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Effect of Firing Temperature on Humidity Sensing Properties of SnO_2 Thick Film Resistor

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Abstract: Thick films of SnO_2 were prepared using standard screen printing technique. The films were dried and fired at different temperatures. Tin-oxide is an n-type wide band gap semiconductor, whose resistance is described as a function of relative humidity. An increasing firing temperature on SnO_2 film increases the sensitivity to humidity. The parameters such as sensitivity, response times and hysteresis of the SnO_2 film sensors have been evaluated. The thick films were characterized by XRD, SEM and EDAX and grain size, composition of elements, relative phases are obtained. *Copyright © 2009 IFSA.*

Keywords: Thick film resistors, Tin oxide, Humidity, Sensitivity, XRD

1. Introduction

Recent developments in automated systems have made ever-increasing demands for various kinds of physical and chemical sensors. As humidity is a very common parameter in our environment, measurements and /or control of humidity are important not only for human comfort but also for a broad spectrum of industries and technologies. so for many methods of sensing humidity and moisture have been introduced, and almost all of them are still in use. Sensors of great variety of application have been developed, numerous materials have been utilized for humidity sensing of which the metal oxides that are physically and chemically stable, have been extensively used at both room and elevated temperature [1-3]. The sensing materials are roughly classified in to three groups, i.e. electrolytes, organic polymers and porous ceramics. In spite of the differences in materials, most sensors utilize a common phenomenon, that is physical or chemical adsorption (or absorption) of water molecules [4].

Among the different types of humidity sensors, those based on electrical properties such as resistance and capacitance is best suited to modern automatic control systems. Various sensors for humidity have been introduced [4, 5], some recent ones [4] of which are of miniature solid state type. Different materials are used for these solid state sensors [4-11]. The semiconductor humidity sensors are characterized by their conductance change due to chemical absorption of water molecules, thus resulting in electron conductivity. These types of humidity sensors are now under development by several researchers [4, 8, 11-14]. Stannic Oxide (SnO_2) is an n-type wide band gap semiconductor of which electrons are majority carrier, and electron concentration of conduction band affected by gas. The resistance of SnO_2 thick film resistors is reported to be a function of relative humidity (RH) [8]. It is widely used as a gas sensor for inflammable gases [11, 15]. Sensors based on change in resistance and capacitance is preferred. In this paper we report the resistance type humidity sensing studies of SnO_2 thick film resistors fabricated by standard screen printing technique on alumina substrate. [16, 17]

2. Experimental Methods

Tin oxides films were prepared on alumina substrates by using screen printing technique [18-20]. The SnO_2 powder (99.99 % Pure, AR grade, Loba Chemicals) was weighed and calcinated in air at 400 ^{O}C (± 2 ^{O}C) for 2 hours. The calcinated SnO_2 powder was crushed and mixed thoroughly with glass frit as permanent binder and ethyl cellulose as a temporary binder. The mixture was then mixed with butyl carbitol acetate as a vehicle to make the paste. The paste was then screen printed onto the surface of alumina substrate. After screen printing the films were dried under IR-lamp for 1 hour and then fired at 580 ^{O}C, 680 ^{O}C and 780 ^{O}C (±2 ^{O}C) for 30 minutes. The SnO_2 Paste are found to have good printability and thixotropic.

The thickness of the SnO_2 thick film resistors was measured by using Taylor-Hobson (Taly-step UK) system. The thickness of the films was observed in the range of 10 μm–20 μm.

Using X-ray diffraction (Miniflex Model, Rigaku, Japan, DMAX-500) analysis from 20-80^{0}, 2θ was carried out to examine the final compositions of the SnO_2 films samples. The average grain size of tin oxide thick film samples were calculated by using the Seherrer formula [21]

$$D = \frac{0.9\lambda}{\beta \cos\theta}, \quad (1)$$

where D is the average grain size, λ =1.542 AU (X-ray wavelength), and β is the peak FWHM in radiation and θ is diffraction peak position. The microstructure and elemental composition of the films were analyzed using a scanning electron microscope [SEM model JEOL 6300 (LA) Germany] coupled with an energy dispersive spectrometer (EDS JEOL, JED-2300, Germany).

2.1. Humidity Measurement System

Fig. 1. shows the schematic diagram of the closed humidity system used for measurement of relative humidity (RH). It consists of a closed flask (total volume 500 ml) with two necks for inserting thermometers and the thick film sensor. The flask is partially filled with water and kept in a cooling container [3, 17, 22, 23].

The % *RH* of the air in the system is given by [24]

$$RH\% = \frac{E_W\,(T_W)}{E_W\,(T_S)} \times 100 \ , \tag{2}$$

where $E_w(T_w)$ is the saturated vapour pressure at the temperature of the water bath $T(_w)$ and $E_w(T_S)$ is the saturated vapour pressure at the temperature of thick film sensor element (T_S) (connected at a time). The RH is adjusted by changing the temperature of the humidity system. The thick film sensor is either kept and stabilized inside the system (for sensitivity and hysteresis measurement) or is to be inserted in it within a second (for response time measurement).

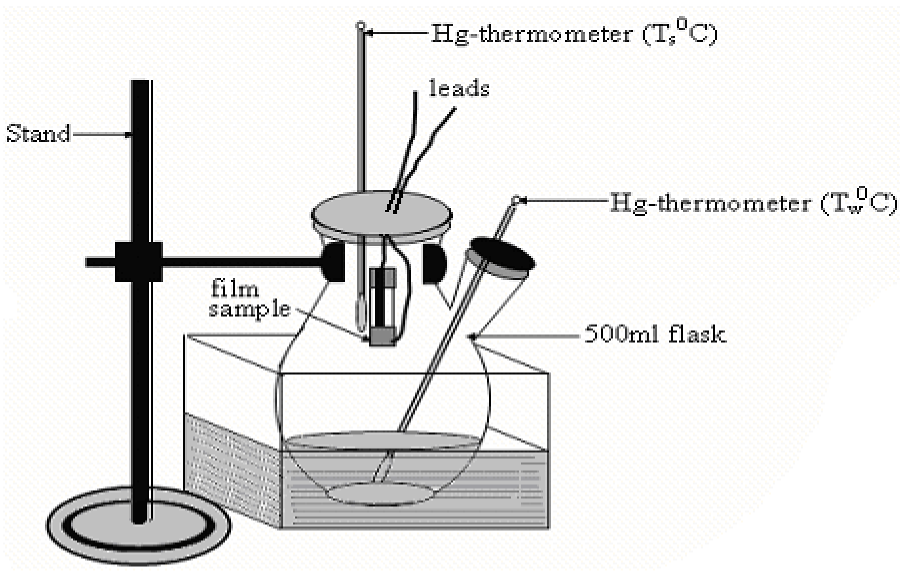


Fig. 1. Humidity measurement system.

The D.C resistance of the thick films was measured by using half bridge method a function of temperature described elsewhere [25]. The films were set a temperature-controlled (±1 ^{O}C) atmosphere. A load resistor R_L was connected in series with the thick film and a D.C voltage was applied to the circuit. The values of the thick film resistor were obtained by measuring output voltage across the load resistor RL. A chromel-alumel thermocouple was used to indicate the operating temperature (±1 ^{O}C). The thick film resistance, (R_f) was calculated by the relation

$$R_f = [(V_{app.} / V_O)\text{-}1]\ R_L \ ,$$

where $V_{app.}$ is the applied voltage;
V_O is the voltage across load resistor $R_{L.}$

2.1.1. Sensitivity

It is defined as the change in resistance due to a change in %RH with respect to the initial resistance R_a, i.e. % S= [(ΔR/R_a)/RH] x100; It is measured by keeping the sample at different RH values at stabilized condition.

2.1.2. Response Time

Response time is the time taken by the thick film sensor to attain 90 % of maximum value of resistance upon exposure to humidity and recovery time is the time taken by the film sensor to attain 10 % of maximum value of resistance.

For measuring the response and reverse response (recovery) time of SnO_2 thick film sensors, two adjacent humidity systems, one at 20 %RH and the other at 80 %RH, respectively, are used. The stabilized resistance is measured in one system, and then the sample is transferred to the other system within a second. Then the variation in resistance is measured with respect to time to study the response time.

2.1.3. Hysteresis

For hysteresis measurements with the sample kept inside the system, the humidity in the system is decreased initially by cooling it down and the corresponding change in resistance is measured. Within 60 minutes the system achieves the minimum humidity level (20 %).To increase the humidity, the system temperature is increased in an open atmosphere without any external heat energy (within 40 minutes). It must be noted that the system response time is very large with respect to the response time of the sensor.

After completing one cycle, the system is allowed to equilibrate (to room temperature and pressure) for 50-60 minute and then the samples are cycled again.

3. Results

3.1. X-ray Diffraction Studies

X-ray diffraction studies of the thick films were carried out using X-ray diffractometer (Miniflex model Rikagu, Japan). Fig. 2. shows the X-ray diffraction pattern of SnO_2 thick films fired at 580, 680 and 780 ^{O}C plotted in the range of 20-80^{O} (2θ) versus intensity having several peaks of tin oxide phases indicating polycrystalline nature. The observed peaks match well with the reported ASTM data of tin-oxide confirming the polycrystalline nature of the film material. The average grain size was determined by using Scherrer formula and was estimated to be 37, 41 and 54 nm (± 2nm) for the firing temperatures of 580, 680 and 780^{O}C respectively. Table 1 illustrates the percentage of relative phases of tin oxide present in thick film samples.

Table 1. Presence of % relative phases of the SnO_2.

Firing temperature (^{O}C)	**% relative presence of phases**					
	T (SnO_2)	O (SnO_2)	Sn_3O_4	Sn_2O_3	Al_2O_3	Average grain size (±2nm)
580	32.00	34.82	22.00	1.937	9.248	37.00
680	32.98	26.92	19.139	2.172	18.79	41.00
780	34.38	29.38	17.07	2.928	16.24	54.00

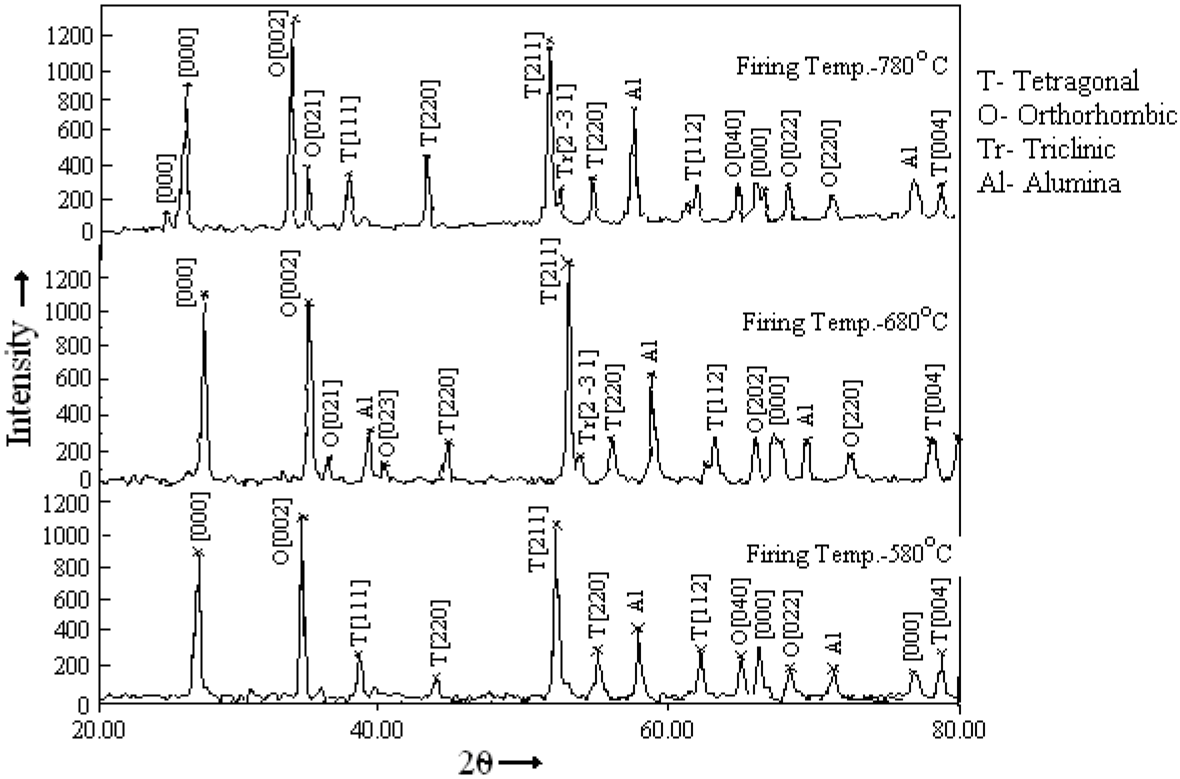


Fig. 2. XRD patterns of SnO_2 thick film samples fired at (a) 580; (b) 680; and (c) 780°C.

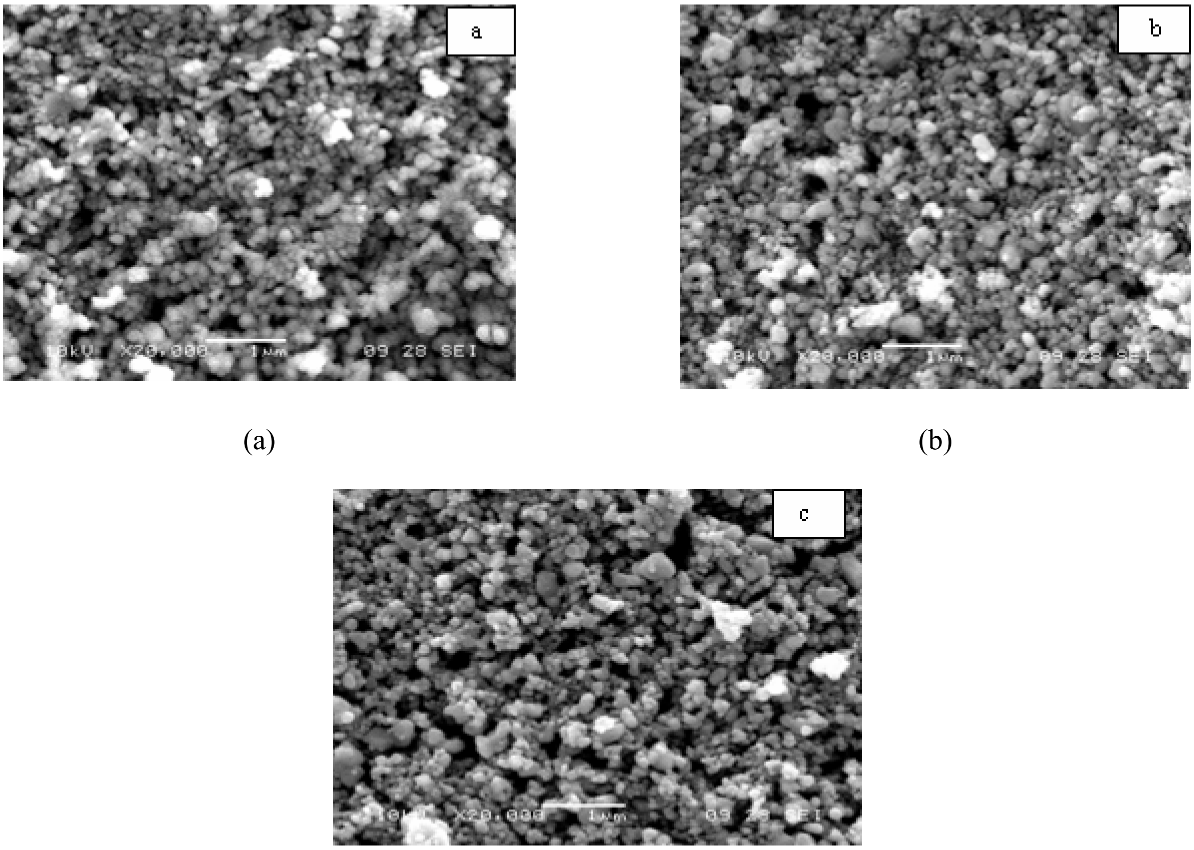
3.2. Microstructural and Elemental Analysis

Scanning electron microscopic (SEM) studies were carried out using SEM model JEOL 6300 (LA) Germany. Fig. 3. depicts SEM images of SnO_2 thick films fired at 580, 680 and 780 ^{O}C. The micrograms show uniform polycrystalline nature of the films indicating increased grain size with firing temperature. The micrograms show voids between the particles basically due to evaporation of the organic solvent during the firing of the films.

The elemental analysis of the films was carried out using EDS (JEOL, JED-2300, Germany). The constituent elements of SnO_2 thick films are tin and oxygen and no other impurity elements are present in the composition. Also from the EDS spectra it is seen that wt% and at% are nearly matched. Table 2 illustrates the quantitative elemental analysis.

Table 2. Quantitative elemental analysis.

Sample	**Wt %**	**Firing temperature(^{O}C)**					
		580		680		780	
		Sn	O	Sn	O	Sn	O
SnO_2	Mass %	88.22	11.78	89.69	10.31	89.57	10.43
	At %	50.23	49.77	53.97	46.03	53.65	46.35
	Error %	0.48	0.11	0.41	0.09	0.44	0.10



(a) (b)

(c)

Fig. 3. Scanning Electron Micrograms of SnO_2 thick film samples fired at (a) 580 (b) 680 and (c) 780^oC.

3.3. Humidity Response

3.3.1. Sensitivity

Fig. 4. shows the humidity sensing response curves for SnO_2 films fired at different temperatures. The response of SnO_2 film resistor fired at temperature 580 ^OC shows that as RH increases, resistance decreases linearly up to 50 %RH and then decreases slightly up to 80 %RH and show average sensitivity of 0.85. Curve for thick film fired at 680 ^OC shows that as %RH increases fast decrease in resistance up to 30 % RH and slowly up to 52 % RH and finally saturates with average sensitivity of 1.40. Curve for SnO_2 film sample fired at 780 ^OC shows rapid decrease in resistance with increase in relative humidity up to 55 % RH and then decreases slightly up to 60 %RH, then finally saturates with average sensitivity of 5.85.

3.3.2. Response Time

The response time is the time taken by sensor to sense a humidity change from lower RH (20%) to higher RH (80 %) and the reverse response time is the time taken by the sensor to sense a humidity change from higher RH (80 %) to lower RH (20 %). Fig. 5. shows a plot of [(Rrh-Ra)/Ra] % versus time for the SnO_2 thick films fired at 580, 680 and 780^oC. From Fig. 5 (a). it is observed that the response time from lower RH (20 %) to higher RH (80 %) is 140 sec for the thick film fired at 580 ^OC, 80 sec for 680 ^OC and 75 sec for 780 ^OC. For the reverse change (80 %RH-20 %RH) in Fig. 5(b), the response time is 2 min for films fired at 580 ^OC, for 10 min for 680 ^OC and 3 min for 780 ^OC.

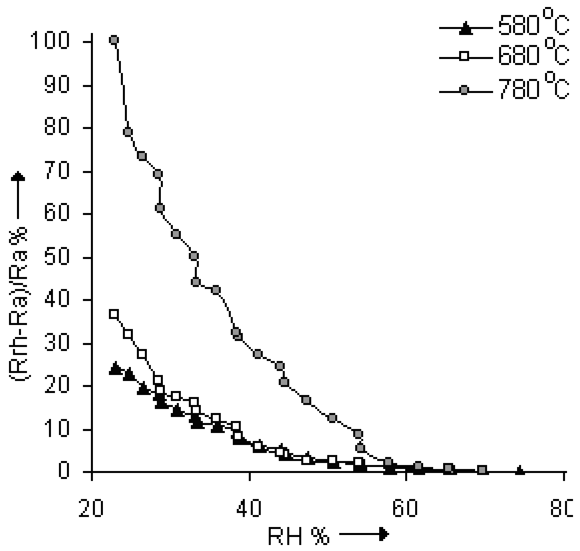
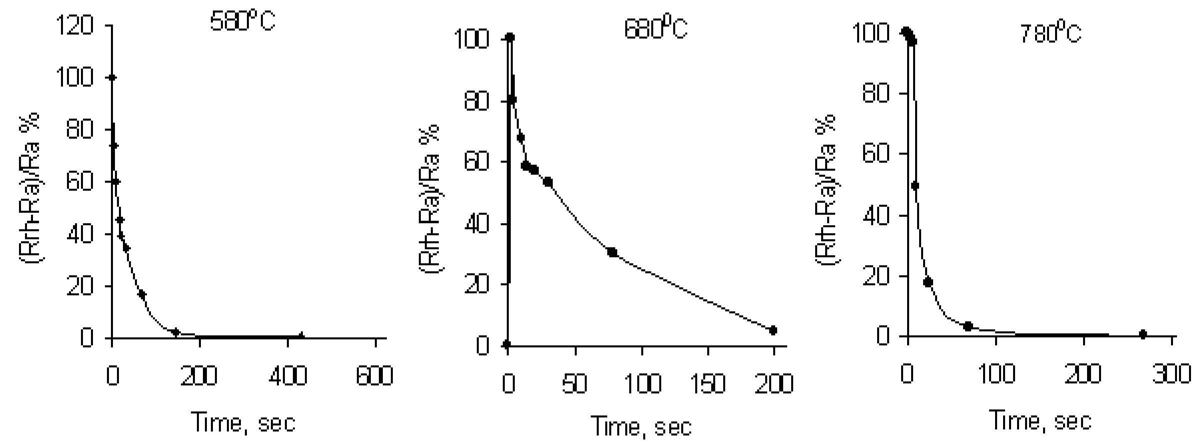
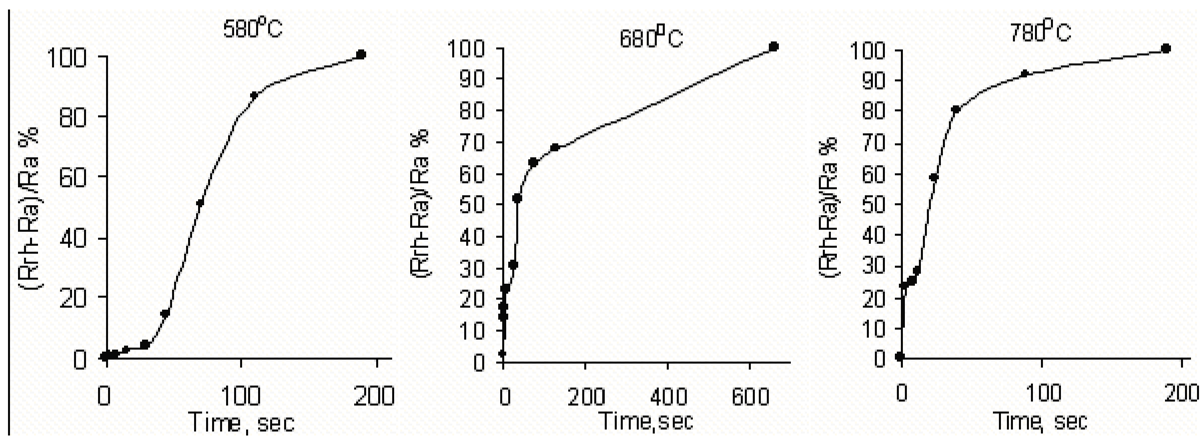


Fig. 4. Plot of [(R_{rh}-R_a)/R_a)] % versus %RH for SnO_2 thick film samples fired at 580, 680 and 780 °C.



(a) Response time (Low to High transition)



(b) Response time (High to Low transition).

Fig. 5. Plots of [(Rrh-Ra)/Ra] % vs time for the SnO_2 thick films fired at 580, 680 and 780°C.

3.3.3. Hysteresis

It is defined as the maximum percentage difference in the two curves at any RH%, when the sample is exposed to decreasing and increasing humidity cycle successively, with respect to RH%. Fig. 6. gives typical cycling hysteresis response of SnO_2 thick film samples. In this case, the film samples fired at 580, 680 and 780 ^{O}C gives maximum percentage deviation 0.125, 0.244 and 0.335 in two curves particularly for 36 %, 46 % and 46 % RH respectively.

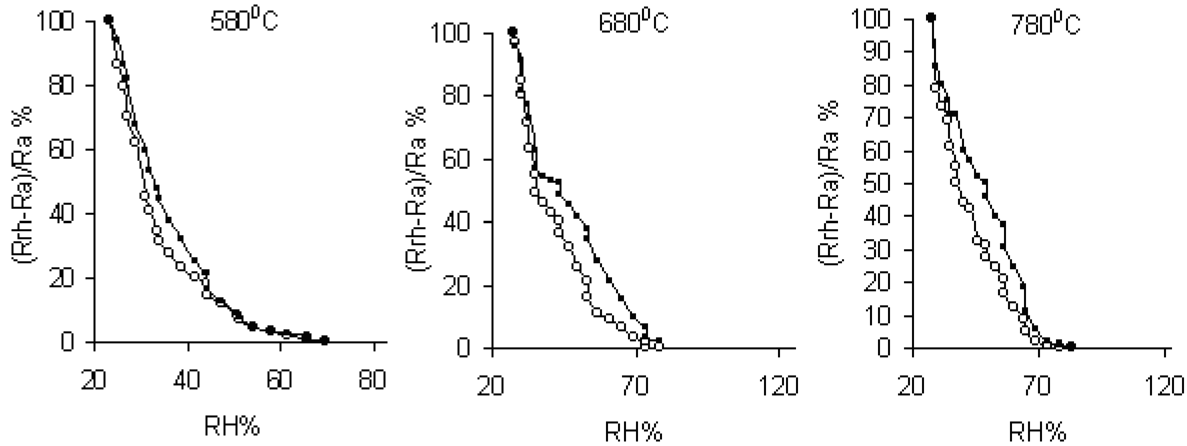


Fig. 6. Hysteresis curves for three samples fired at 580, 680 and 780 ^{o}C.

4. Discussions

In Order to understand the phases in SnO_2 film samples, the X-ray diffraction study was undertaken. Fig. 2. shows an XRD patterns of SnO_2 film samples fired at 580, 680 and 780 ^{O}C, plotted in the range 20-80^{0} (2θ) verses intensity. The XRD pattern consists of several peaks of tin oxide phases indicating polycrystalline nature. The tetragonal, orthorhombic and triclinic phases were observed. The observed peaks match well with the reported ASTM data of tin-oxide, confirming the polycrystalline nature. The higher peak intensities of an XRD pattern is due to the better crystallinity and bigger grain size can be attributed to the agglomeration of particles. The average crystallite size is increasing with increase of firing temperature**.** The total number of peaks increases with increase in firing temperature. Fig. 3 shows the surface morphology of SnO_2 films observed by Scanning Electron microscope (SEM). 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. It can be seen from Fig. 3 (a) to (c) that the grain size increased with increase in firing temperature. Another fact observed is of some segretion in film fired at higher temperature. The micrograph also shows the presence of more agglomeration in the film samples. The EDS spectrum indicates that the wt.% and at.% are nearly matched from EDS spectra it is seen that the major peaks are for tin and oxygen and no other impurity elements are present in the film sample.

The Fig. 4. shows decrease in relative resistance of SnO_2 film with increase in RH% is due to the increase thickness of the absorbed H_2O film on the surface of SnO_2 film resistor [4, 9, 23, 26]. The decrease in resistance with increase in humidity is also due to the dissociation of H_2O molecules [27], into proton (or another H_3O^+) and hydroxyl groups (OH^-). H_3O^+ is responsible for the reversible reaction. These hydroxyl groups combine with surface oxygen ions forming water and releases back an electron to the material. Thus the conductance of n-type semiconductor increases (resistance decreases) with increase in humidity [4, 28].

The physisorbed water dissociates because of high electrostatic field in the chemisorbed layer. The possible reaction can be written as

$$2H_2O \rightarrow H_3O^+ + OH^- \tag{3}$$

The response of SnO_2 film resistor fired at temperature 580 ^OC shows that as RH increases, resistance decreases linearly 50 %RH, then decreases slightly up to 80 %RH. Response of SnO_2 thick film fired at 680 ^OC shows that as %RH increases resistance decreases rapidly up to 30 %RH and slowly up to 52% RH and saturates at constant value. Curve for SnO_2 film Sample fired at 780 ^OC shows that as %RH increases, the resistance decreases rapidly up to 55% RH and then decreases slightly up to 60 %RH, and finally saturates. The maximum sensitivity of 5.85 for the film sample fired at 780 ^OC is due to presence of large number of pores in the film. The nonlinearity in response at lower RH level is probably due to single layer coverage of the water molecules (chemisorbed water) on the surface of the film [9, 23, 29]. This result in smaller change in resistance with RH, as shown by the lower end of the response curves. Similarly at high RH, the capillary condensation effects are expected to play a significant role in getting non–linearity by saturation. [9, 29]. However, this effect of such type of interaction is observed in case of film sample fired at 780 ^OC.

Response times of typical SnO_2 thick film resistors are given in Fig. 5. (a) and (b). The film resistor gives response to rising humidity from 20 to 80 %RH (Low to High). It is observed that the response time to a transition from low to high RH is of the order of 140 sec for the thick film fired at 580 ^OC, 80 sec for 680 ^OC and 75 sec for 780 ^OC. For the reverse change (80-20) %RH, it is 2 min for films fired at 580 ^OC, for 680 ^OC 10 min and for 780 ^OC 3 min. This may be due to the closed humidity system used. Desorption via evaporation is a ventilation dependent process. The response time can be decreased by reducing the size of film sample. The thermal mass of the samples is very small with as compared to that of the humidity chamber.

Typical cycling hysteresis response of the three SnO_2 thick film resistors fired at temperatures of 580,680 and 780 ^OC is given in Fig. 6.The adsorption-desorption cycles of thick film resistors show hysteresis. The hysteresis in humidity sensors is due to the slow desorption of chemisorbed water molecules (at lower RH) and capillary condensation (at higher RH). The hysteresis in humidity sensors can be explained with the help of Kelvin effect [14], which prevents or retards the desorption of water, condensed in the pores of SnO_2 during adsorption process as it forms a concave meniscus. The vapour pressure of water over the concave meniscus is much lower than that in surrounding atmosphere, hence desorption can take place only when external vapour pressure becomes less than vapour pressure above the meniscus which mainly occurs in the range of 85 to 50 % RH [9].

5. Conclusion

Following conclusions can be drawn from the experimental results.

1. The D.C. resistance of SnO_2 thick films decreases with increasing relative humidity.
2. The SnO_2 films showed extremely high sensitivity to humidity at higher firing temperatures.
3. The sensing mechanism of the SnO_2 thick film resistors was the surface controlled mechanism (adsorption/desorption).
4. The present SnO_2 thick film device has advantages of ease of fabrication, small size, high sensitivity, low hysteresis and response time.

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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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