

ISSN 1726-5479

# SENSORS & TRANSDUCERS

vol. 116  
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## Sensor Buses and Interfaces

International Frequency Sensor Association Publishing



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Volume 116  
Issue 5  
May 2010

www.sensorsportal.com

ISSN 1726-5479

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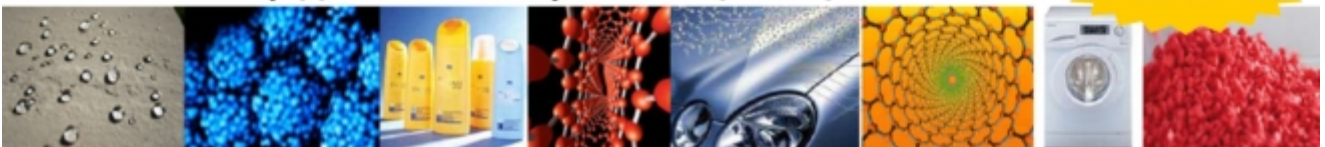
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## Effect on Ethanol Gas Sensing Performance of Cu Addition to TiO<sub>2</sub> Thick Films

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*Received: 8 March 2010 /Accepted: 24 May 2010 /Published: 31 May 2010*

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**Abstract:** The preparation, characterization and gas sensing properties of Cu loaded thick films with TiO<sub>2</sub> 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<sub>2</sub> on alumina substrate by using screen printing technique. Pure TiO<sub>2</sub> was observed to be insensitive to ethanol gas. However, TiO<sub>2</sub> 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<sub>2</sub> films were measured by half-bridge method in an atmosphere at different temperatures. The crucial gas response was found to ethanol gas at 300 °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.  
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**Keywords:** TiO<sub>2</sub>, Cu, Thick films, Screen printing, Ethanol

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### 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<sub>2</sub>, SnO<sub>2</sub>, ZnO, Fe<sub>2</sub>O<sub>3</sub>, and WO<sub>3</sub> 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 ( $\text{TiO}_2$ ) 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  $\text{TiO}_2$  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  $\text{TiO}_2$  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  $\text{TiO}_2$  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  $\text{TiO}_2$  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  $\text{TiO}_2$  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 $\text{TiO}_2$ based Gas Sensors**

Analar Reagent (AR) grade  $\text{TiO}_2$  powder (99.9 % pure) and Cu were mixed mechanochemically to obtain Cu-doped  $\text{TiO}_2$  powders in various weight percentages such as 1, 3, 5, 7, and 10 wt. %. This prepared powder was then calcined at  $400^\circ\text{C}$  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 ( $\text{TiO}_2$ ), doping material (Cu) and glass frit (70 wt.%  $\text{PbO}$ , 18 wt.%  $\text{Al}_2\text{O}_3$ , 9wt.%  $\text{SiO}_2$  and 3wt.%  $\text{B}_2\text{O}_3$ ) as permanent binder. The organic part consists of 8 % ethyl cellulose (EC) and 92% butyl carbitol acetate (BCA).  $\text{TiO}_2$  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.  $\text{TiO}_2$  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  $800^\circ\text{C}$  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<sub>2</sub> films were investigated using X-ray diffraction analysis from 20-80° [Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having CuK $\alpha$ ,  $\lambda=0.1542$  nm radiation] with 0.1°/step ( $2\theta$ ) 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<sub>2</sub> thick film samples were analyzed by Energy Dispersive spectrometer (JEOL-JED 6360 LA). The thickness of the TiO<sub>2</sub> 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  $\mu\text{m}$  to 25 $\mu\text{m}$ . 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 ( $R_a$ ) and in the presence of Ethanol 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 [20].

## **3. Results**

### **3.1. Elemental Analysis**

The elemental compositions of the pure and doped TiO<sub>2</sub> 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).

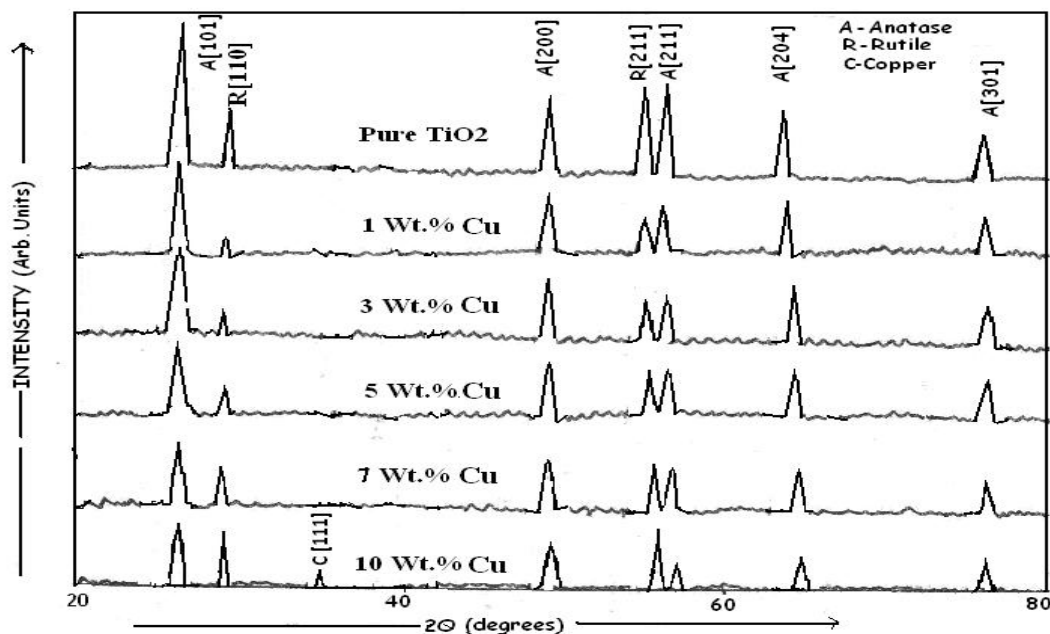
### **3.2. Structural Analysis (X-ray Diffraction Studies)**

Fig. 1 depicts XRD pattern (20 - 80°) of the TiO<sub>2</sub> 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 °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<sub>2</sub>.Cu composite film samples fired at 800 °C [22]. In the  $2\theta$  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 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<sub>2</sub> and Cu observed to be identical and polycrystalline in nature. As seen from XRD of Cu-doped TiO<sub>2</sub>, small peak of Cu was found at  $2\theta = 35.54^\circ$  and no peak was observed for lower doping concentrations, this may be due to Cu<sup>2+</sup> substituted in Ti<sup>4+</sup>. G.N. Chaudhari et. al also reported similar observation for Al<sub>2</sub>O<sub>3</sub> doped TiO<sub>2</sub> [25].

**Table 1.** Composition of Cu-doped TiO<sub>2</sub> obtained from EDS.

Element (Wt. %)	TiO <sub>2</sub>	Cu-doped TiO <sub>2</sub>				
		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

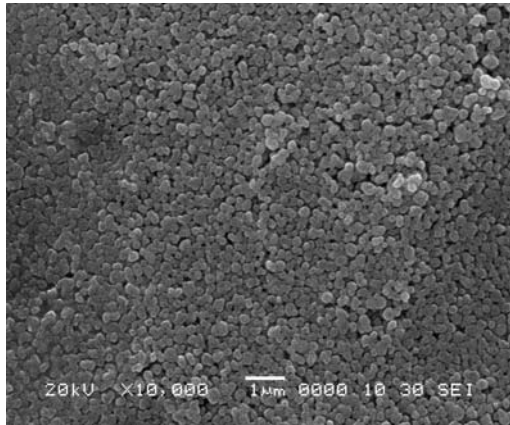


**Fig. 1.** XRD pattern of pure and Cu-doped TiO<sub>2</sub> films.

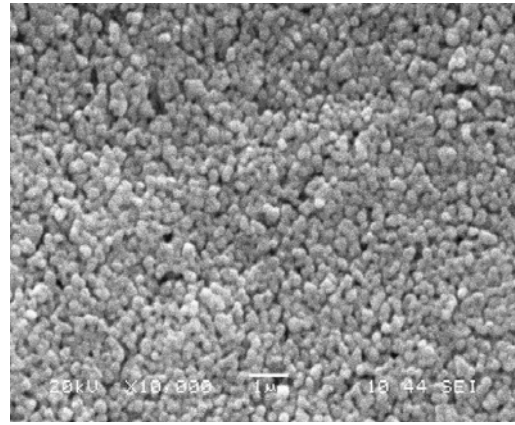
### 3.3. Microstructural Analysis

Fig. 2 depicts the SEM images of pure and Cu-doped TiO<sub>2</sub> films. Fig. 2 (a) depicts the microstructure of pure TiO<sub>2</sub> 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<sub>2</sub> thick films. It is clear from these figures that the doping of Cu affected the microstructure of TiO<sub>2</sub>. The film consists of voids and grains

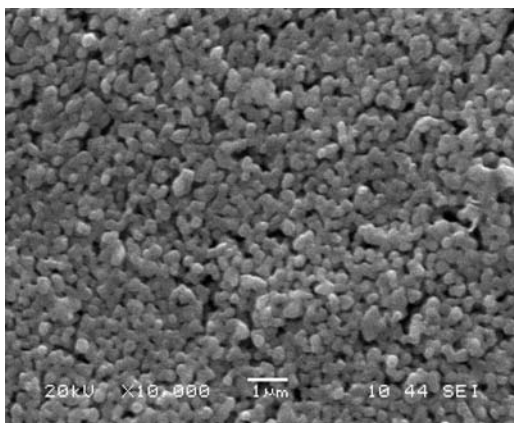
distributed nonuniformly. The increased porosity increases the in-pore adsorption of oxygen and tends to improve the adsorption-desorption mechanism of target gas. The inclusion of Cu in samples promoted the phase transformation yielding an increase of the grain size. In this case, it has been published 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].



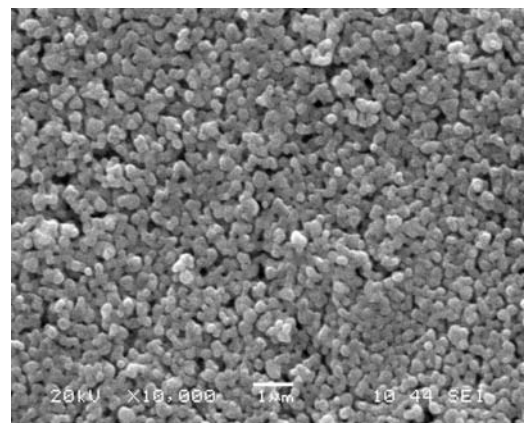
(a) SEM image of pure TiO<sub>2</sub>;



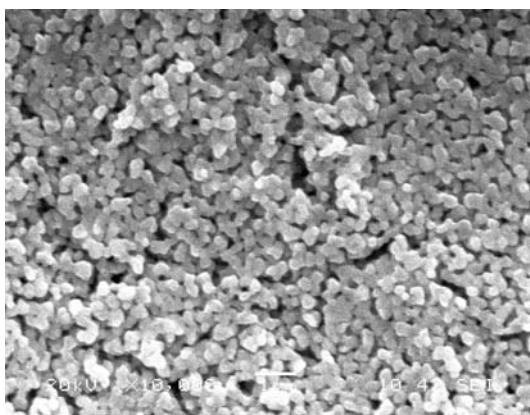
(b) SEM image of 1 Wt. % Cu-doped TiO<sub>2</sub>;



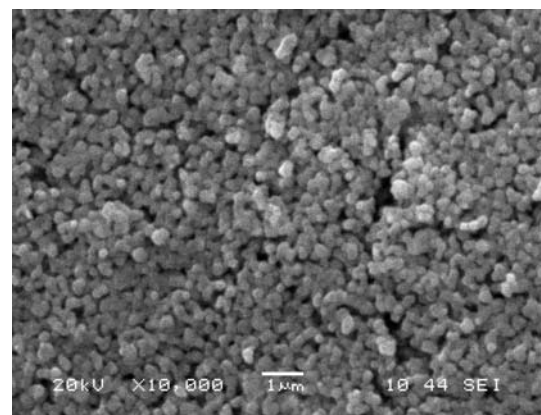
(c) SEM image of 3 Wt. % Cu-doped TiO<sub>2</sub>;



(d) SEM image of 5 Wt. % Cu-doped TiO<sub>2</sub>;



(e) SEM image of 7 Wt. % Cu-doped TiO<sub>2</sub>;



(f) SEM image of 10 Wt. % Cu-doped TiO<sub>2</sub>;

**Fig. 2.** Microstructure of pure TiO<sub>2</sub> thick film.

### 3.4. Gas Sensing Characteristics

Electrical characterization of Cu-doped TiO<sub>2</sub> 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 Ethanol (1000 ppm) diluted in air. Fig. 3 shows gas sensing response to 1000 ppm of Ethanol, at different operating temperatures (100–550 °C) for thick films of pure TiO<sub>2</sub> and TiO<sub>2</sub> 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<sub>2</sub> 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 TiO<sub>2</sub> 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<sub>2</sub> thick film had the largest sensing response (84.16 %) in the range of the operating temperature studied, exhibiting a slightly marked maximum at 300 °C. The doping higher than 10 Wt. % resulted in the deteriorated Ethanol sensing.

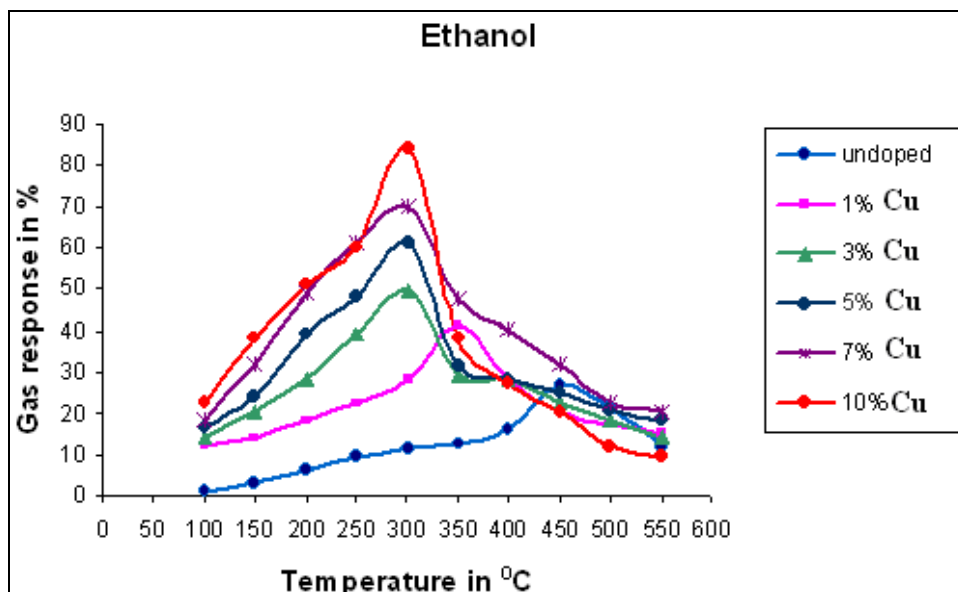


Fig. 3. Variation of Ethanol Gas response with operating temperature for 1000 ppm.

Fig. 4 shows histograms indicating the selectivity of 10 % Cu-doped TiO<sub>2</sub> films for different gases. It is clear from the histogram that 10 Wt. % Cu-doped TiO<sub>2</sub> sample is most selective to Ethanol at 300 °C against all other tested gases viz: NH<sub>3</sub>, LPG, CO<sub>2</sub>, NO<sub>2</sub> and H<sub>2</sub>S. As easily seen, the doped TiO<sub>2</sub> was most selective to Ethanol among the gases tested, while it was least selective to H<sub>2</sub>S.

Fig. 5 depicts the variation of sensitivity of 10 % Cu-doped TiO<sub>2</sub> film sample with Ethanol concentrations at 300°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.

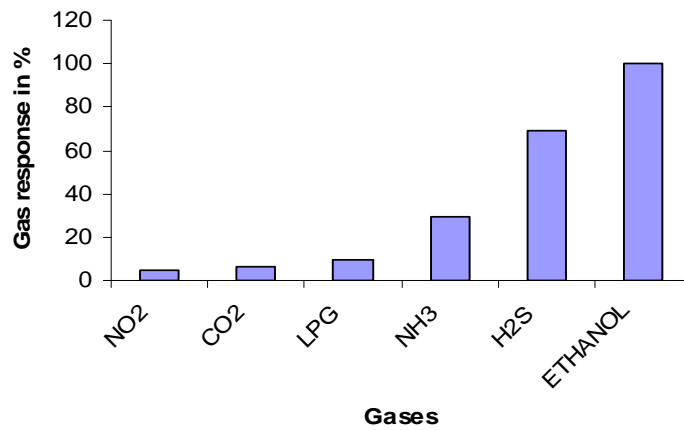


Fig. 4. Selectivity of 10 Wt. % Cu-doped TiO<sub>2</sub> films for temperature for 1000 ppm.

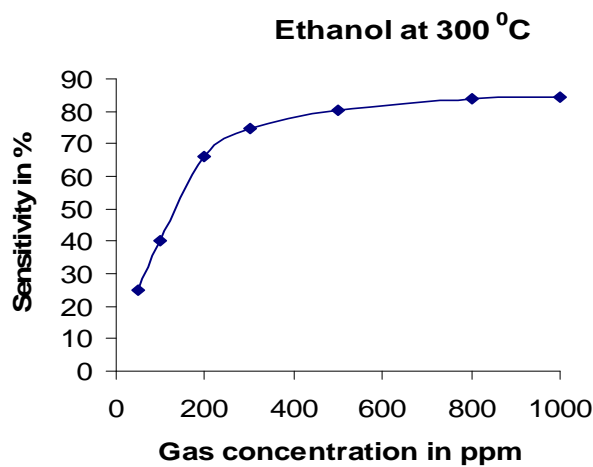


Fig. 5. Variation of gas response with gas concentration.

The response and recovery times of 10 % Cu-doped TiO<sub>2</sub> 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.

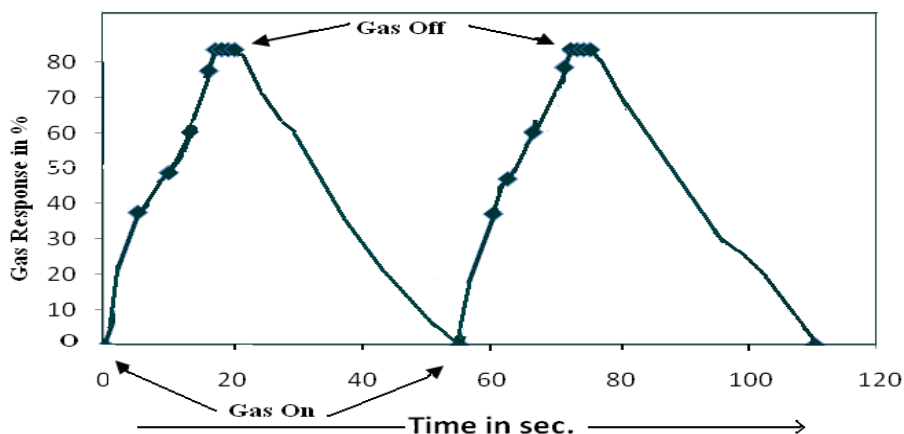


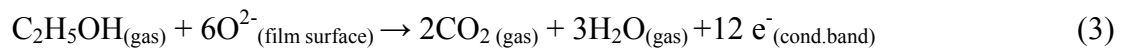
Fig. 6. Response and recovery of 10 Wt. % Cu-doped TiO<sub>2</sub> film.

## 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  $O^-_{TiO_2}$ . The reaction is as follows:



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 an 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:



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 to  $TiO_2$ . 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 introduce 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 ( $0.68\text{\AA}$  for  $Ti^{4+}$  and  $0.70\text{\AA}$  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.

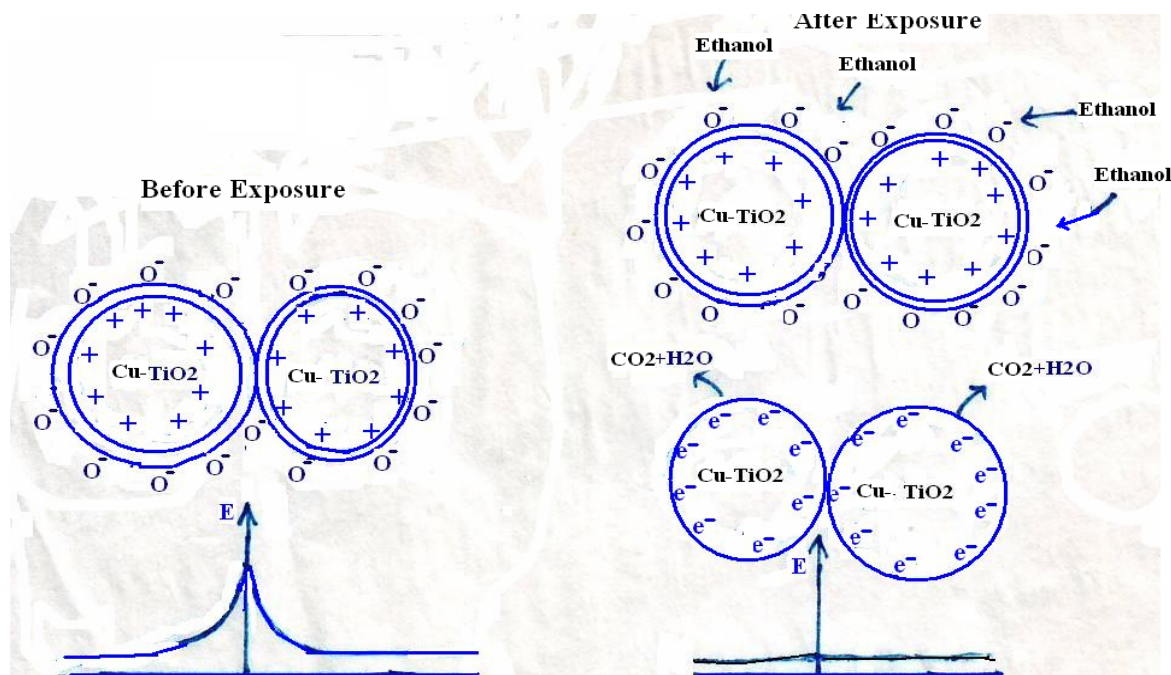


Fig. 7. Gas sensing mechanism of Cu-doped TiO<sub>2</sub> Samples: Before and after exposure of Ethanol gas.

## 5. Conclusions

From the results obtained, pure TiO<sub>2</sub> showed low response to Ethanol. Cu doped TiO<sub>2</sub> thick films were found to be selective for Ethanol. Among all other additives 10 wt. % Cu-doped TiO<sub>2</sub> thick film was found to be optimum and showed highest response to Ethanol at 300 °C. Response to Ethanol gas increases with increase in operating temperature attains 84.16% at 300 °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<sub>2</sub> thick films sensor has good selectivity to Ethanol against LPG, NH<sub>3</sub>, CO<sub>2</sub>, H<sub>2</sub>S and NO<sub>2</sub> at 300 °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<sub>2</sub> (10 wt. % Cu- doped) on alumina substrate can be used as Ethanol sensor.

## Acknowledgment

The authors are thankful to management authorities of M.G. Vidyamandir Malegaon camp Dist:-Nasik, The Principal, M.S.G. College, Malegaon and The Principal, L.V.H. College, Nashik for providing all the required infrastructural facilities for doing this work.

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## Guide for Contributors

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### Aims and Scope

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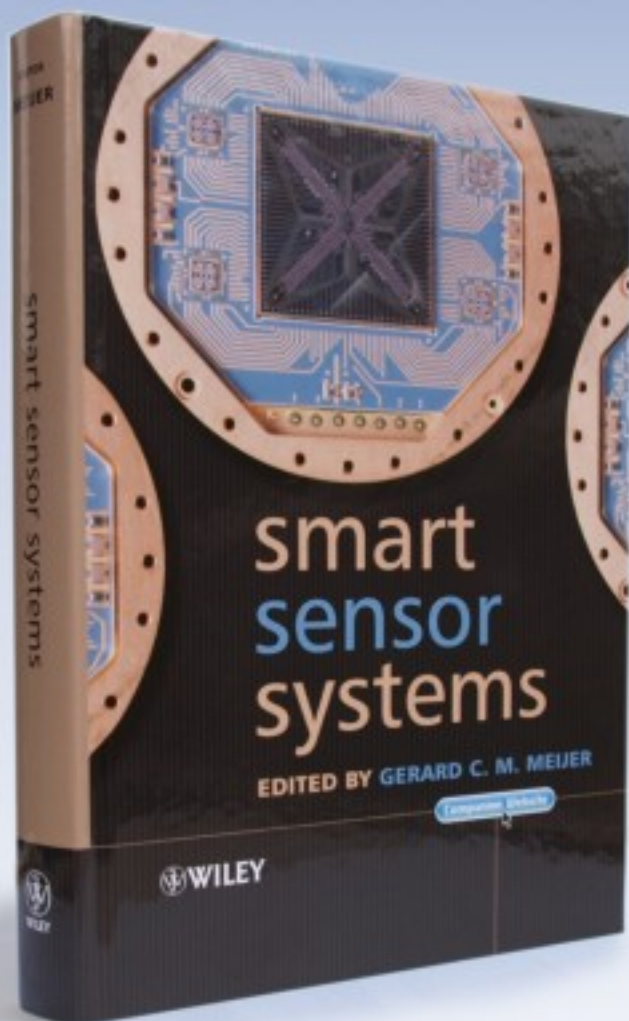
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