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## Fabrication and Testing of SnO<sub>2</sub> Thin Films as a Gas Sensor

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### ABSTRACT

Tin dioxide (SnO<sub>2</sub>) thin films were prepared using the thermal spray pyrolysis method from SnCl<sub>2</sub>·2H<sub>2</sub>O isopropyl mixing with water solution on the glass substrate heated at 450°C. The films were characterized by X-ray diffraction (XRD), The crystallite size was evaluated to be 4.413 nm by using the Scherrer's equation. Atomic Force Microscope (AFM) and optical absorption spectra were taken to examine the surface morphology and optical properties to determine the band gap energy 3.9 eV, increase in band gap energy is revealed that nanostructure SnO<sub>2</sub> films. These films were tested in butane gas at operating temperature 470 °C. The response and recovery time is 13 sec, 63 sec respectively.

**Keyword:** SnO<sub>2</sub> Thin films; spray pyrolysis; butane gas sensing; sensitivity.

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### INTRODUCTION

Semiconductors are used as gas sensors based on the principle of the variability of electrical conductivity of metal oxides when exposed to these gases [1]. These properties can be utilized to detect NO<sub>x</sub>, H<sub>2</sub>, volatile organic compounds (VOCs), SO<sub>x</sub>, CO<sub>2</sub> and O<sub>2</sub>, etc [2-4]. Metal oxide semiconductor (MOS) sensor technology is based on the change in resistance of a sensitive metal oxide layer which is induced by the interaction between a surface and ambient gases. Metal oxide semiconductors demonstrate good detection sensitivity, robustness and the ability to withstand high temperatures and the technique is commonly used to monitor a variety of toxic and inflammable gases in a variety of air pollution monitoring systems, the food industry, medical diagnosis equipment and gas leak alarms. Since the last decade there has been a great deal of interest in the preparation of inexpensive thin films of SnO<sub>2</sub>. This is because tin dioxide based thin films with large band gap ( $E_g > 3$  eV) *n*-type semiconductors are attractive from the scientific and technological point of view [5].

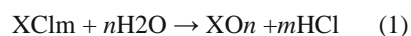
A variety of technique has been used to deposit tin oxide (SnO<sub>2</sub>) thin films. These include spray pyrolysis [6], chemical vapor deposition [7], ion-beam assisted deposition, sputtering [8], and sol-gel methods [9, 10]. Among these techniques, spray Pyrolysis has proved to be simple, reproducible and inexpensive, As well as suitable for large area applications. Besides the simple experimental arrangement, high growth rate and mass production capability for large area coatings make them useful for industrial as well as solar cell application. In addition, spray pyrolysis opens up the possibility to control the film morphology and particle size in the nm range [11]. Up to now, many researchers have prepared SnO<sub>2</sub> using chemical spray pyrolysis. For example, growth of tin dioxide thin films [12]. Reports have shown that crystal size in the films could be controlled over a nm range by varying the film thickness, deposition method and post deposition annealing temperature [13]. Taguchi (1962), had reported that

partially sintered SnO<sub>2</sub> pellets respond similarly [14]. These were the beginning for what has been a rapid gas sensor development phase. Also some recent studies on the sensing properties of pure nanocrystalline SnO<sub>2</sub> thin films toward H<sub>2</sub>S, H<sub>2</sub> and butane [15-18] seemed to contradict the general trend that higher sensitivity is to be expected for smaller crystals, and it was, therefore, concluded that small size of crystals was an essential but not sufficient condition for the achievement of maximum gas sensitivity and fast response. It is also reported that Malyshev *et al.* observed highest sensitivity of sputtered undoped SnO<sub>2</sub> thin films at 450 °C in presence of butane and propane [19].

In this work is to prepare SnO<sub>2</sub> thin films by the spray pyrolysis method, and demonstrates of the butane sensing properties.

## MATERIALS AND METHODS

SnO<sub>2</sub> thin films were deposited using an aqueous – isopropanol solution including SnCl<sub>2</sub>·2H<sub>2</sub>O (99.8%, Aldrich) (0.1 M), H<sub>2</sub>O and CH<sub>3</sub>CH<sub>2</sub>OH (1:1) and a few ml of hydrochloric acid by the spray pyrolysis technique. For film deposition, All substrates (the glass substrates with 1 mm thickness and 25 × 75 mm dimensions), were cleaned in acetone and methanol solutions in an ultrasonic bath for 30 minutes and rinsed in deionized water and dried in high purity N<sub>2</sub> gas stream and were placed on the hot plate at temperatures ranging from 440-460°C, which is known to be the optimal range for the formation of SnO<sub>2</sub> films [20]. Then solution was sprayed at the following conditions: carrier-air pressure: 1-2 atm, flow rate of solution: 9 ml/min and substrate to nozzle distance: 35 cm. For preventing reduction at hot plate temperature, spraying was done in short time intervals. The metallic salt solution, when sprayed onto a hot substrate, pyrolytically decomposes and a chemical reaction takes place on the heated substrate and at least a thin layer of SnO<sub>2</sub> is deposited. The deposition process needs fine droplets to react on the heated substrate, owing to the pyrolytic decomposition of the solution. The hot substrate provides the thermal energy for the thermal decomposition and subsequent recombination of the constituent species. In many cases large droplets of the solution do not vaporize before reacting to deposit on the substrate. The phenomenon for the preparation of a metal oxide thin film depends on surface hydrolysis of metal chloride on a heated substrate surface in accordance with the equation [21].



Where X is the metal such as Sn, Zn, Cu etc of the oxide films.

The structural of the thin films were examined by X-ray diffractometer (6000-Shimadzu) using CuK<sub>α</sub> radiation with a wavelength, λ=1.54060 Å. The morphological of the films were analyzed using scanning Probe Microscope (SPM, model AA3000 Angstrom Advanced .Inc). The optical absorbance of the films was measured using UV-visible spectrophotometer (SP-3000 Optima) in the wavelength range 200-1200 nm at room temperature. The deposition of aluminum electrodes using masks where the finger width is 1µm and distance between two fingers is 1µm. The gas sensing chamber had been employed for testing of the films to gases. The heater was placed on a base plate to heat the sample under test up to required operating temperature 470°C, a fixed bias voltage of 5 V was applied across the films. The current was measured using a Scope digital Multimeters (UT81). Films response to butane gas was studied by introducing the gas of known concentration (5-15%) volume ratio to the air and recording current as a function of time.

## RESULTS AND DISCUSSION

The typical XRD spectra of SnO<sub>2</sub> films with thickness is 86 nm are shown in Figure 1.

We observe in XRD the peaks characteristic are polycrystalline with low intensity and presence of additional reflections characteristic for amorphous or highly dispersed randomly orientated particles. And we observe peaks, which are characteristic for structure of the cassiterite SnO<sub>2</sub> phase; this indicates that the films of tin oxide, deposited by spray pyrolysis method are stoichiometric. In our films the XRD spectra show predominance of the peaks, corresponding to reflection from the crystallographic (110) planes, parallel to the substrate. The intensity of other peaks is small it indicates that our films are textured. The degree of the texturing depends on kind of sprayed solution. SnO<sub>2</sub> Films deposited from alcohol solution they indicate that the change of predominant orientation of crystallites, forming gas sensitive matrix. For thin dense films only external surface contacts with surrounding atmosphere. Therefore, for such films observed effect can play an important role. Adsorption

/desorption are structure sensitive processes. Hence, the changing in predominant orientation of active surface must change the energy of adsorption /desorption processes, and consequently, change both response and recovery times. The size of crystallites SnO<sub>2</sub> films deposited from alcohol solution depends on both the pyrolysis temperature and crystallographic orientation. The average crystallite size increases with the deposition temperature. The average grain size of tin oxide is 4.413 nm for thin film samples were calculated by using the Scherrer's equation (Cullity 1956)[22].

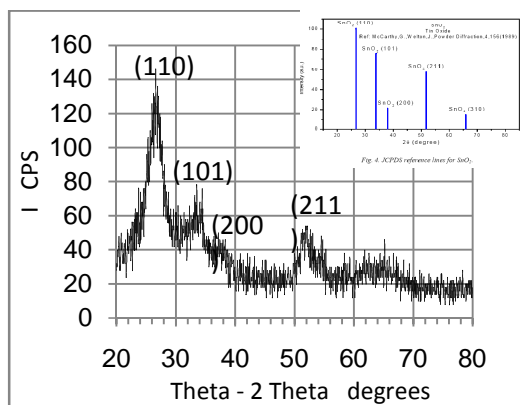


Fig. 1. XRD spectra of SnO<sub>2</sub> thin films (d=86nm) deposition at temperature 450°C.

$$D = 0.9\lambda/\beta\cos\theta \quad (2)$$

Where D is the average grain size  $\lambda=1.542 \text{ \AA}$  X-ray wavelength,  $\beta$  the peak FWHM and  $\theta$  the diffraction peak position. This is considered as an evidence of the good quality and structural stability of SnO<sub>2</sub> thin films, deposited by spray pyrolysis. A structural and morphological study was carried out by means of Atomic Force Microscope (AFM), (Figure. 2) show typical AFM images of SnO<sub>2</sub>, although z-range suggests that asperities are of the order of 49.7 nm, And RMS roughness 7.3 nm.

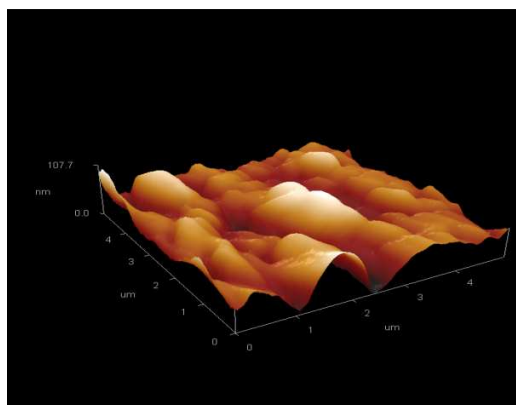


Fig. 2. AFM image of SnO<sub>2</sub> thin film deposited on glass substrates at temperature 450°C.

Films are smooth enough due to optimization of deposition condition such as distance between the nozzle and substrate and quantity of spraying solution. Moreover, smoothness of the present films can also relate with the orderedness caused by substrate temperature.

The transmittance and absorbance spectra of SnO<sub>2</sub> films were recorded in the UV-visible near infrared regions (200 nm to 1200 nm) optical properties of SnO<sub>2</sub> thin films such as transmittance, absorbance, indirect band gap, were calculated on as-deposited films. Figure 3 shows the variation of transmittance with wavelength for as-deposited SnO<sub>2</sub> thin films.

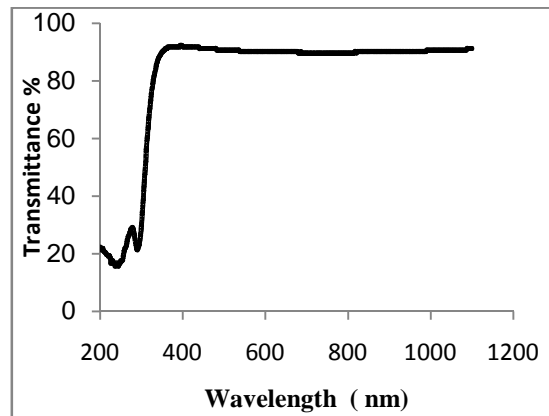


Fig. 3. Optical transmittance of SnO<sub>2</sub> thin film deposited on a glass substrate at 450 °C.

It is seen that the values of transmittance is high in the visible and IR region and minimum at wavelength ~ 300 nm. The highest transmittance about 90 %. The band gap of the films was calculated by plotting  $(\alpha h\nu)^2$  vs.  $h\nu$  using the relation,

$$\alpha h\nu = B(h\nu - E_g)^n \tag{3}$$

Where  $\alpha$  is absorption coefficient, A is a constant,  $E_g$  the optical bandgap energy,  $h\nu$  the photon energy and n a constant. The value of n is 1/2 or 2 depending on presence of the allowed direct and indirect transitions in Figure 4.

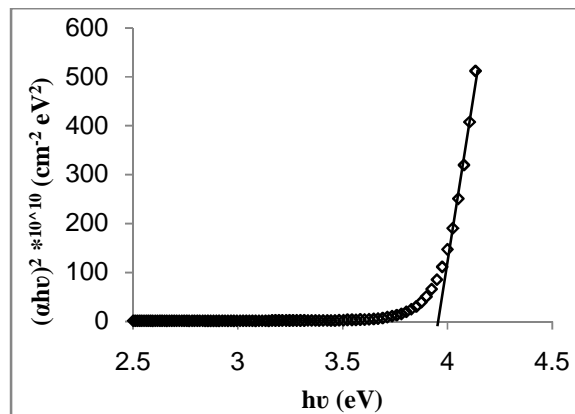


Fig. 4. Plots of  $(\alpha h\nu)^2$  vs. photon energy,  $h\nu$ , of SnO<sub>2</sub> thin films.

The optical band gap is 3.9 eV, Often an increase in band gap energy is observed when nanostructure SnO<sub>2</sub> increase in grain size [23].

In air, I – V characteristics of nanostructured SnO<sub>2</sub> films are shown in figure 5 increase nearly linear relationship curve was noted for positive applied bias voltages up to 10 V.

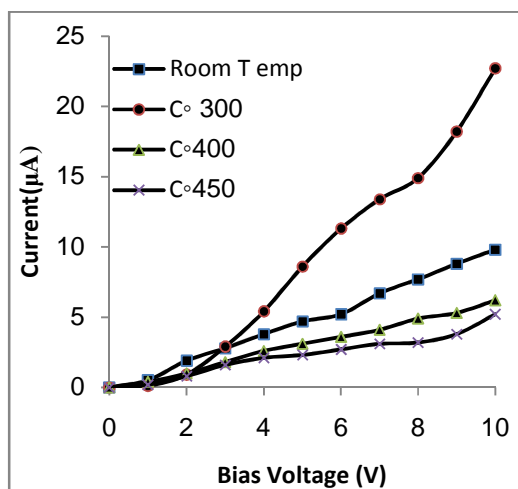


Fig. 5. I - V characteristics of SnO<sub>2</sub> films in the air with thickness (d=216 nm).

The resistance variation in air is attributed to the effect of oxygen chemisorption. It is generally accepted that oxygen is chemi-adsorbed at a surface site such as oxygen vacancy in the form of an ionized oxygen atom or molecule, i.e. O<sup>-</sup> or O<sub>2</sub><sup>-</sup>, resulting in a reduced concentration of free electrons at the surface and the observed reduction in the conductivity [24]. The effect of temperature on the I - V characteristics confirms the enhancement of the current with temperature from room temperature up to 300 °C. As the temperature is increased, more electrons have sufficient energy to surmount the barrier height between the grains. It can be observed that there is a decrease in the measured current as the temperature is further raised above 300 °C indicating an increase in the film's resistance. This effect is observed in the chemisorption region at elevated temperatures (300 – 500 °C) [25]. Where the oxygen is adsorbed at the surface of the metal oxide that enable an electron trapping. Hence the charge carrier density is reduced which leads to an increase in the resistance of the SnO<sub>2</sub>. This reaction can be expressed as follows:



Where O<sub>2</sub> is the adsorbed oxygen molecules, O<sup>-</sup> is the chemisorbed oxygen and e<sup>-</sup> is the trapped electrons from the SnO<sub>2</sub> surface. In the butane gas (Figure. 6) the current was increased especially at 450 °C Where the butane return electrons to the conduction band .

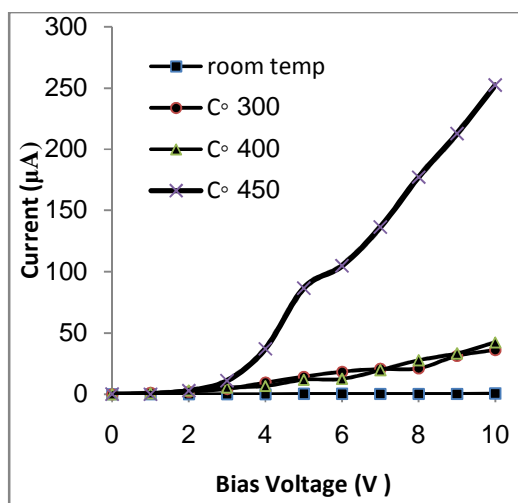


Fig. 6. I - V characteristics of SnO<sub>2</sub> films in the butane gas with thickness (d=216 nm).

To improve the gas-sensing properties it is necessary to prepare very thin sensing layers with high surface-to-volume ratio. In The large number of grains which leads to high porosity and large effective surface area available for adsorption of gas species. In case of reducing gas, the adsorption of oxygen on the surface extracts conduction electrons from the near surface region forming an electron depleted surface layer, which results in an electric field and a potential barrier associated with this electric field [26]. The potential barrier is depending upon the concentration of adsorbed oxygen. When reducing gas butane adsorbed on the SnO<sub>2</sub> surface then adsorbed oxygen is consumed by reacting with butane gas, and the entrapped electrons are returned to the SnO<sub>2</sub> grains resulting in a decrease of the potential barrier and the resistance [27, 28]. From Figure 7, it is found that sensitivity increases with increase in operating temperature and show maximum peak values at certain temperatures called optimal temperature and then decreases with further increase in temperature.

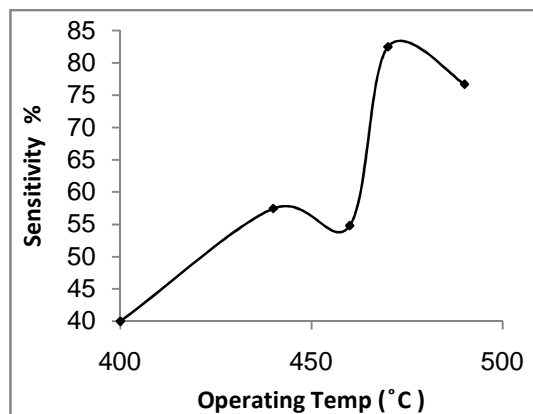


Fig. 7. Variation of sensitivity with temperature of SnO<sub>2</sub> thin film for 5 vol% butane in air and 5 bias volt.

At the optimal temperature, the activation energy may be enough to complete the chemical reaction. The observed increase and decrease in the sensitivity indicates the adsorption and desorption phenomenon of the gases. The optimal temperature was confirmed for each SnO<sub>2</sub> film samples for five cycles. For n-butane gas, the response was observed to increase with operating temperature up to 400 °C. The resulting equation is

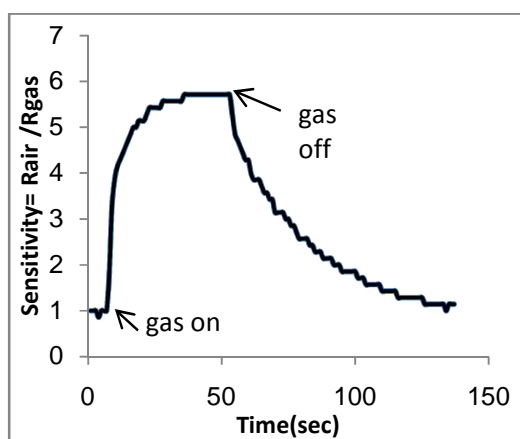
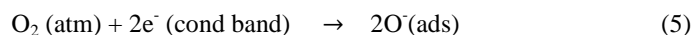


Fig. 8. Sensing response of SnO<sub>2</sub> films exposed to 5 vol % butane in air and operating temperature 470 °C.

After 470 °C temperature the surface would be unable to oxidize the gas so intensively and the LPG may burn before reaching the surface of the film at higher temperature. Thus, the gas sensitivity decreases with increasing temperature [29, 30]. From Figure 8, the higher sensitivity may be attributed to the optimum number of misfits on

the surface, porosity, largest surface area and the larger rate of oxidation of LPG at 470 °C for film. The maximum response of SnO<sub>2</sub> film to LPG gas was found to be 5.71 at 470°C.

The sensitivity factor  $S$  was defined as the ratio of the resistance in the air ( $R_a$ ) to the resistance in the butane gas ( $R_g$ ), which was expressed as

$$S = \frac{R_a}{R_g} \quad (6)$$

The response time was defined as the time taken for sensor to attain 90 % of maximum change in resistance or conductance upon exposure to LPG. The time taken by the sensors to get back 90 % of original resistance or conductance is the recovery time [31]. Figure 9 shows the typical change in response with time measurement of the SnO<sub>2</sub> thin film sensors are kept at the optimal temperature and the 5 vol% n-butane gas is injected in the measurement chamber.

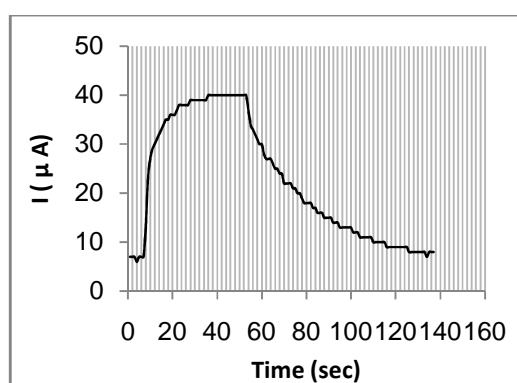


Fig. 9. Response and recovery time of the SnO<sub>2</sub> film exposed to 5 vol % butane in air and operating temperature 470 °C.

The change in the sensitivity of the sensor after the sensor comes in contact with 5 vol% n-butane gas at an optimal temperature of 470 °C. The response time is approximately 13 sec and the recovery time is obtained just by removing the chamber. It is also found to be approximately 63 sec. The quick response may be due to faster oxidation of gas. (Figure. 10) shows the sensitivity  $S$  of the sensor when exposed to n-butane gas with concentration levels (5,10,15) vol % butane to the air for SnO<sub>2</sub> at operating temperature is 470°C .

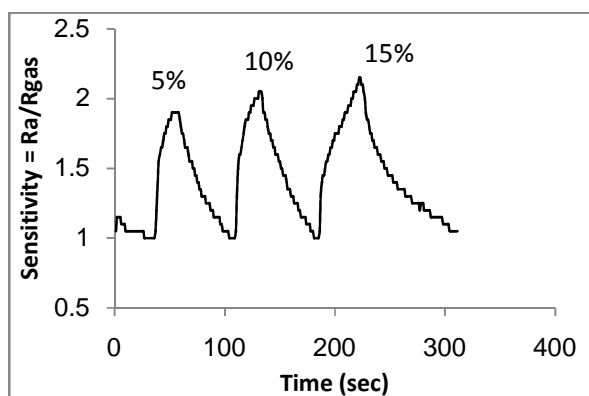


Fig. 10. change in sensitivity with respect to time of SnO<sub>2</sub> sensor in butane gas ( 5-15 vol%) at operating temperature of 470 °C

It can be observed that the sensor sensitivity increased when exposed to butane with higher concentration levels, as shown in Figure 11, which revealed to change of sensitivity with concentration of gas.

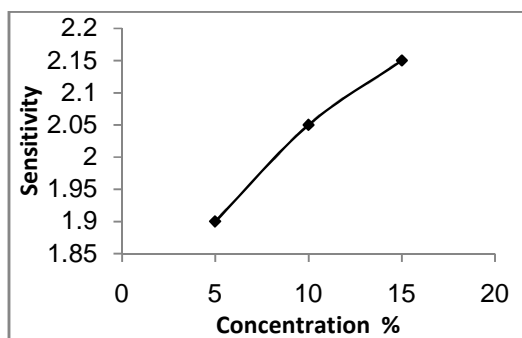


Fig. 11. Sensitivity of the sensor when exposed to butane for different concentration levels at the operating temperature of 470 °C.

The gas sensing mechanism depends on the surface reaction between chemisorbed oxygen and reducing gases. The adsorption of oxygen on the film surface has two forms: physisorption and chemisorption. At elevated temperature, chemisorption is dominant. The transition from physisorption to chemisorption needs activation energy, which can be accomplished by increasing operating temperature. It has been reported that the amount of oxygen adsorbed on the sensor surface goes on increasing with an increase in temperature, reaches to maximum and then decreases with further increase in operating temperature. The response to the gas to be tested follows the same behavior [32].

Figure 12 shows reducing gas sensing mechanism. Barrier height before exposure of is high and therefore the resistance is high.

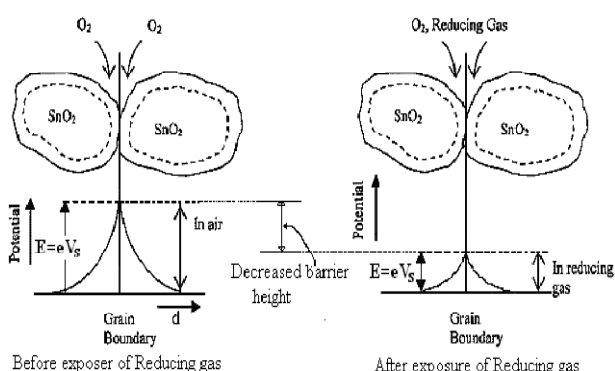
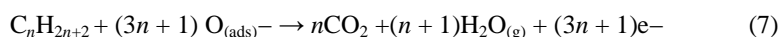


Fig. 12. Gas sensing mechanism of SnO<sub>2</sub> film sensor. (Model of inter-grain potential barrier)[33].

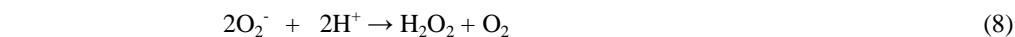
When reducing gas comes in contact with the sensor surface, it gets oxidized. Therefore the resistance of the sensor abruptly reduced. The rate of oxidation would be the function of the amount of adsorbed oxygen on the surface and the type of gas to be tested. The larger the rate of oxidation, the larger would be the number of electrons released, and in turn the larger would be the gas response.

As the butane is the major constituent (55 vol %) of LPG. It requires high temperature (~ 350-450 °C) to dissociate into lower alkanes. Carbon-Carbon and Carbon-hydrogen bonds are quite strong due to strong Vander Waals forces. They break only at higher temperatures resulting in carbon and hydrogen separation. The atmospheric oxygen O<sub>2</sub> adsorbs on the surface of the film. When alkanes react with oxygen, a complex series of reaction takes place [34, 35]. Butane gets oxidized to CO<sub>2</sub> and H<sub>2</sub>O. Ultimately converting the alkanes to carbon dioxide and water as:



Here, C<sub>n</sub>H<sub>2n+2</sub> represents a mixture of hydrocarbons like butane (C<sub>4</sub>H<sub>10</sub>; n = 4). This shows n-type conduction mechanism [30, 36]. At higher temperature, molecular oxygen O<sub>2</sub> becomes O<sup>2-</sup> and alkanes decompose producing hydrogen ions H<sup>+</sup> in the reaction. The anion superoxide O<sub>2</sub><sup>-</sup> reacts with H giving water molecule and molecular oxygen O<sub>2</sub>:





Catalase



LPG gas on exposure decomposes into carbon and hydrogen species, which react with adsorbed oxygen, liberating the captured electrons into conduction band resulting in enhancing the catalytic activity of the film surface.

### CONCLUSION

We have successfully prepared SnO<sub>2</sub> films by spray Pyrolysis method using SnCl<sub>2</sub>·2H<sub>2</sub>O. The resulting SnO<sub>2</sub> films were characterized by XRD measurements and AFM. The band gap values obtained from the absorption spectra was found to be 3.9 eV. The maximum sensitivity was obtained at an operating temperature of 470 °C for the exposure of 5% of butane gas. The results of the butane sensing studies reveal that the SnO<sub>2</sub> films prepared by spray pyrolysis method are a suitable material for the fabrication of the butane sensor.

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