Advancement in Structural, Electrical and Gas Sensing Performance of Surface Modified SnO_2 Metal Oxides

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Abstract : Metal oxide based solid state gas sensors are the best selection to the development of commercial gas sensors for a wide range of applications. The great interest of industrial and domestic solid state gas sensors comes from their versatile advantages like portable size, high sensitivity in detecting very low concentrations (ppm, ppb or sub ppb level). Tin Oxide (SnO_2) powder is one such challenging material for the fabrication of gas sensors. Bulk Tin Oxide (SnO_2) powder in the form of thick film was observed to be less sensitive to the polluting gases. So, synthesized nanostructured SnO_2 powder was fabricated by simple screen printing technique followed by the calcinations at 500°C for 1 hr. Thick films of pure nanostructured SnO_2 powder were surface activated by dipping them into 0.01 M aqueous solution of Strontium Chloride and / or Bismuth Chloride for different intervals of time followed by calcinations at 500°C for 30 min. The films exhibit the semiconducting nature due to non-stoichiometry and respond to various gases. Optimizing the particular conditions, the thick films can be used for gas sensing to detect hazardous gases, viz. H_2, H_2S, Cl_2, NH_3, C_2H_5OH, LPG, etc. The surface morphology, chemical composition, crystal structure, electrical and thermal properties of the bulk and nanostructured SnO_2 have been investigated by Field Emission Scanning Electron Microscope (FE-SEM), Energy Dispersive Analysis by X-rays (E-DAX), X-Ray Diffraction (XRD), etc.

Keywords **- Bulk and Nanostructured SnO_2, Synthesis, Thick Films, Polluting gases, etc.**

I. INTRODUCTION

The concentration of the gas in the environment which produces an undesirable and disastrous change in the physical, chemical or biological characteristics of air, soil and water that can harmfully affect the living beings leads to a pollution. Heavy industrialization, uncontrolled urbanization and careless application of technology can cause pollution [1-6]. Air pollution is a major threat for modern society. Current burning issues are global warming, the cruelest episodes like Bhopal gas tragedy, etc. are the effects of air pollution. Along with this, some domestic threats are also occurring all over the world. The main culprits behind all such hazards are the toxic, inflammable and explosive gases. Gases play the key role in many industrial or domestic activities. In the last twenty years, the demand for gas detection and monitoring has increased. SnO_2 along with additives, viz. Bi_2O_3 and SrO_2 are the most easily available and low cost materials. These materials are found suitable for the gas sensing applications.

II. OBJECTIVES

- To synthesize the nanostructured semiconducting oxide by one of the simplest and cheapest process known as disc type ultrasonicated microwave assisted centrifuge technique.
- To prepare the thick films of pure and modified nanostructured SnO_2 by screen printing technique, one of the simplest and low cost techniques.
- To ensure longer life by maintaining proper thixotropy and rheology of the thick films.
- To analyze the synthesized pure and modified materials by different characterization techniques.
- To achieve a suitable surface modification by dipping the thick films of SnO_2 into SrO_2 and / or Bi_2O_3 for enhancing the gas response and selectivity.
- To investigate the electrical and gas sensing performance of pure and modified SnO_2 thick films.
- To investigate and modify the response and recovery profile of pure and modified SnO_2 thick films.

III. LITERATURE REVIEW

Table 1: Literature Review

Sr. No.	Material	Technique	Gas / Investigation	Year	Ref. No.
1	SnO_2	Thick films	H_2, CO_2	1996	7
2	SnO_2	Thick films	H_2	2005	8
3	MoO_3, SnO_2	Thick films	NO_2, C_2H_5OH, H_2	2010	9
4	SnO_2, RuO_2	Thick films	LPG	2007	10
5	SnO_2, CuO	Thick films	H_2S	2006	11
6	SnO_2, Sr	Thick films	LPG	2007	12
7	SnO_2	Thick films	CO, C_2H_5OH, CH_4	2004	13
8	SnO_2	Thick films	CO	2009	14
9	SnO_2	Thick films	NH_3, C_2H_5OH, etc.	2009	15
10	SnO_2	Thick films	H_2	2006	16
11	SnO_2, Ga_2O_3	Thick films	Hazardous gases	2009	17
12	SnO_2, Pt	Thick films	CO, NH_3, C_6H_6, etc.	2005	18
13	SnO_2, ZrO_2, etc.	Thick films	H_2, CO, NO, NO_2	2006	19
14	SnO_2	Thick films	Hydrocarbons	2007	20
15	SnO_2	Thick films	Automobile exhaust	2006	21
16	SnO_2	Thick films	CO, CH_4	2006	22
17	SnO_2, CuO	Thick films	H_2S	2014	23
18	SnO_2, WO_3, etc.	Thick films	NH_3	1997	24

IV. MATERIALS USED

4.1 Tin Oxide (Stannic Oxide, SnO_2)

Tin is principally found in the ore cassiterite (tin oxide). It is obtained commercially by reducing the ore with coal in a furnace. SnO_2 is a wide band gap semiconducting oxide having energy gap of 3.6 eV. It crystallizes in the rutile structure. The unit cell of tin oxide contains two atoms of Sn and four atoms of O_2. It has tetragonal symmetry. Each tin atom is surrounded by distorted octahedron of six oxygen atoms and each oxygen atom has three tin nearest neighbors at the corners of an almost equilateral triangle. SnO_2 is used as a polishing powder and is sometimes known as putty powder. Tin oxide is used for ceramics and gas sensors. In gas sensors, the sensor area is heated to a constant temperature (few hundreds of degrees Celsius) and in the presence of a test gas, the electrical resistivity drops. For carbon monoxide detection, SnO_2 wires are commonly used.

4.2 Strontium Oxide (SrO_2)

Strontium is a chemical element with symbol Sr and atomic number 38. Strontium is an alkaline earth soft metal in silver white or yellowish color. When it is exposed to air, it forms a dark oxide layer. Strontium occurs naturally found mainly in the minerals celestite and strontianite. The properties of strontium are intermediate between and similar to those of its group neighbors, calcium and barium. Strontium is harder than barium and softer than calcium. The melting point of strontium is 777°C and boiling point is 1377°C. The density of strontium is 2.64 gm/cc. When Strontium metal burn in air, it will result in strontium nitride as well as in strontium oxide. However, it does not react with nitrogen below 380°C. It spontaneously forms only the oxide at room temperature. Strontium is widely used in fireworks and flares. Also, it is used for the production of ferrite magnets and in Zinc refineries.

4.3. Bismuth Oxide (Bi_2O_3)

Bismuth occurs as silver pink, and white in its original form as with an iridescent oxide tarnish showing many colors from yellow to blue. The Bismuth oxide has base centered monoclinic crystal structure. Bismuth burns with a blue flame and its oxide forms yellow fumes, when burnt in oxygen. It is less toxic as compared with its other neighboring elements in the periodic table, viz. lead, antimony and polonium. Bismuth occurs as the native metal, and in ores such as bismuthinite and bismite. Bismuth oxide is a high-density metal. As Bismuth is a brittle metal, it is usually mixed with other metals to make it useful. Bismuth can be used in fire detectors and extinguishers, solders and electric fuses when it is mixed with low melting point metals, viz. tin or cadmium. Bismuth oxide is also used as cosmetics and paints.

V. EXPERIMENTAL

5.1 Material Synthesis

Fig. 1 (a): Disc type ultrasonicator **Fig. 1 (b):** Microwave assisted centrifuge technique

The nanomaterials were synthesized in the laboratory by disc type ultra-sonicated microwave treatment followed centrifuge technique [25]. Fig. 1 (a) shows disc type ultrasonicator and Fig. 1 (b) shows microwave assisted centrifuge technique. An initial aqueous-alcohol solution was prepared from distilled water and propylene glycol in the ratio of 1:1, by volume. This solution was then mixed with aqueous solution of tin chloride with the alcohol to water ratio as 1:1. The special arrangement was made to add drop wise aqueous ammonia (0.1ml / min.) with constant stirring until the optimum pH of solution becomes 7.9 - 8.9. After complete precipitation, the hydroxide was washed with distilled water until chloride ions were not detected by $AgNO_3$ solution. Then the washed precipitate in a glass beaker was placed in a microwave oven for 10 minutes with on-off cycles, periodically. The dried precipitate was ground by agate pestle-mortar and annealed in a muffle furnace at 500°C for 12 h.

5.2 Thick Film Fabrication Technique

Thick film fabrication technique using screen printing mechanism is the most suitable, simple and economical method in the fabrication of sensors. The screen is held at about few millimeters above the substrate on a printing set up board (Fig. 2). The required thixotropic paste is kept on the top surface of stencil and squeegee pushes the paste and poured through the pores while it moves from one end to the other. The sensor patterns printed on the glass substrates are in rectangular form. The length of the sensor pattern kept was 9 mm and the width 4 mm. The desired amounts of samples are prepared by repeating the above procedure. These samples are then subjected to a temperature in the range of 70-130°C for drying under an IR lamp. Next, curing is done in order to avoid the blistering of the film. It has two steps: removal of organic binder and high temperature curing / firing. In the first step, the residues of organic binder (even after drying process) are eliminated by heating at low temperature. In the second step, the film is subjected to a furnace under controlled conditions. The chemical reactions that take place, give the required electrical and physical properties of the film.

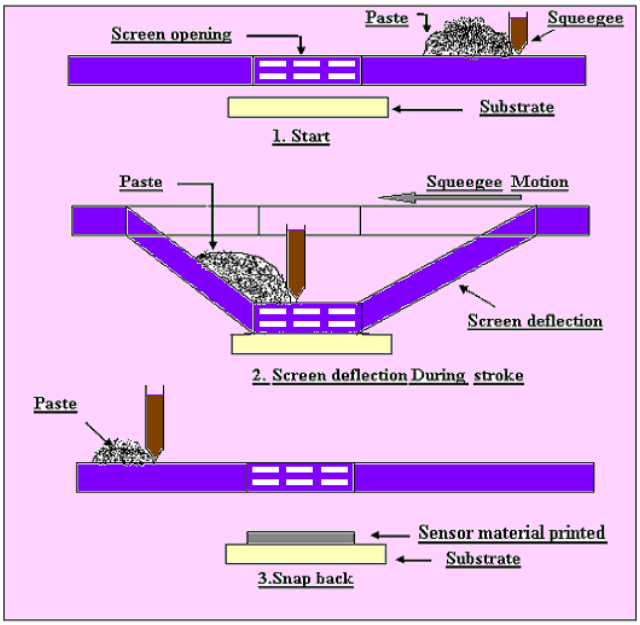


Fig. 2: Screen printing mechanism

The thick film technique using screen printing offers a good control over the thickness and microstructure. The life time of thick films is expected to be larger. The use of thick film technology in the production of chemical sensors has opened up the possibility of manufacturing sensors in a cost effective manner. Such properties of a thick film sensor are highly desirable for chemical applications.

Furthermore, thick film technology has the ability to produce the sensors from nano-scaled materials. This is an advantage because minute sample volume is required. Also, the portability of the interface instrument for the sensor can be realized. Thick film technology based on glass and ceramic compositions is very stable in severe conditions such as high temperature or corrosive environments. Deposition of the layers is most commonly carried out by using screen printing for high volume and low cost production. Each layer is printed with a paste comprising of a functional material and a temporary organic vehicle. After deposition, solvent was removed by drying followed by firing, in order to eliminate the organic binder and sinter the materials. Glass frits are commonly used alone for over glazes and as a permanent binder in thick film technology [26-32]. Commonly ceramic substrates made of mostly alumina (Al_2O_3), silicon, glass-ceramic and sapphire with appropriate surface finish are used.

5.3 Surface Functionalization of Thick Films

Surface activation [33-36] of thick films of pure SnO_2 powder was achieved by dipping them into a 0.01 M aqueous solution of Strontium Chloride or Bismuth Chloride for different intervals of time such as 5 min., 15 min., 30 min. and 45 min. and dried at 80°C under an IR lamp, followed by firing at 500°C for 30 min. in ambient air. The particles of Strontium Chloride or Bismuth Chloride dispersed on the film surface would be transformed to SrO_2 or Bi_2O_3 during firing process. Thus, the sensor elements with different mass percentage of SrO_2 or Bi_2O_3 incorporated in to thick films of pure SnO_2 were prepared. Silver contacts were made by vacuum evaporation for electrical measurements and monitoring the gas sensing performance of the thick films.

5.4 Static Gas Sensing System

Fig. 3 shows a static gas sensing system. This system is used in the laboratory for the gas sensing application. It contains gas chamber with heater. Sample holder provided with silver contacts along with gas injection system. To measure the gas response a Pico-ammeter is used. Power supply is also required to provide the necessary d.c. voltage. A variable a.c. source (dimmerstat) is used to control the temperature of the system. The variation in temperature is measured by means thermoelectric sensor. Stop watch is used to measure the response and recovery time of the sensor.

Fig. 3: Static gas sensing system

VI. CHARACTERIZATIONS

6.1 Measurement of Sensor Parameters

The characteristics that are desirable to measure the sensing performance [33-38] of any sensor are as follows.

a) Gas Response
b) Selectivity
c) Selectivity Factor
d) Response Time
e) Recovery Time
f) Sensitivity
g) Repeatability
h) Long Term Stability
i) Detection Limit
j) Linearity
k) Operating Temperature

a) Gas Response (S)

Gas response is defined as the ratio of the change in conductance of the sensor in presence and absence of target gas to the conductance in absence of target gas (air). The gas response (S) is given by the relation;

$$S = \frac{G_g - G_a}{G_a} \qquad \text{----------(1)}$$

Where, G_g and G_a are the conductance of sensor in air and in a target gas medium, respectively. The percentage gas response is given by the relation;

$$\% S = \frac{G_g - G_a}{G_a} \times 100\% \quad \text{---------- (2)}$$

b) Selectivity

Selectivity is defined as, the ability of a sensor to respond to certain gas in the presence of mixture of number of gases. Selectivity is also termed as, specificity. A good gas sensor should be very much selective for a particular gas in presence of some other gases or mixture of gases, even at high concentrations.

c) Selectivity Factor (K)

Selectivity factor of one gas over other is defined as, the ratio of the maximum response of the target gas to the maximum response of the other gas at optimum conditions, viz. temperature, gas concentration, etc.

$$K = \frac{S_{\text{target gas}}}{S_{\text{gas}}} \quad \text{---------- (3)}$$

d) Response Time (RST)

The time taken for the sensor to attain ninety percent of the maximum increase in conductance on exposure of the target gas, is known as response time.

e) Recovery Time (RCT)

The time taken by the sensor to get back ninety percent of the maximum conductance when the flow of gas is switched off, is known as recovery time.

f) Sensitivity

The sensitivity of a sensor is defined as the change in output of the sensor with respect to unit change in the parameter being measured.

g) Repeatability

The ability of the sensor to produce the stable response upon number of successive exposures of target gas is called as repeatability.

h) Long Term Stability

The ability of the sensor to produce the stable response over longer time span, irrespective of the number of target gas exposures is called as long term stability.

i) Detection Limit

It is the lowest concentration of the gas (ppm / ppb) that can be detected by the sensor under given conditions, particularly at a given temperature.

j) Linearity

It is the relative deviation of an experimentally determined calibration graph from an ideal straight line.

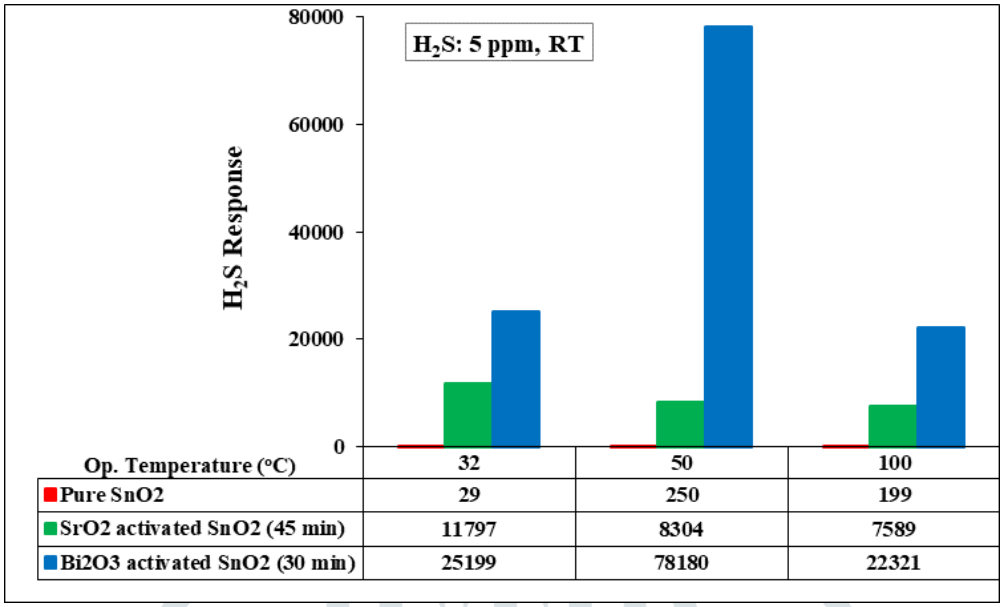
k) Operating Temperature

It is usually the temperature that corresponds to maximum response to a particular gas.

6.2 Structural and Electrical Characterizations

The studies on microstructures and surface morphology of thick film samples were carried out by using X- Ray Diffractometer (XRD) and Field Effect Scanning Electron Microscopic (FE-SEM) techniques, respectively. Also, electrical characteristics, viz. I-V characteristics and conductivity profile of all the samples were studied in the laboratory [33-38].

VII. Results and Discussion



Op. Temperature (°C)	32	50	100
Pure SnO2	29	250	199
SrO2 activated SnO2 (45 min)	11797	8304	7589
Bi2O3 activated SnO2 (30 min)	25199	78180	22321

Fig. 4: H_2S response of pure and surface activated SnO_2 thick films

Fig. 4 depicts the H_2S response of pure and surface activated SnO_2 thick films. From Fig. 4, it is observed that, Bi_2O_3 activated SnO_2 (30 min.) thick film is most sensitive and selective to H_2S (5 ppm) at room temperature as well as at 50°C. Upon exposure, H_2S gas gets oxidized by utilizing the lattice oxygen present in the bulk of the material of thick film, trapping behind the free electrons in the conduction band and enhances the conductivity of the material. The high performance of this film in H_2S sensing may be attributed to the enhancement of active surface to volume ratio due to its nanostructure. But, as the temperature increases, the H_2S response decreases. However, SrO_2 activated SnO_2 (45 min.) thick films also exhibit crucial response to H_2S at room temperature and response decreases at high temperatures.

VIII. Conclusions:

- A review of the existing literature suggests that the modified and unmodified SnO_2 thick films possess in several important areas of application of modern microelectronic techniques, because of their uses in production of advanced infrared detectors and sensors for sensing toxic gases and magneto-resistive sensors.
- The gas response of the sensor must be as high as possible to trace level (ppm / ppb) of the gas with very high selectivity and no cross sensitivities.
- The response and recovery times must be very low (~ few seconds).
- Pure semiconducting oxides are expected to be stoichiometric and hence are less sensitive and less selective to toxic, hazardous and polluting gases.
- It was studied that, the non-stoichiometric tin oxide behaves as semiconductor and act as gas-sensing varistor.
- Dipping process is the most suitable and simple technique as far as surface modification is concerned.
- The nanostructured SnO_2 material has the great potential to fabricate the gas sensors which have most advanced and highly applicable features.
- The review also reveals that, the number of researchers studied on SnO_2 thick films, but very few attempts have been made to study the dipping time variation with the response of gas. Hence, it is the need to study the effect on structural, electrical and gas sensing properties by changing dipping time.
- There is a great space to work for the development of SnO_2 based gas sensors.

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