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Design and Fabrication of Low Cost and Miniaturized Setup for Gas sensor

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Abstract: *In this communication, a simple and low cost gas sensor setup has been design and fabricated to measure the gas sensing response of metal oxide thin films. The setup is also extended for temperature dependent gas response by designing a micro furnace using a small resistive heating element that is easily available. Importance of prepared setup lies in its easy design, portability, reliability and low cost to carryout gas sensing measurements both at laboratory and field applications. It is tested using tin oxide thin films deposited on glass substrate using chemical spray pyrolysis. It was found to be very sensitive even for a small amount of gas and the recorded gas response to electrical resistance change are quite reproducible. Hence here in it is proposed that the present set is an innovative design to achieve low cost and portable gas sensing device.*
Key words: *Metal oxide, design and fabrication of gas sensor, tin oxide film, temperature dependent gas response.*

I. Introduction

Gas sensors have a great influence in many important areas viz., environmental monitoring, domestic safety, air conditioning in aero planes, space crafts, gas leak detection in various domains [1]. Gas detection methods became a concern after the effects of harmful gases on human health were discovered. Chemical gas sensor is a device, which upon exposure to gaseous species or molecules alters one or more of its physical properties, such as mass, electrical conductivity, dielectric response and optoelectronic properties in a way that is possible to measure and quantify [2]. These changes deliver an electrical signal with a magnitude that is proportional to the concentration of gas under the test, which is measured as quantity of gas to which the measuring sensor is exposed.

Presently many analytical techniques are used for sensing different gases that are critically important due to various reasons. The techniques include electrochemical sensors; they function via electrodes signal when a gas is chemically absorbed on electrode surface [3], catalytic sensors; these works via catalytic oxidation/reduction [4], Infrared sensor or IR detectors; these work on the basis of absorption and emission of a small amount of energy by chemical bond due to the resonance phenomena [5], FTIR based sensor: if a gas is present in the optical path, it well interfere with the power of the light transmission between transmitter and receiver. The altered state of light helps to quantify the amount of gas present in a given volume [6]. Metal oxide film chemical sensors; here the change in the physical properties of the film upon gas exposure is measured [7]. Sensing behavior is the most important and well-known property of the metal-oxide materials. Metal oxides demonstrate high sensitivity to their chemical environment [8]. Further, these are basically wide band gap semiconductors and are more stable and hence operate in harsh chemical environments, they surpass other chemical sensors in their sensitivity, reliability, durability and ease of synthesis [9]

The working mechanism of metal oxide sensors is the variation of surface electrical conductance in the presence of a gaseous environment. This induced the change in electrical conductivity by the adsorption of gaseous species on the dangling bond at the surface film by a chemical reaction. The metal oxide causes the gas to dissociate into charged ions or complexes on the surface, which results in the transfer of electrons from film to gas molecules or vice versa. This property has been exploited and used for the detection of toxic gases. At present the research on these gas sensors is aimed at obtaining new materials to achieve high sensitivity, a good stability and a long operating life [10]. In the present communication an innovative gas sensor setup has been designed and fabricated as low cost, reliable and portable method to carry out the gas sensing measurements. The present setup tested well for tin oxide (SnO_2) films with respect to Hydrogen sulphide (H_2S) gas

II. Experimental technique

All the chemicals used for the synthesis of SnO_2 thin films were of analytical grade and were used without further purification. Stannous chloride $SnCl_4.5H_2O$ used as the precursor that was obtained from Thomas Beker, India. Ethanol (C_2H_5OH) was used as the solvent for all the synthesis is obtained from sdfine chemicals, India. Our home made chemical spray pyrolysis setup is adopted for the synthesis of SnO_2 films.

Here the spraying system consists of spray nozzle, air compressor and mechanical arrangement for one dimensional motion. And heating unit consists of a hot plate, thermocouple, temperature indicator and variac. Spray nozzle and hot plate with glass substrate are housed in a metallic box and the out let of the box is fitted with an exhaust fan to remove the toxic gases produced during the decomposition of spray solution. In a typical synthesis of these films, $SnCl_4.5H_2O$ was dissolved in ethanol and stirred well for a long time and filtered using filter paper to get a clear solution. Here 0.2 M solution of $SnCl_4.5H_2O$ was prepared in 20 ml of ethanol. The commercial laboratory glass slides of dimensions 25 mm x75 mm and thickness 1mm were used as substrates to deposit films. The slides were washed with de-ionized water, rinsed in ethanol and dried and placed in laboratory oven at temperature approximately 50˚C. The prepared 20 ml solution was sprayed on a preheated glass substrate. The temperature of the substrate was maintained at a constant value of 400˚C with ±1˚C by using variac and digital temperature controller. The nozzle to substrate distance was kept constant at 35 cm. The entire solution was sprayed in about 8-10 minutes. For the uniform deposition of the solution the substrate is kept stationary while the nozzle is made to move to and fro on a line with a programmed stepper motor using micro controller, where the program is such that one can set the speed of nozzle motion and the number of cycles to be repeated during the deposition. Once the spray is completed the heater was turned off and the SnO_2 films were allowed to attain the room temperature by naturally cooling.

The as prepared SnO_2 films were used for further characterization. X-ray diffractometer (Ultima IV Japan) with CuK_α radiation (λ=1.5405 Å) at 40 mA and 40 kV at a scanning rate of 0.02˚ per second was used to study the crystal state of these films. Optical properties of the films were studied using UV-VIS spectrometer (Specord- 200 plus Germany) in the wavelength region 200 – 1100 nm. The current-voltage (I-V) characteristics of the films were studied using programmed Keithley source meter (Keithley 2636A). The sensing behavior of the films in terms of variation in the resistance was studied using digital multimeter and the temperature of the micro heater by digital thermometer using alomel- chromel thermocouple.

In order to use SnO_2 for gas sensing application, the as prepared SnO_2 films were cut into small pieces having width 5 mm and length 7 mm. These films were washed with de-ionized water and rinsed in boiling trichloroethylene (C_2HCl_3) solution to degrease them. Later on either side of the film surface along the length two thin copper wires are attached by applying silver paste and allowed to dry for an hour under table lamp and then an adhesive araldite is applied at the edges and kept for about 10-12 hours in order to have physically firm contacts. The schematic diagram of the as prepared sensing element is shown in figure (1). The area available between the two copper wires is an active region for gas sensing measurements. To test the workability of the sensing element H_2S gas was used. It was generated using FeS and dilute HCl in a sealed container. The chemical reaction is given below as equation (1)

$$FeS\downarrow + 2\,HCl\downarrow \rightarrow FeCl_2\downarrow + H_2S\uparrow \quad (1)$$

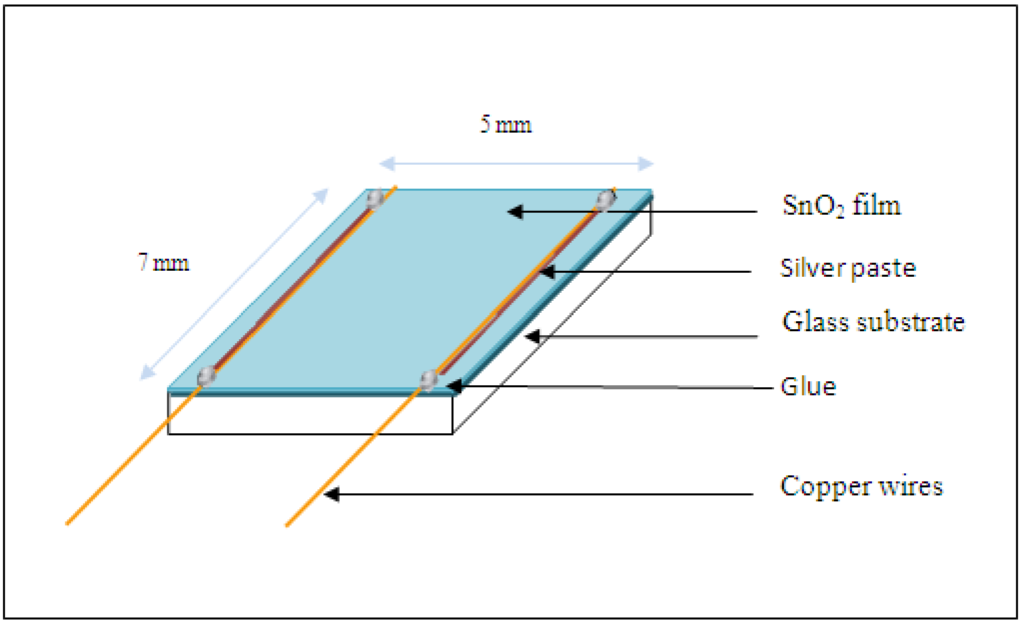

Figure 1: Schematic diagram of sensor element

III. Results and Discussion

3.1 Experimental setup

The schematic diagram of the experimental setup of our miniaturized gas sensing unit is shown in figure (2). A printed circuit board (PCB) strip (1) of width 1 cm and length 7 cm is used as platform to design the present setup. Four copper contacts (2) along the length are made on PCB by etching out unwanted copper using ferric chloride solution, of these contacts; the two are used for electrical resistance measurements and the other two for power to micro heater. An electrical heater is recovered from multiple resistors that are commercially available at any electronic shop; the retrieved micro heater has dimensions 10 mm x 7 mm x 2 mm with resistance 101 Ω. A d.c voltage of 12.5 V is applied to the heater that gives temperature of the oven about 110˚C within three minutes. A small oven (3) having dimensions 10 mm x10 mm x 8 mm with a micro heater (4) embedded in it is made as shown in figure (2). The inner volume of 10 mm x 6 mm x 5 mm is available for the mounting of sensor element. The oven is fixed at one end of the PCB and the other end is passed through the cork of the bottle such that the PCB is projected about 1 cm outside the bottle surface. The sensor element (5) of SnO_2 thin film is loaded in the oven and the contacts are soldered to the copper electrodes meant for sample on the PCB. This entire assembly is housed in a 15 ml laboratory cultured bottle, figure (3) shows the photograph of the complete setup.

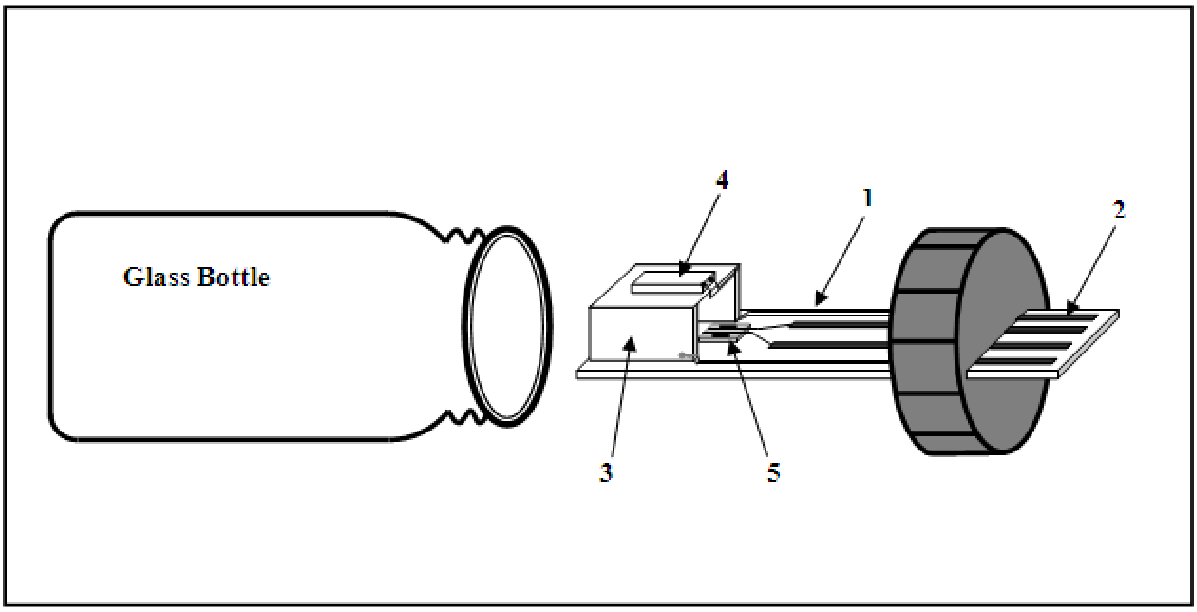

Figure 2: Schematic diagram of gas sensor setup
(1) printed circuit board, (2) copper contacts (3) oven (4) micro heater and (5) sensing element

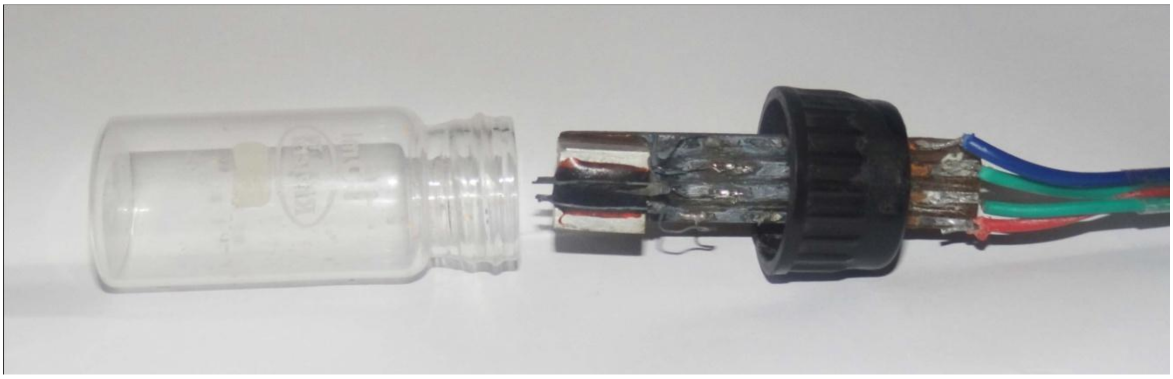

Figure 3: Photograph of gas sensor setup

The above sensing element with electrical contacts is placed in a small oven designed for localized heating. The gas response in terms of the variation in resistance when exposed to gas at different temperatures is recorded. H_2S gas is used to test the performance of the gas sensor unit. Every time a known amount of H_2S gas is injected into the bottle and sealed tightly and the variation in the resistance of the thin film is recorded as a function of time. It is found that there is decrease in resistance of the sensing element in presence of H_2S gas.

When the H_2S gas is injected into the unit, the resistance of the element decreases rapidly and reaches a constant value in a short course of time. When the element is exposed to air by opening the cap and keeping bottle quite away the resistance of the element increases and reaches its initial value. The decrease in resistance with respect to time is recorded when exposed to H_2S gas and the increase in resistance with respect to time is recorded when exposed to open air. This procedure is repeated for several times and obtained reproducibility curves are discussed further in this section.

The as deposited SnO_2 films using chemical spray pyrolysis were uniform and almost transparent on glass slide. Already there are several techniques to synthesize SnO_2 thin films, such as aerosol technique [11], controlled solid – vapor process [12], laser ablation technique [13], wet chemical rout [13], thermal evaporation [14] etc. However among all the available techniques the chemical spray pyrolysis is one of the simple, versatile and easy techniques that do not require vacuum and other special accessories. Moreover it gives good quality films with uniform thickness [15, 16]

The films were used for X-ray analysis, the recorded powder diffraction pattern is shown in figure (4). The peaks are observed at 2θ =26.61˚, 33.89˚, 37.95˚, 51.78˚ and 54.75˚ corresponds to the Miller indices (110), (101), (200), (211) and (220) respectively. This reveals that the observed peaks are comparable to the characteristic peaks of tetragonal phase of SnO_2 (JCPDS Card No. 41-1445). The broad peak in the XRD pattern indicates that the prepared SnO_2 films are having small particle size. So the small size of the particles would lead to high surface to volume ratio leading to high density of surface atoms and hence can be a useful material for gas sensing applications. From the pattern, the average crystallite size is estimated using Debye Scherer formula $d = k \lambda / \beta \cos \theta$ where k is shape factor which has value about 0.9, λ is the X ray wave length, β is the full width half maxima (fwhm) and θ is the Bragg angle. The particle size is estimated from fwhm of broad peak is found to be less than 5 nm and θ. And the particle size is found to be less than 5 nm. There are many reports on the chemical spray by others of SnO_2 film due to the potential application in various areas. Th. Becker et al. have reported the grain size of SnO_2 films to be 50 nm, [17] U Nerle et al. have reported the crystallite size of SnO_2 film deposited using spray technique is about 50 nm to 70 nm [18]. Soumen Das et al have reported the diameter of the nano particles to be 10 nm [19]. In the present study the XRD peak is broad which indicate the small size of the particles the size of the particle. This gives large density of surface dangling bonds on the surface of film, which is more suited for gas sensing applications that helps in obtaining the improved gas sensitivity of a material.

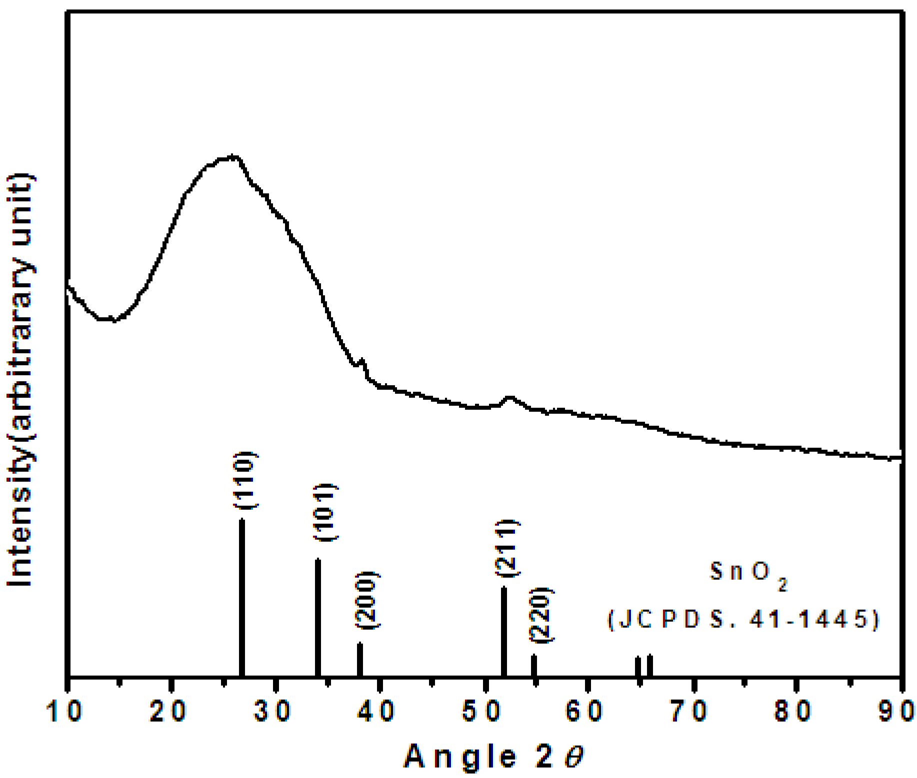

Figure 4: X-ray powder pattern of spray deposited SnO2 film on glass slide.

The films were used to study the optical absorbance and transmittance in the wavelength region from 200-1100 nm, a typical curve is shown in figure (5). The absorption curve shows its optical gap transition close to 350 nm which corresponds to the optical band gap (E_g) of about 3.5 eV Figure (6) shows the transmittance behavior of the same film. It is observed that the films have highest transmittance that is close to 80%. T Serin et al have carried out optical absorption studies and reported optical band gap of SnO_2 films in the range 3.94-3.96 eV [17]. In the present work absorption curve of intrinsic SnO_2 shows its optical gap transition close to 350 nm corresponding to the band gap (E_g) equal to 3.5 eV , similar results were reported by U Nerle et al. and it has almost flat base prior to absorption edge [17].

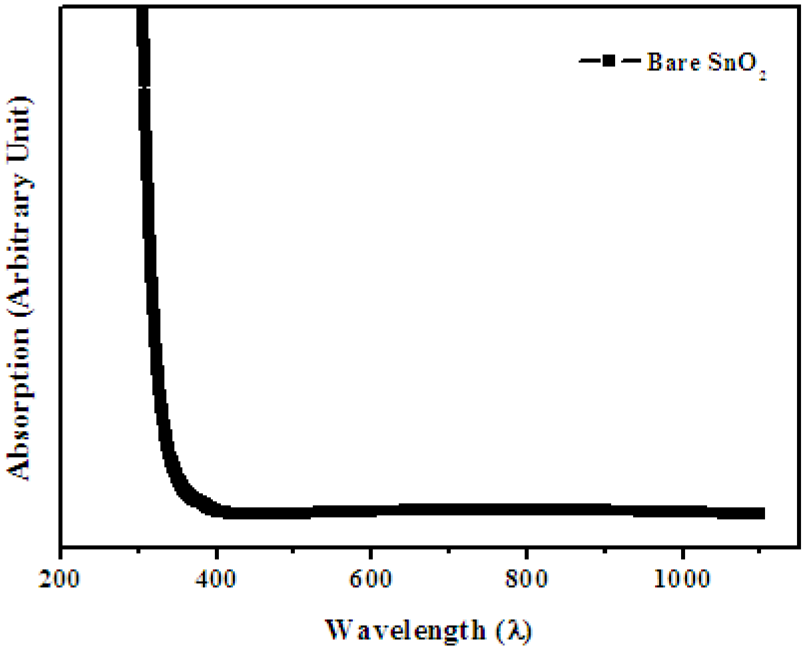

Figure 5: Optical absorption of SnO2 thin film recorded between 300 nm to 1100 nm.

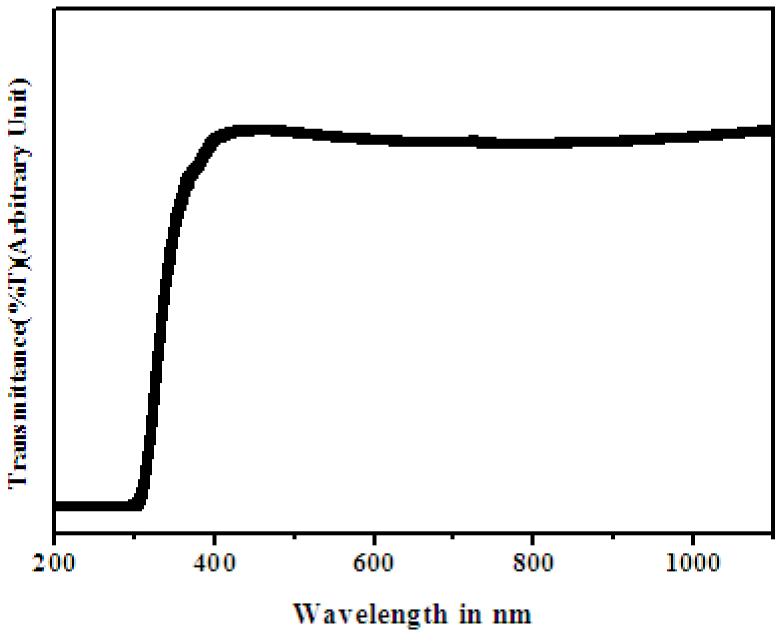

Figure 6: Optical transmittance spectrum of SnO2 thin film of glass slide.

Further, the electrical conductivity is measured by measuring current voltage curve of these films in two probe configuration. Figure (7) shows the I-V characteristics of the SnO_2 thin film. The curve shows the pseudo ohmic behavior. From the slope of the curve the electrical resistance is estimated that is about 3 kΩ

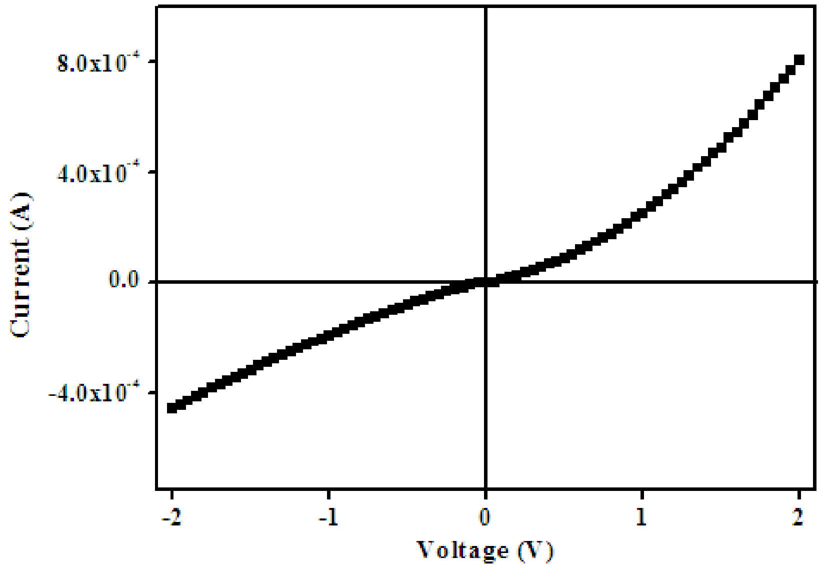

Figure 7: Current – Voltage (I–V) curve of SnO2 thin film.

The gas sensing characteristics of thin film of SnO_2 were investigated. Sensitivity S is defined as the ratio of effective change in resistance for the test gas to the original value of the resistance in air. It is given by $S=\frac{(Ro-Rg)}{Ro}$ x100%. Where R_g is the sensor resistance in the presence of the test gas and R_o is the sensor resistance in dry air, measured at that temperature. In our measurements, first the temperature of the oven is set to a particular value by applying 12.5 V to the heater to produce stable temperature 110˚C and the resistance of the sensor in air is recorded. A known amount of H_2S gas is injected into the bottle and the cap is locked tightly the fall in resistance of the sensor with respect to time is recorded. When once the minimum constant resistance value is reached the bottle is opened and the sensor is exposed to open air. Now the increase in the resistance with respect to time is recorded. Similar procedure was repeated for several times and it is observed that the recorded behavior is quite reproducible. Figure (8) shows the sensing characteristics of SnO_2 thin film measured at 110˚ C when exposed to a fixed amount of H_2S gas. Upon exposure to the H_2S gas the film resistance is seen to decrease. From the sensing characteristics it is found that the response time is less, where as the recovery time is slightly more. The reproducibility is very good. G Patil et al. have tested SnO_2 thin films for various gases at different operating temperatures ranging from 50˚C to 450˚C and found the SnO_2 films deposited by spray technique shown maximum sensitivity to H_2S gas at 100˚C [19] . Comparision of gas sensing characteristics of SnO_2 based gas sensors by others [20] (nanofibres, nanocrystals, thin films, submicrotubes, etc) to H_2S for different concentration is made and established that SnO_2 thin films show maximum response to H_2S at an operating temperature of 100˚C for 80 ppm. In present study we have recorded the gas sensor response using our miniaturized setup to sense H_2S gas at 110˚C and the sensor response curves are shown in figure (8)

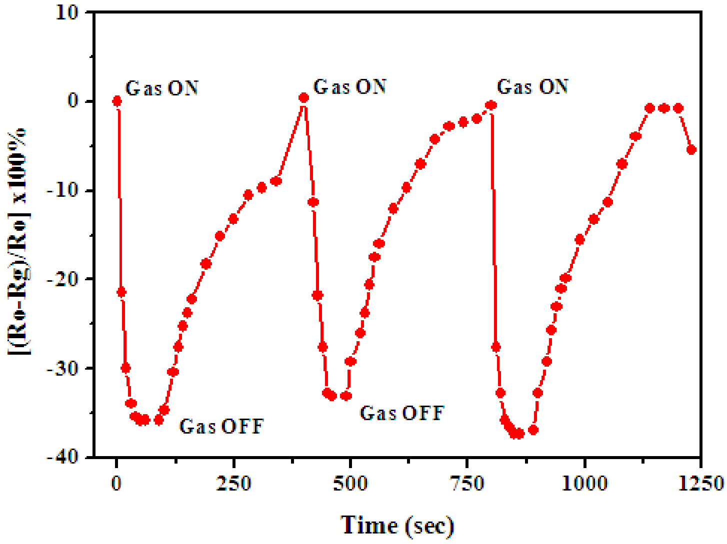

Figure 8: Gas response of SnO2 film loaded in prepared setup @ 110˚C recorded for H2S

IV. Conclusions

The setup has been proposed to measure the gas response of different gases in terms of variation in electrical resistance of sensing element. It is quite simple, portable, cost effective, reliable and sensitive to detect H_2S gas. The setup is able to measure sensing property of the samples at different temperatures. The workability of the present setup is clearly shown in the gas response characteristic curve that is electrical resistance verses time. The present setup has been used successfully to study the sensing property of SnO_2 films of nano grain size for H_2S gas. It can be easily extended to the other sensing materials and also for other gases of interest.

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