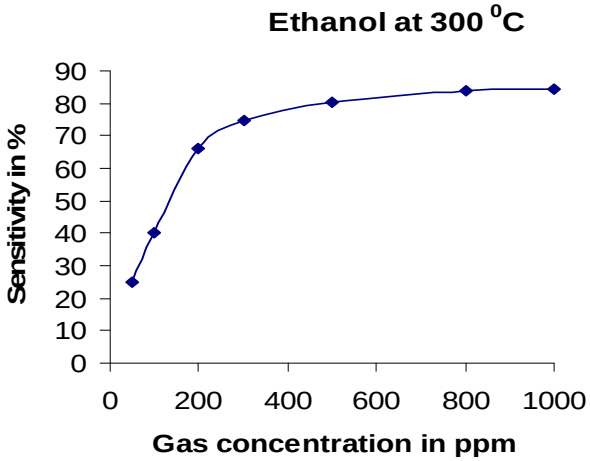


Fig. 5. Variation of gas response with gas concentration.

The response and recovery times of 10 % Cu-doped TiO_2 films are represented in Fig. 6. The response was quick (~ 18 s) to 1000 ppm of Ethanol while the recovery was fast (~ 25 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.

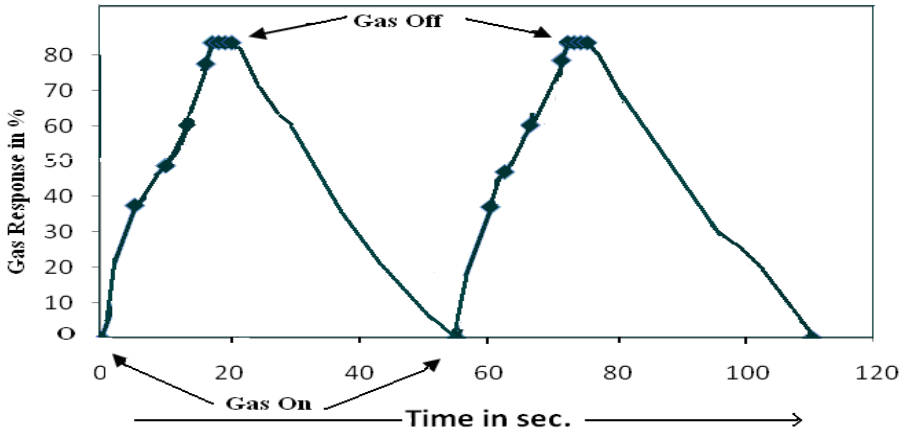


Fig. 6. Response and recovery of 10 Wt. % Cu-doped TiO_2 film.

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 O^{2-} thereby decreasing the electronic conduction. Atmospheric oxygen molecules take electron from the conduction band of n-type TiO_2 to be adsorbed as $O^-_{TiO2.}$ The reaction is as follows:

$$O_2\{g\} + 2e^- \rightarrow 2O^-_{TiO2} \quad (2)$$

The TiO_2 material is oxygen deficient. The excess Ti ions (due to oxygen vacancies) act as electron donors [28]. When reducing gas molecules like Ethanol reacts with negatively charged oxygen adsorbates, the trapped electrons are given back to conduction band of TiO_2. The energy released during decomposition of adsorbed Ethanol molecules would be sufficient for electrons to jump up into conduction of TiO_2, causing on increase in the conductivity of the sensor. When ethanol reacts with oxygen, a complex series of reactions take place, ultimately converting the ethanol to carbon dioxide and water. The possible reaction is:

$$C_2H_5OH_{(gas)} + 6O^{2-}_{(film\ surface)} \rightarrow 2CO_{2\ (gas)} + 3H_2O_{(gas)} + 12\ e^-_{(cond.band)} \quad (3)$$

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 of Ethanol (Eq. (3)). The reducing gas (Ethanol) donates electrons toTiO_2. 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 [29]. 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 [30]. As the species are desorbed from the surface, oxygen is adsorbed again.

When the optimum amount of Cu (10 Wt. %) is incorporated on the surface of TiO_2 film, the Cu species would be distributed uniformly throughout the surface of the film. It has been reported that transition metals introduces electronic states at the surface or into the bulk that modifies the base material [31]. Conductivity of Cu-doped TiO_2 is higher than pure TiO_2. Since ionic radii for Cu and Ti are close to each other ($0.68A^0$ for Ti^{4+} and $0.70A^0$ for Cu^{2+}). Cu 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 Ethanol gas leading to high sensitivity. When the amount of Cu 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 Cu on the surface is more than the optimum, the Cu 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 Cu-doped TiO_2 oxide, abstracting electrons, and thus, causing an increase in potential barrier at the grain boundaries.

The reaction occurring on the surface of the Cu-doped TiO_2 is catalytic and is rate determining. In case of catalytic reaction, the Ethanol 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 Cu doped as compared to undoped TiO_2 can be attributed to the distribution of the Cu-species on the TiO_2 surface. When the

film is doped, all the Cu misfits are readily available on the surface and act as a very efficient catalyst for adsorption-desorption reactions of oxygen and Ethanol gases.

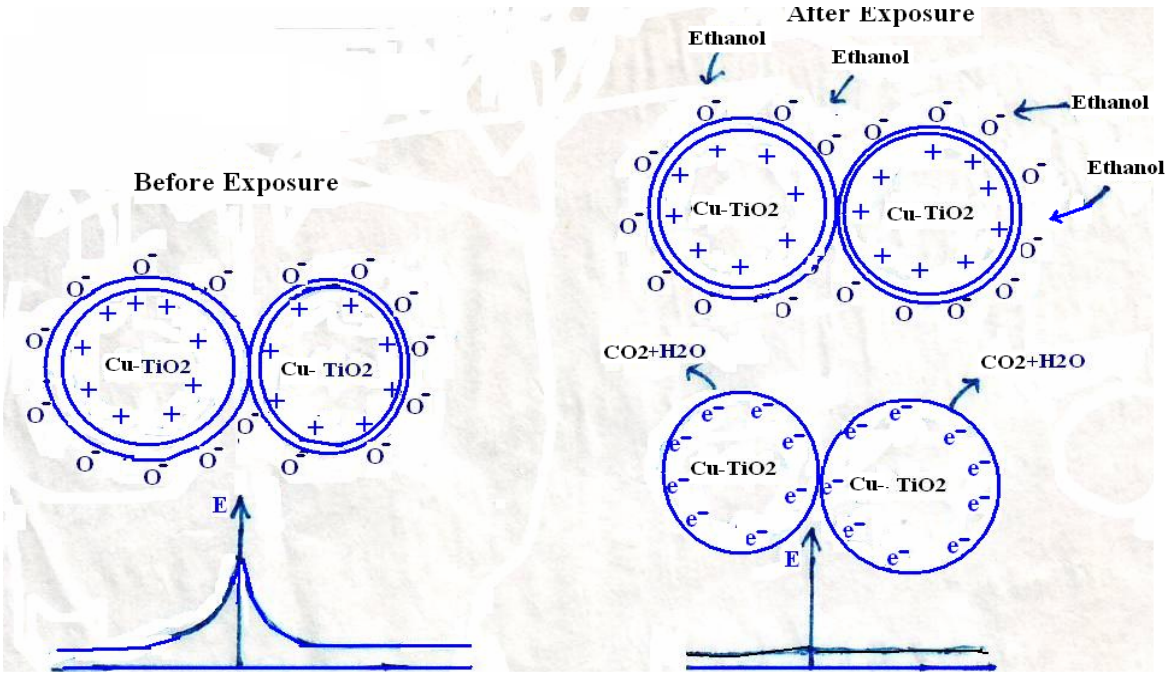


Fig. 7. Gas sensing mechanism of Cu-doped TiO_2 Samples: Before and after exposure of Ethanol gas.

5. Conclusions

From the results obtained, pure TiO_2 showed low response to Ethanol. Cu doped TiO_2 thick films were found to be selective for Ethanol. Among all other additives 10 wt. % Cu-doped TiO_2 thick film was found to be optimum and showed highest response to Ethanol at 300 ^{0}C. Response to Ethanol gas increases with increase in operating temperature attains 84.16% at 300 ^{0}C and then decreases further with an increase the temperature. The gas response increases with the test gas concentration up to 1000 ppm. The 10 wt. % Cu- doped TiO_2 thick films sensor has good selectivity to Ethanol against LPG, NH_3, CO_2, H_2S and NO_2 at 300 ^{0}C. Also this sensor showed very rapid response and recovery to Ethanol. 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 (10 wt. % Cu- doped) on alumina substrate can be used as Ethanol sensor.

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Guide for Contributors

Aims and Scope

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 additional, 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.

Topics Covered

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:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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Articles should be written in English. Authors are invited to submit by e-mail editor@sensorsportal.com 8-14 pages article (including abstract, illustrations (color or grayscale), photos and references) in both: MS Word (doc) and Acrobat (pdf) formats. Detailed preparation instructions, paper example and template of manuscript are available from the journal's webpage: http://www.sensorsportal.com/HTML/DIGEST/Submition.htm Authors must follow the instructions strictly when submitting their manuscripts.

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