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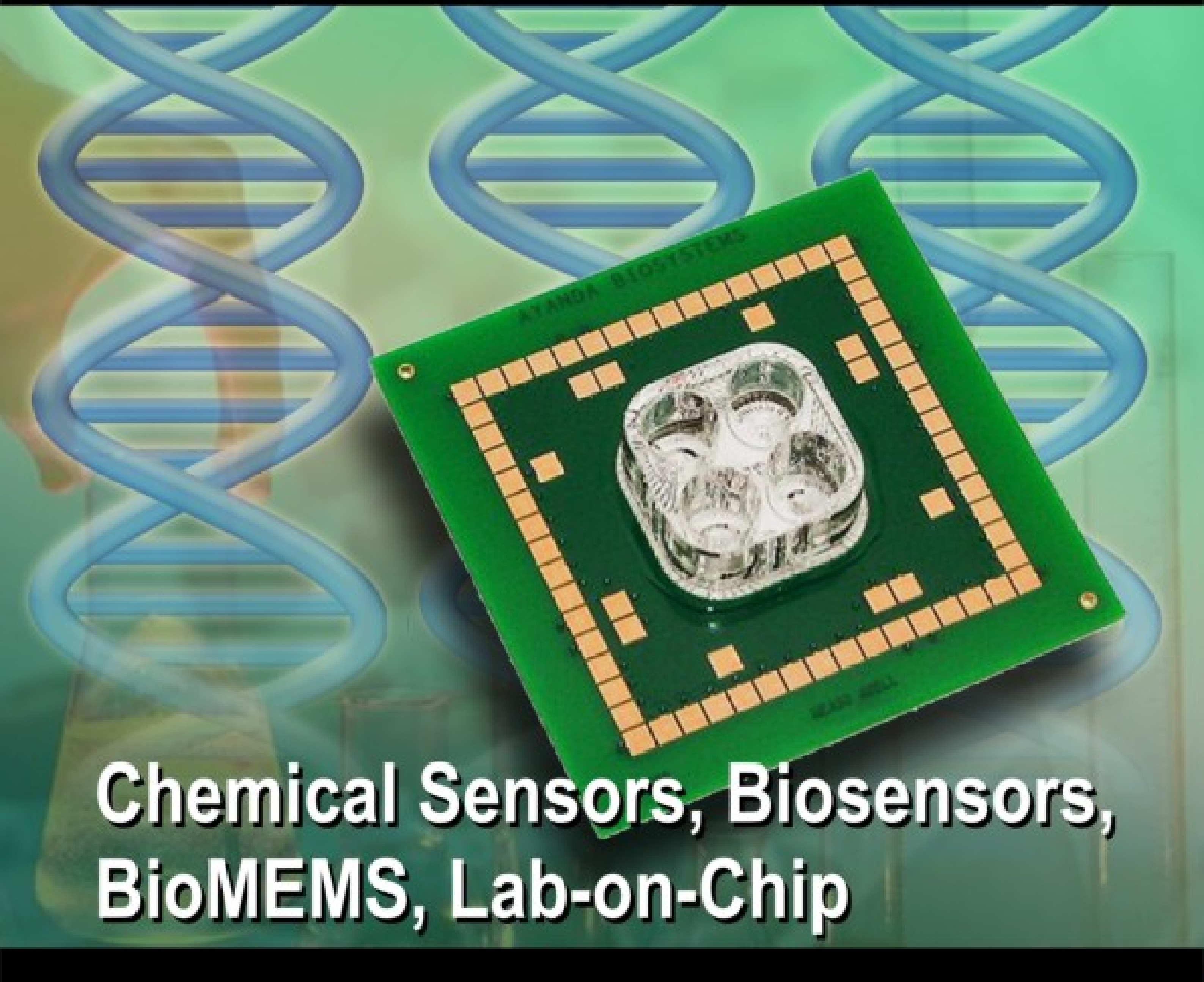

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Study on Gas Sensing Performance of TiO_2 Screen Printed Thick Films

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Abstract: Titanium dioxide (TiO_2) thick films were prepared on alumina substrate by using screen printing technique. After preparation, the films were fired at temperature range 600 -1000 ºC for two hour. Morphological, compositional and structural properties of the film samples were performed by means of several techniques, including scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), X-ray diffraction techniques. We explore the various gases to study the sensing performance of the TiO_2 thick films. The maximum response was reported to film fired at 800 ^{0}C for LPG gas at 350 ^{0}C operating temperature. *Copyright © 2009 IFSA.*

Keywords: Thick films, Alumina substrate, Gas sensitivity, SEM, XRD

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1. Introduction

Titanium dioxide (TiO_2) is extensively studied because of its broad range of applications in different fields. The increased interest in both the application and the fundamental research of this material in the last decade due to its remarkable optical and electrical properties. It has high dielectric constant and high refractive index hence it is widely used in optical coating, beam splitters and antireflection coating. It is reported on its use in gas sensor, humidity and temperature sensor [1]. It is widely used in confectionery, cosmetics and foods in plastic industry [2]. It is investigated as key material for applications in photovoltaic cells, batteries, chemical sensing [3]. TiO_2 Thin and thick films were made by sputtering and screen printing technique for NO_2 and CO gas sensors respectively. Nano structured Nb-doped TiO_2 thick films have been prepared screen printing process for CO gas sensors [4]. Also for same doping O_2 sensitivity has reported [5]. TiO_2 behaves as semi conducting oxide due to non-stoichiometry behavior. Recently buried contact solar cells have been fabricated using TiO_2 as an optical coating .It is used as piezoelectric crystal sensor for the detection of organic vapors using nanocrystalline [6].It has interesting applications in chemistry due to its behavior as a reaction catalyst [7]. It is used as photo catalytic degradation of phenol one of the most water pollutants [8] and environmental purification [9]. Titanium dioxide occurs in nature in three crystalline forms: anatase, brookite and rutile. These crystals are essential pure titanium dioxide but certain amount of impurities such as iron, chromium or vanadium which darken them. Rutile is thermodynamically stable form at all temperatures and is one of the most stable forms at all temperatures and is one of the important ores of titanium [10]. Several deposition techniques have been developed to grow undoped and doped TiO_2 films such as spray pyrolysis evaporation, chemical vapor deposition, sol gel technique, magnetron sputtering pulsed laser deposition and screen printing technique. Thick films are suitable for such sensors since the gas sensing properties are related to the material surface and the gases are always adsorbed and react with the films surface [11]. Furthermore, development of gas sensors to monitor combustible gases is imperative due to the concern for safety requirements in homes and for industries, particularly for detection of liquefied petroleum gas (LPG) which is one of extensively used but potentially hazardous gases, because explosion accident may be caused when it leaks out accidentally or by mistake. So the detection of LPG in domestic appliances must be no false or missing alarms during cooking, which requires the equipment to identify LPG. Also H_2S and NO_2 gases are hazardous for our respiration system.

The aim of the present study is to prepare TiO_2 thick films by using Screen Printing Method on alumina substrates and study its gas sensing performance.

2. Experimental

AR grade powder of TiO_2 was calcined at 400°C for two hours in muffle furnace. Then the powder was crushed and mixed thoroughly with glass frit as a permanent binder and ethyl cellulose as temporary binder. The mixture was then mixed with butyl carbitol acetate as a vehicle to make the paste. The paste was then screen printed on to the surface of alumina substrate using standard screen printing technique [12-17]. The details of the technique were described elsewhere [14]. After screen printing the films were dried under IR-source for 1 hr and then fired at 600ºC, 700ºC, 800°C, 900°C and 1000°C for 45 minutes. D.C. resistance of TiO_2 films were measured by half-bridge method in an atmosphere at different temperatures. The thickness of the films was measured by using Taylor-Hobson (Taly step UK) system. The thickness of the films was observed in the range of 20 µm – 50 µm. The structural properties of TiO_2 films were investigated using X-ray diffraction (XRD) technique. The X-ray diffraction pattern were recorded with a Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having CuKα (λ=0.1542 nm) radiation. The Scanning electron microscopy (SEM) was employed to characterize the surface morphology with JOEL JED 2300. Composition analysis was performed to determine the Ti and O content in the film by using 6360 LA Energy Dispersive Spectrometer. The gas response of TiO_2 films was studied in a home-made gas sensor assembly. The electrical resistances of a TiO_2 film in air (R_a) and in the presence of gas (R_g) were measured to evaluate the gas response or sensitivity (S) defined as:

$$S = \frac{R_a - R_g}{R_a} \tag{1}$$

3. Results and Discussion

3.1. Structural Analysis

The XRD patterns of TiO_2 thick films for different firing temperatures are presented in Fig. 1. It indicates that [101] anatase peak located at 25.8° for temperatures 800 and 900°C. This is the most pronounced peak of an anatase structure. All values of [hkl] are matched with JCPDS data 21-1272 and 21-1276 for anatase and rutile structure respectively [18]. Up to 900°C temperatures both the phases of anatase and rutile were observed. At 1000°C firing temperature only rutile phase was found. This reduction of intensity indicates a decrease in the anatase content of the TiO_2 film, while the increase in width of peak indicates a decrease in anatase grain size. The similar results have been reported elsewhere [19]. The intensity of anatase peak decreases at 900°C temperature and at 1000°C, it disappears. It is clearly shows that phase transformation takes place up to 900 ° C and at 1000°C only rutile phase found. The crystallite size measurements were also carried out using the scherrer equation [20].

$$D = \frac{0.9\lambda}{\beta\ Cos\theta}, \tag{2}$$

where D is the crystallite size; λ is the wavelength of the X- ray radiation (1.54056 A°); β is the line width; and θ is the angle of diffraction. The average particle size obtained for anatase and rutile phases from XRD data are 17.86 nm and 37.99 nm respectively.

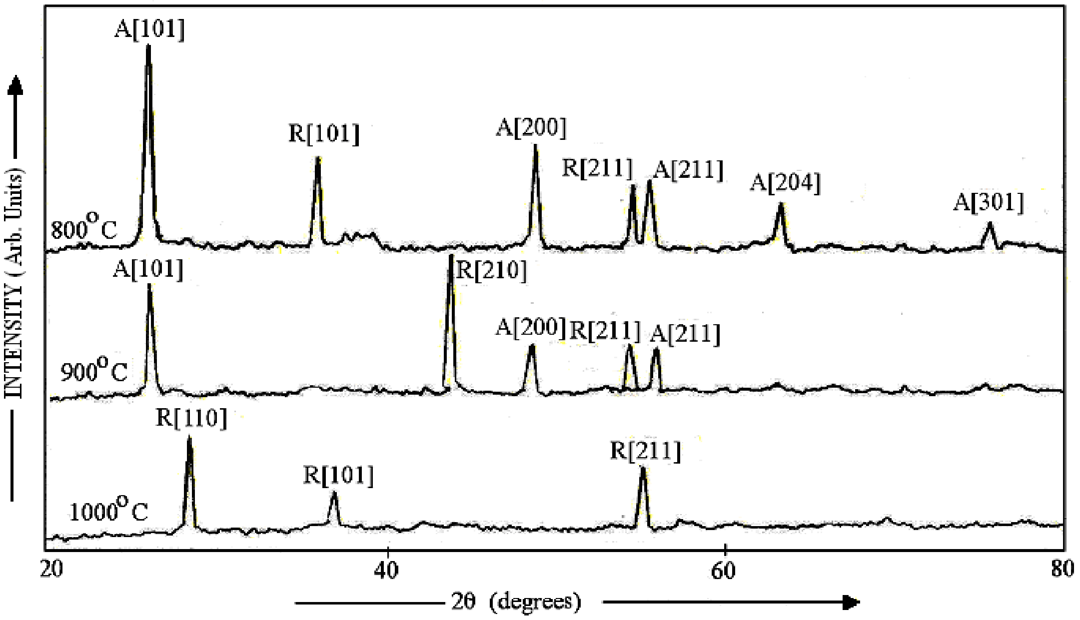

Fig. 1. XRD Pattern of TiO_2 films at different Firing Temperatures.

3.2. Surface Morphology Analysis

Scanning electron microscopy is convenient technique to study the microstructure of TiO_2 thick film samples. Fig.2. (a), (b), (c) shows the surface morphology of TiO_2 films fired at different temperatures observed by Scanning Electron Microscopy. All the images are recorded at 10000x magnification for the comparison. The micrograph of these samples shows voids between the particles are basically due to evaporation of the organic solvent during the firing of the films. The micrograph also shows the presence of more agglomeration in the film samples. The TiO_2 films show increase in grain size with increase in firing temperature.

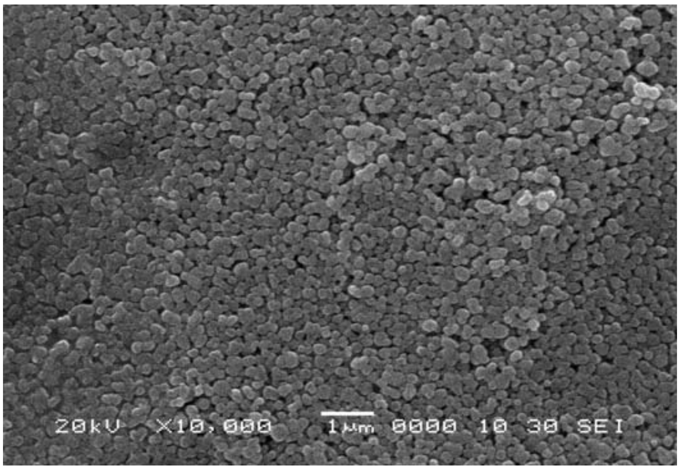

Fig. 2. (a) SEM image of TiO_2 Thick Film fired at 800°C.

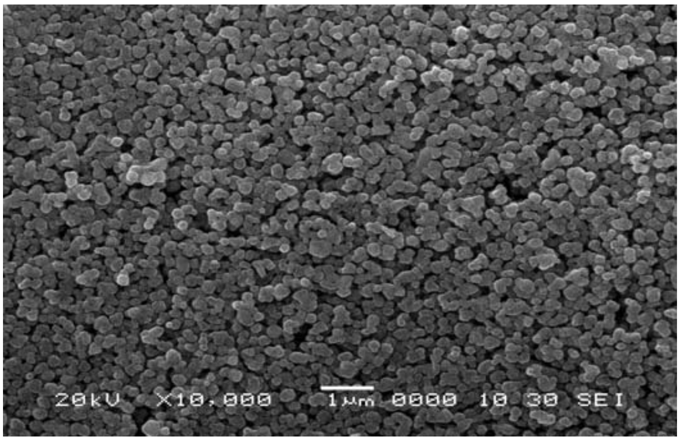

Fig. 2. (b) SEM image of TiO_2 Thick Film fired at 900°C.

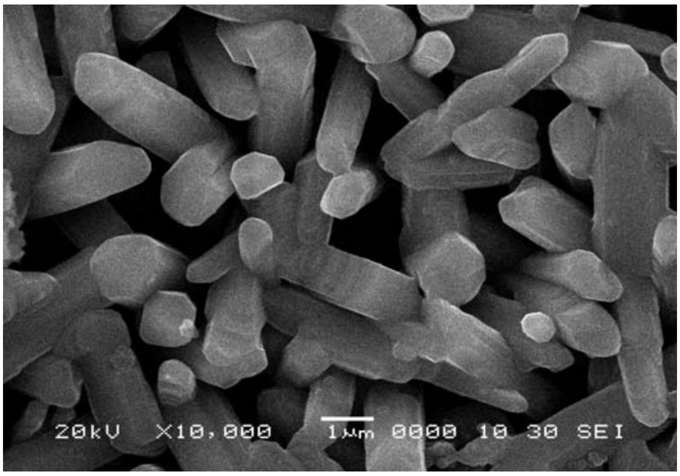

Fig. 2. (c) SEM image of TiO_2 Thick Film fired at 1000°C.

3.3. EDAX Analysis

The EDAX analysis was used to examine the composition of the film materials. Table 1. shows the composition of the elements of the TiO_2 films fired at different temperatures. It is evident from Table 1. that the EDAX analysis shows the major peaks of Ti and O and no other impurity elements are presents in the composition. Also from the spectra it is seen that weight % and atomic % are nearly matched as illustrated in Table 1. The EDAX analysis also shows that by firing at higher temperatures, excess oxygen is released. [21].

Table 1. Quantitative elemental analysis of TiO_2 films obtained from EDAX.

Firing Temp. / **Sample- TiO_2**	**800^0C**	**900^0C**	**1000^0C**
Ti (Mass %)	57.63	59.92	60.27
O (Mass %)	42.37	40.08	39.73
Total	100.00	100.00	100.00

From the analysis it was found that TiO_2 films are non- stochiometric.

3.4. Gas Sensing Response

3.4.1. LPG Sensitivity

TiO_2 thick films for anatase phase is found to be sensitive to oxygen, nitrogen and reducing gases such as ethanol and hydrogen [22]. The sensing properties of the material are based on reactions between metal-oxide semiconductors and gases in the atmosphere. These reactions produce changes in the semiconductor electrical properties. There are many kinds of possible reactions. For metal-oxide semiconductor gas sensors, the more common reaction that leads to changes in the resistivity is the adsorption of gases on its surface. The details of LPG sensing mechanism are found in the literature [23]. In present paper, the TiO_2 film exposed to various gases, every gas was allowed to equilibrate inside the gas chamber at an operating temperature for 2 minute and the stabilized resistance was taken as R_a. The TiO_2 film fired at 800 ºC was found to have a good sensing response to LPG at operating temperature 350 ºC for 1000 ppm shown in Fig. 3. The sensitivity rises up to 350^0C and then falls at higher temperatures. It is generally accepted [24-26] that the surface conductance of the sensor increases with the partial pressure P_g of the test gas in ambient air according the relation

$$G_g = G_a + \gamma (P_g)^{1/2},$$

where G_a is the conductance of the sensor in absence of the test gas in ambient air and γ is the constant proportionality governed by the adsorption process. Also above 350^0C temperature, the surface of TiO_2 film sample would be unable to oxidize the LPG gas so intensively. Therefore, the sensitivity decreases further with increasing temperature [27].

3.4.2. Gas Concentration

The variation of gas response of the TiO2 sample with LPG gas concentration at 350 ^0C temperature is shown in Fig. 4. This film was exposed to varying concentrations of LPG. The sensitivity values were observed to increase continuously with increasing the gas concentration up to 2000 ppm. It is seen that the gas response increases as gas concentration increases.

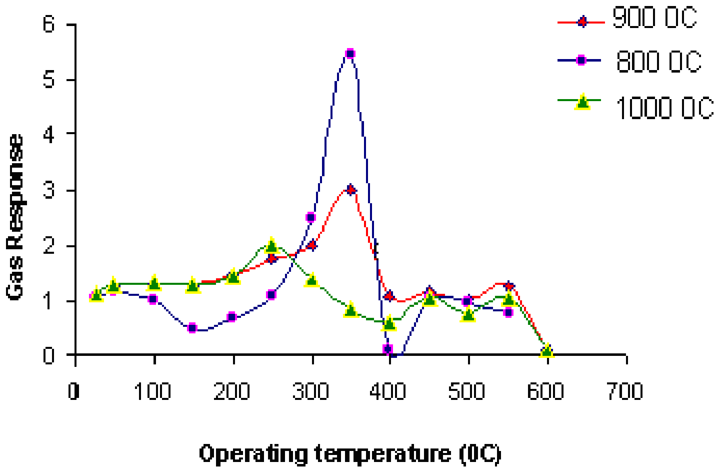

Fig. 3. Variation of gas response with operating temperature for 1000 ppm**.**

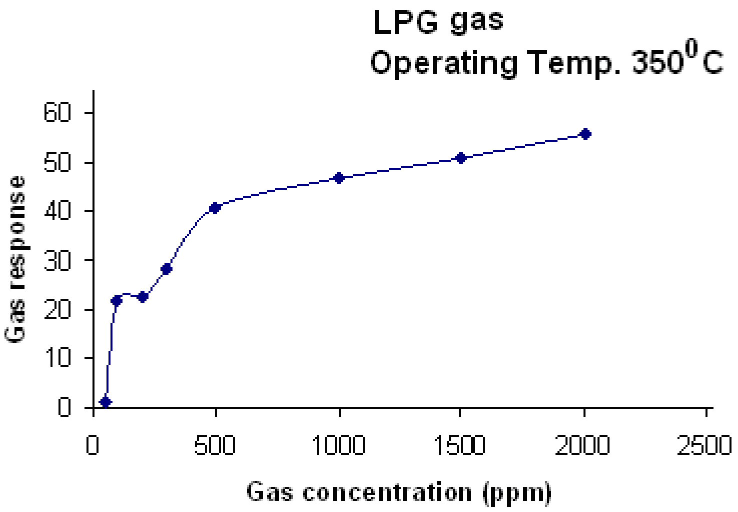

Fig.4. Variation of gas response with LPG gas concentration.

3.4.3. Gas Selectivity

Fig. 5. shows the bar diagram indicating the selectivity of the pure TiO_2 sensor operated at 350°C to LPG gas against CO_2, H_2S, NO_2, NH_3 and Ethanol gases. It is evident from the figure that the TiO_2 sensor was highly selective to LPG gas against CO_2, H_2S, NO_2, NH_3 and Ethanol gases. The TiO_2 films fired at high temperature provides a large high surface area to interact LPG molecules. It is well known that the response of metal oxide semiconductor sensors is mainly determined by the interactions between the target gas and the surface of the sensors. So, it is obvious that for the greater surface area of the materials, the interaction between the adsorbed gases and the sensor surface is stronger, i.e. the gas response is larger [28]. LPG sensing has reported 35.8 % for 800 ppm [29]. In our results it was found to be 5.44 (544 %) gas response for 1000 ppm at 350 ^{0}C operating temperature for TiO_2 film deposited on alumina substrate fired at 800 ^{0}C. Above 800 ^{0}C film has better adhesion to alumina surface. At firing temperature of 900^{0}C antase phase reduces greatly and at 1000^{0}C this phase

was completely disappeared. So above 900^{0}C films was not shown gas response as 800^{0}C. It was observed that anatase phase is more sensitive to gas response than rutile phase.

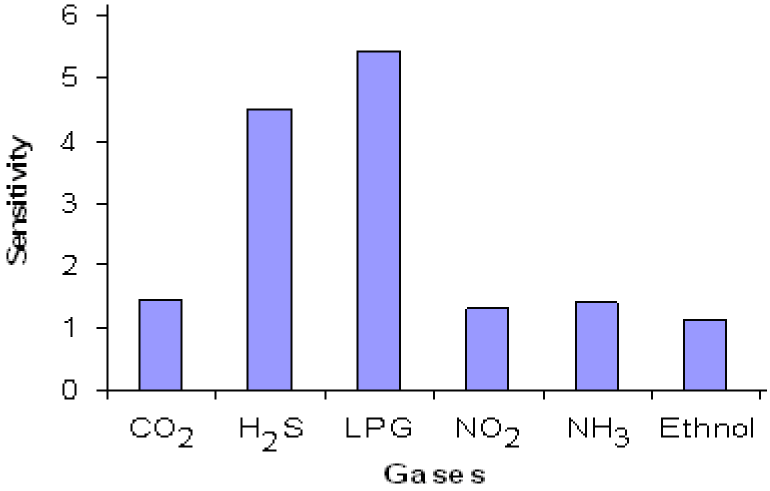

Fig. 5. Selectivity of TiO_2 film fired at 800 ^{0}C to various gases.

3.4.4. Gas Response and Recovery Time

The response and recovery of TiO2 sample are represented in Fig. 6. The response was quick (~ 45 s) to 1000 ppm of LPG while the recovery was fast(~ 75 s).The quick response may be due to faster reduction of gas. Its high volatility explains its quick response and fast recovery to its initial chemical status.

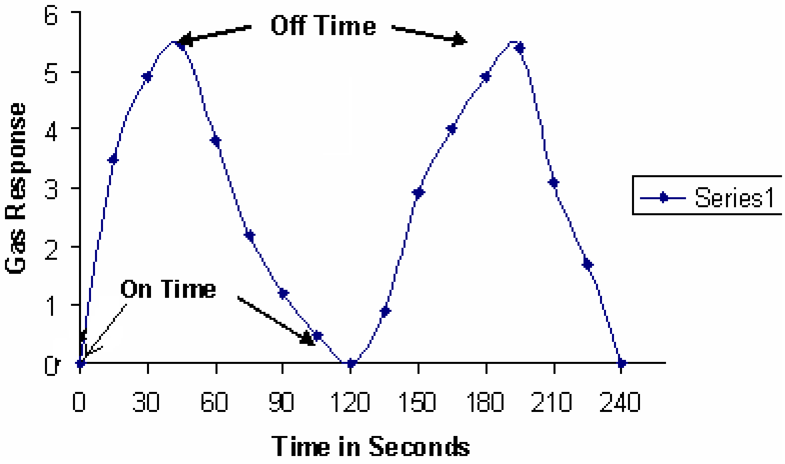

Fig.6. Response time for LPG gas at 350 ^{0}C operating temperature.

4. Conclusions

This work demonstrated the successful preparation of TiO_2 screen printed thick film shows good adhesion to alumina substrate employing a simple, inexpensive method and capability of the TiO_2 films for LPG sensing. The film fired at 800 ºC has better stickiness to alumina surface and exhibited good sensing performance to LPG gas.

Acknowledgement

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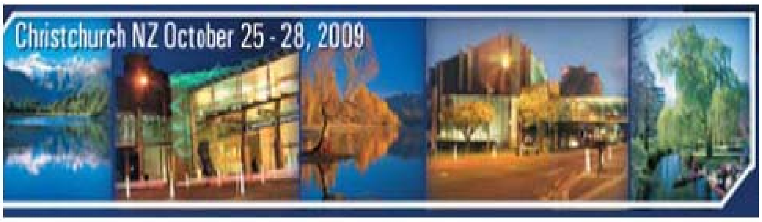

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