

Sensing Mechanism of GO and V₂O₅ Metal Oxides Films for Ethanol Gas

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Abstract: GO and V₂O₅ powders and films are frequently developed by a screen-printing technique. The sensing properties of mixed metal oxide-based material depend upon its chemical, physical characteristics and amount of mixing of two metal oxides, which are strongly kept in to the preparation conditions, dopant and grain size. This praisures that the development of the sensor thick film is a one of important step within the preparation of good mixed metal oxide semiconductor gas sensors. Sensing properties of thick film studied by at different concentration of carbon dioxide gas and also study surface morphology of sample by using SEM. Also studied the stability and dynamic response of sensor against sensing gas.

Keywords: GO:V₂O₅; screen-printing technique; Ethanol gas sensor

I. INTRODUCTION

The last century has seen increased industrial growth worldwide. A side effect of this development is an exponential increase in pollution of earth, air and water, especially in densely populated areas. While land pollution is locally restricted and great efforts have been made during the last decades to improve the quality of rivers and larger bodies of water, air pollution is not so easily reduced.

Nowadays, there is a great interest in implementing sensing devices in order to improve environmental and safety control of gases. The most used gas sensor devices can be divided in three big groups depending on the technology applied in their development: solid state, spectroscopic and optic.

While spectroscopic and optic systems are very expensive for domestic use and sometimes difficult to implement in reduced spaces as car engines, the so-called solid-state sensors present great advantages due to their fast-sensing response, simple implementation and low prices [1,2,3]. These solid-state gas sensors are based on the Change of the physical and /or chemical properties of their sensing materials when exposed to different gas atmospheres. Although the number of materials used to implement this kind of devices is huge, this work was centered in studying the semiconductor properties, in those material using GO and V₂O₅ as sensing materials.

The main purpose of this paper is to study new materials for gas sensing elements starting from the knowledge in thick film production using screen-printing technique.

II. EXPERIMENTAL WORK

2.1 Materials

The list of chemicals and materials along with the sources and grades used for the preparation of sensors are given in the table 1.

TABLE I: Chemicals and materials with sources and grades

Chemicals	Acronym	Grade	Source
Graphene oxide	GO	AR	SD Fine, India
Vanadium Pentoxide	V ₂ O ₅	AR	SD Fine, India
Alumina	Al ₂ O ₃	GR	LOBA Chemi, India
Methanol	CH ₃ OH	AR	SD Fine, India

Acetone	CH ₃ COCH ₃	AR	SD Fine, India
Ethyl Cellulose	[C ₆ H ₇ O ₂ (OC ₂ H ₅) ₃] n.	AR	SD Fine, India
Butyl Carbitol	C ₈ H ₁₈ O ₃	AR	Merck, India

Synthesis and Sensor Preparation

As mentioned in the aim of the present work, the work concentrates on thick film sensor of GO-V₂O₅. The experimental method for the preparations of materials, fabrication of sensors, effect of dopant, screen-printing method and fabrication of gas chamber and gas flow meter is discussed.

Synthesis and Sensor Preparation

Calcination, the initial heat treatment, the precursor (hydrated metal oxides) undergoes provides thermal energy, which enables the growth of metal oxide particles. An increase in calcination temperature increases monotonously the mean grain size[4].

In addition, the full width half maximum of the grain size distribution also increases monotonously with calcination temperature. A longer calcination time results in larger grains and a broader grain size distribution. Grinding results in a reduction of grain size too. The smallest grains are obtained by grinding before and after the calcination [5].

The powders of GO, V₂O₅ and Al₂O₃ were calcinated at 820⁰C in an automatically temperature-controlled muffle furnace for 5 to 6 hrs. The powders of these samples were crushed in pestle before after the calcination to get the homogeneity in the powders. The Series of the samples were prepared. The different combinations are shown in table 2.

TABLE III: *Sample codes and mole percent for series GO and V₂O₅*

Sr. No.	Series	Composition of GO(mole %)	Composition of V ₂ O ₅ (mole %)
1	A	100	0
2	B	80	20
3	C	70	30
4	D	60	40
5	E	50	50
6	F	40	60
7	G	30	70
8	H	20	80
9	I	00	100

The ink or paste of the sample was prepared by using screen-printing (thick film technique) [6,7] technique. The standard basic materials: EC (ethyl cellulose), BCA (Butyl Carbitol Acetate) were used for the screen-printing process [8,9]. The EC and BCA were used as binders. The active powder and Ethyl cellulose were mixed thoroughly. During this mixing process, the BCA was added drop by drop to obtain the proper viscosity of the paste. For thixotropic property for printing on the substrate, the ratio of active powder to binder was kept as 3:7. Also ratio of Ethyl cellulose (EC) and Butyl Carbitol acetate (BCA) is kept at 8:92.

The glass substrate of size 7.5 x 2.5 cm² was used. The substrate is an important part of any thick-film process. It must also be proper shaped. For normal electronic purpose, the substrates structure should be rectangle. And washed the substrate. These dried samples were further heated at 140⁰C for 50-60 minutes with a heating and cooling rate of 22⁰C/min to remove binder. The thickness of all the prepared sensor samples was measured by digital micrometer. And following gas chamber(Figure 1) is used for characterization.

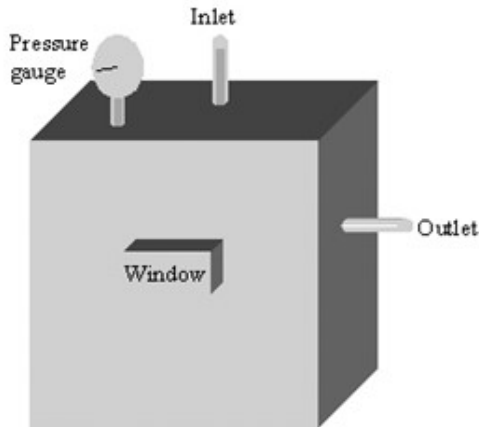


Figure 1: Gas chamber for Ethanol

III. RESULT AND DISCUSSION

Gas sensing properties of GO: V₂O₅ composites-

The variation of sensitivity of sensors of pure and GO-V₂O₅ composite materials with concentration of Ethanol gas at room temperature as shown in following Figure 2.

From above Figure shows that the sensitivity increases linearly upto 80 ppm and beyond that it shows saturation. Sensitivity of 80GO:20V₂O₅ composite has found maximum value i.e. 0.42 at 100 ppm as compare to other composites. It is also observed that with decreasing concentration of doping of V₂O₅ in GO:V₂O₅ composites, the sensitivity increases and becomes maximum for 80GO:20V₂O₅ composite. For pure GO and V₂O₅ sensitivity is less as compare to 80 and 20 composition. It is due to the high porosity of 80GO:20V₂O₅ composite as compared to other and pure GO and V₂O₅. Thus active surface area may available due to high porosity.

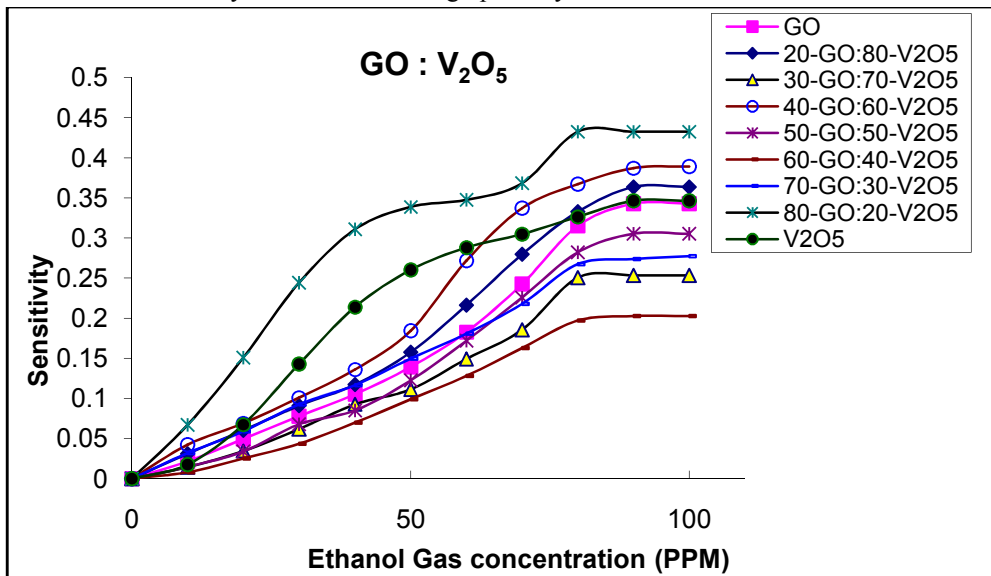


Figure 2: Variation of sensitivity of pure GO, pure V₂O₅ and GO: V₂O₅ composites with Ethanol gas concentration (ppm) at room temperature (300K).

3.1 Dynamic Response

Dynamic responses of GO:V₂O₅ series for 30 ppm, 60 ppm and 90 ppm are shown in the following Figure.

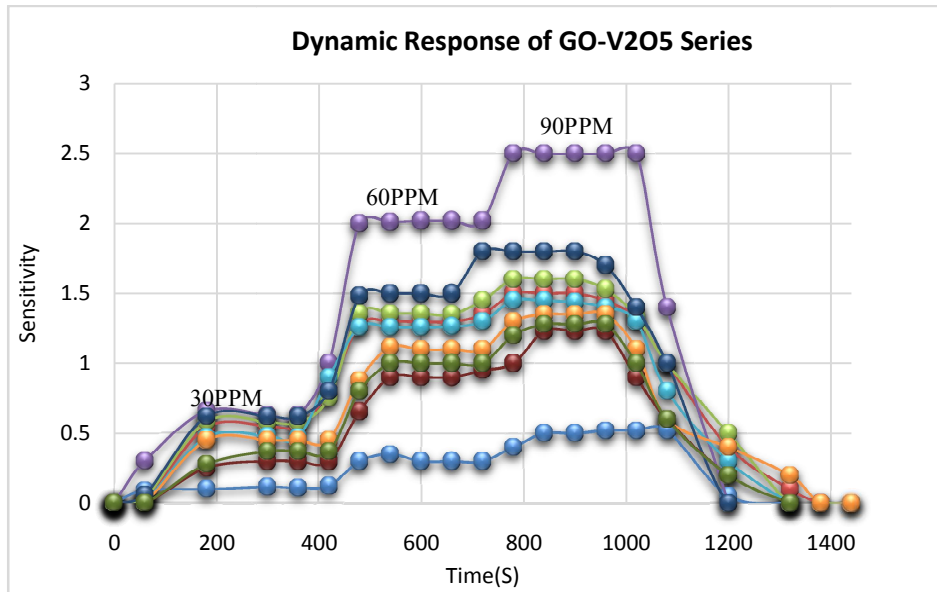


Figure3: Dynamic response of pure and composite sensors at 30, 60 and 90 ppm of Ethanol gas concentration at room temperature (303 K).

From above Figure, it is observed that 80GO:20V₂O₅ sensor shows fast recovery as compared to other composition sensors therefore 80GO:20V₂O₅ sensor is the best among the various reported sensors.

3.2 Stability of Sensor

Sensor stability is expressed in terms of measurement of resistance with time and shown in Figure. 4. It is defined as the change in resistance of sensor with time [10,11].

The resistance values of optimize sensors 80GO:20V₂O₅, measured with time at room temperatures are listed in the table 4.4 for time 60 h.

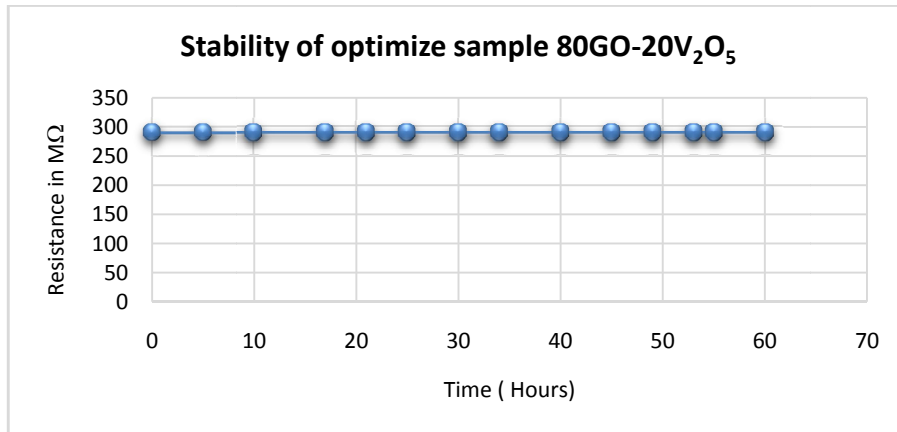


Figure 4: Stability curve

Table 3: Change in resistance of sensors in air with respective time.

Time (hour)	Sensor resistance in air (MΩ)		Time (hour)	Sensor resistance in air (MΩ)	
	80GO:20V ₂ O ₅			80GO:20V ₂ O ₅	
0	272.4561		34	272.4561	
5	272.4561		40	272.4561	
10	272.4561		45	272.4561	

17	272.4561		49	272.4561	
21	272.4561		53	272.4561	
25	272.4561		55	272.4561	
30	272.4561		60	272.4561	

From the Figure 4 and table 3, it is observed that all the sensors are stable for long-time use.

3.3 SEM Analysis

The surface morphology of optimized sample 80GO:20V₂O₅ material was studied by SEM and its picture is shown in the following Figure.5

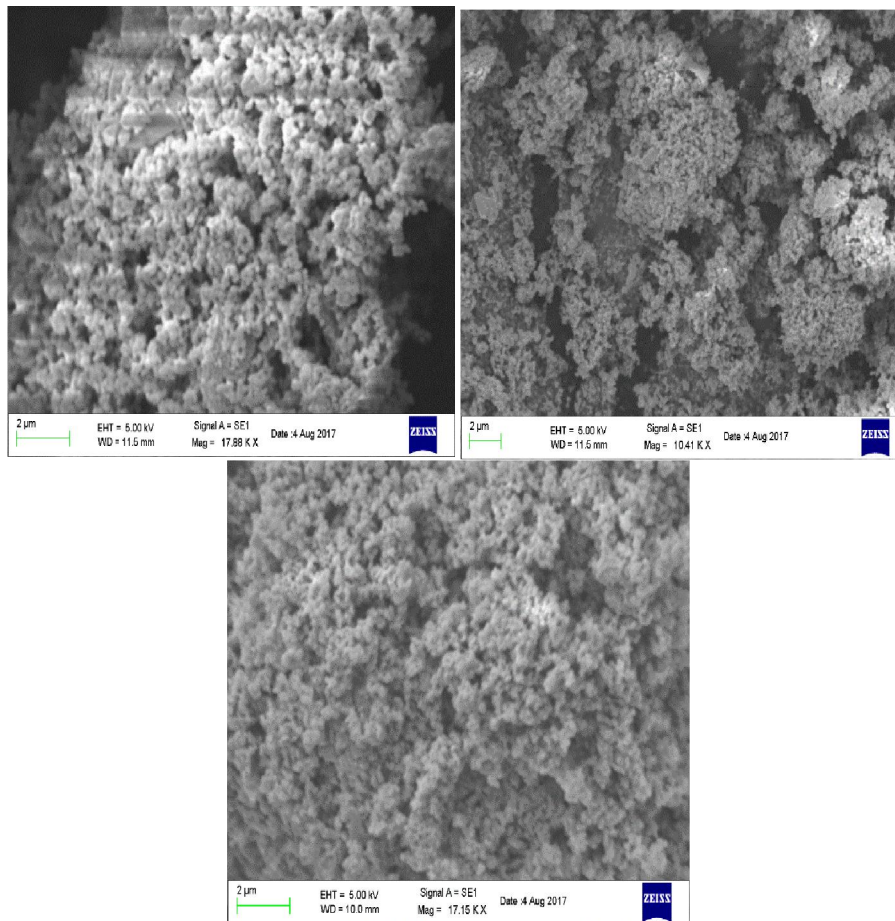


Figure 5: SEM pictures of 80GO:20V₂O₅ at different magnifications

The surface morphologies of 80GO:20V₂O₅ studied and the average diameter and number of pores per inch composites are 240 & 105 respectively. And number of pores per inch for pure and other composites is less.

It is also found that average diameter of pore in case of 80GO:20V₂O₅ composition is small as compared to other compositions. This also tends to exhibit large surface area and high response of the sample [12-18].

IV. CONCLUSION

We observed that due to grinding and calcination, the grain size of metal oxide powder can be improved. Calcination is initial heat treatment, the precursor undergoes provides thermal energy, which enables the growth of GO and V₂O₅ metal oxide particles. A longer calcination time results in larger grains and a broader grain size distribution. Grinding results in a reduction of grain size too. The smallest grains are obtained by grinding before and after the calcination.

If the grains are smaller than surface area will be larger. The sensitivity towards test gases increases due to increase in surface area. Thus, the calcination of metal oxide powder before paste preparation of film enhances the sensitivity.

From analysis of SEM, it is analyzed that the crystalline size of optimized sample 80GO:20V₂O₅ is smaller as compared with pure GO and V₂O₅. Also observed that, optimized sample is more pores and hence has large surface area as compare to other combination. It has been also observed that enhancement in gas (Ethanol) response for optimized sample 80GO:20V₂O₅ as compare to other composition. SEM analysis confirmed the surface morphology. From dynamic response and stability graphs, it has been observed that optimized sample shows good sensitivity and stability.

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