

Water Vapour Sensing mechanism of ZnO doped with SnO₂ Nanocomposite Thick Film

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Abstract: In this paper ZnO, SnO₂ nanoparticles was synthesized by a co precipitation method. Structural and particle size was calculated by using X-ray powder diffraction (XRD) method. Surface morphologies of thick film sensor were studied by Field Emission Scanning Electron Microscopy (FE-SEM). The structural morphology was studied by using Transmission Electron Microscopy (TEM). The sensor was made in the form of thick film. Water vapour or Humidity sensing investigations of these sensing materials were studied. Our result indicates that ZnO doped SnO₂ nanocomposite in form of thick film was most sensitive for humidity in comparison to pristine material under same conditions. The hysteresis plot between increasing and decreasing the RH range of 30–90% Rh and vice versa. The resistance of sample was decreases from 1010 Ω to 107 Ω in comparison with the pristine materials. The similar change was also observed in sensitivity and conductivity also. The results were re- producible up to $\pm$ 78% after 2 months of observations.

Keywords: Humidity Sensing, ZnO, SnO₂ nano particle.

I. INTRODUCTION

Recently the nanotechnology has a significant impact on several sectors, and utilizing nanomaterial development aimed at multifunctional applications in a range of disciplines is a major concern [1]. Since metal and metal oxide nanoparticles have many valuable properties due to small size and high surface area such as catalytic, optical, electrical, and magnetic properties, they have become very appealing materials in several fields over the past few years [2]. The ZnO nanostructures have can be synthesized by varrious methods and resulted samples have good potential applications. The synthesized methods can be effect on size and shape of products such as chemical precipitation [3], thermal decomposition [4]. ZnO has a hexagonal unit cell and wurtzite structure that crystallizes. Also has unique features, with a wide band gap 3.36 eV, at room temperature, it has high exciton binding energy, large electrochemical coupling coefficient, low toxicity, biocompatibility, and great photostability [5]. On integrating these kinds of materials producing their composite variations can advance their properties for sensor based applications. Notable illustrations of such type of composites contain Zn-SnO₂, Sn-ZnO, and ZnO-SnO₂. Mostly, the benefits of such composite-based sensors advance their surface reactions, which result from modifications to surface morphology that can be controlled during the preparation process [6-9]. Although there has been an increasing amount of research on the nanocomposite material like ZnO, ssSnO₂ the majority of earlier investigations focused mainly on gas sensing, leaving a notable gap in the exploration of their capabilities for humidity sensing.

II. EXPERIMENTAL

2.1 Synthesis of zinc oxide (ZnO)

The chemical used for the synthesis of ZnO are Zinc acetate dehydrate Zn(O₂CCH₃)₂(H₂O)₂, sodium hydroxide, Methanol and deionised water during reaction.

Initially, 0.2M Zinc Acetate dehydrates was dissolved in 100 ml deionised water was ground for 15 min and then mixed with 0.02 M solution of NaOH with the help of glass rod and solution was kept under constant magnetic stirring for 15

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58

min. and then again it was ground for 30 min. The white precipitate product was formed at the bottom. Then abundant liquid was discarded and the product was washed many times with the deionized water and methanol to remove by products. The final products were then filtered and obtain precipitate in the form of white paste, now this paste was kept in a vacuum oven at 80 °C for 4-5 hrs. so the moisture will remove from the final product and obtained dry product. Then this dry product was crushed into a find powder by using grinding machine and finally this fine nano-powder of ZnO was calcinated at temperature 800 °C for 6 hrs. so that the impurities from product will be completely removed and get a final product of ZnO nanoparticles.

2.2 Synthesis of Stannic oxide (SnO₂)

In preparation of SnO₂, 0.1 M of stannous chloride dehydrate (SnCl₂.2H₂O) is dissolved in 100 ml water. After complete dissolution, about 4 ml ammonia solution is added to above aqueous solution with constant magnetic stirring. Stirring is continued for 20 minutes. White gel precipitate is immediately formed. It is allowed to settle for 12 hrs. Then it is filtered and washed with water 2-3 times by using deionized water. The obtain precipitate were mixed with 0.27 g carbon black powder (charcoal activated). The obtained mixer is kept in vacuum oven at 70 °C for 24 hours so that the mixer gets completely in to dried powder. The obtained dry product was crushed into a find powder by using grinder. Now obtained product of fine nano powder of SnO₂ was calcinated at 700°C up to 6 hours so that the impurities from product will be completely removed.

2.3 Characterization

2.3.1 X-ray diffraction (XRD)

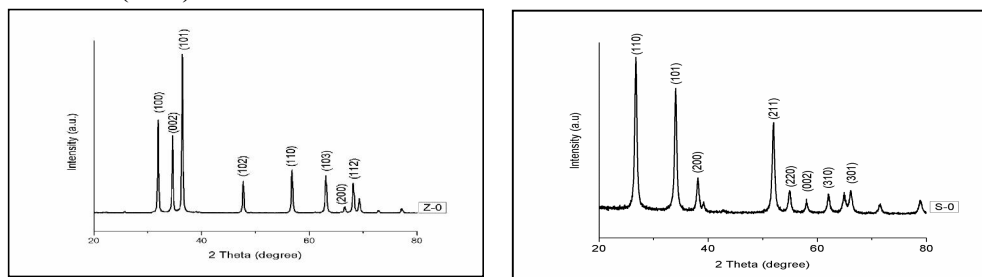


Figure (1) XRD pattern of pristine (a) ZnO (b) SnO₂

The XRD pattern of pristine zinc oxide (ZnO) nanostructure synthesized by liquid phase method via solid state method calcinated at 800°C as shown in figure (1) (a). The crystalline nature with 2θ peak lying at (100), (002), (101), (102), (110) and (103) planes. All the peaks match well the standard hexagonal wurtzite structure of zinc oxide with lattice constants $a = b = 0.3249$ nm and $c = 0.5206$ nm [JCPDS card no. 36-1451]. All the peaks are perfectly match with pure ZnO structure, which indicates the high purity of the obtained ZnO nanoparticle. The average crystalline size was found to be 37.32 nm calculated by Deye-Scherrer formula [10].

Figure (1) (b). shows the XRD pattern of pristine stannic oxide nanostructure synthesized by liquid phase via co-precipitation method calcinated at 400°C it is clearly observed that the highest intensity peak is obtained at (110) crystal planes and other peaks lying at (101), (200), (211), (220) and (002) of SnO₂. All the peaks match well with the standard tetragonal structure of SnO₂ with lattice constant $a = 4.723$ nm and $c = 3.238$ nm and its unit cell volume ($V=72.24\text{Å}^3$) with JCPDS card no. 71-0652. All the peaks are perfectly match with pure SnO₂ nanostructure, which indicates the high purity of obtained SnO₂ nanoparticles. The average crystalline size was found to be 23.19 nm calculated by using Debye-Scherer formula [11].



2.3.2 Field Emission Scanning Electron Microscopy (FE-SEM)

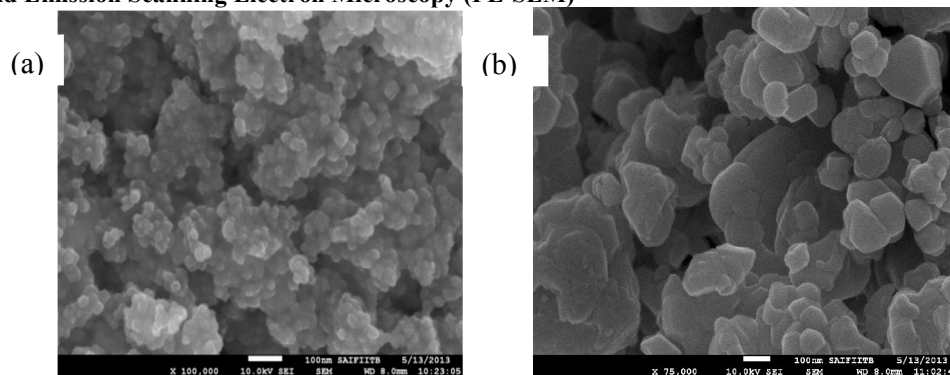


Figure (2) FE SEM images of (a) Tin Oxide (b) Zinc Oxide

Figure (2) shows the FE-SEM micrograph of pristine SnO_2 and ZnO , nanoparticles. The micrograph of sample pure SnO_2 thick films in this the particles are found to in the tetragonal shape within the particle size in the range of about 15nm to 31.2 nm [12]. Also, FE SEM micrograph of pure ZnO thick films shows particle are in both spherical and hexagonal shape with the average size 41 nm. The particle size observed in FE-SEM measurement is less than the crystalline size 56.78 nm [13].

III. RESULT AND DISCUSSION

3.1. Hysteresis loop

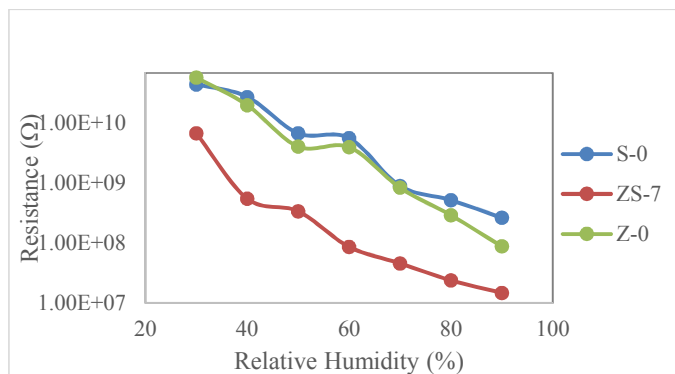


Figure (3) Hysteresis curve

Hysteresis curve shows the variation between resistances of sample with respect to the relative humidity in increasing and decreasing order from 30 to 90 % RH as shown in the figure (3). A very substantial and small hysteresis present during forward and reverse cycle of relative humidity, where as a very significant average change was observed in the value of resistance of sample the change in value of resistance is from $10^{10} \Omega$ to $10^7 \Omega$, these is a very notable change in the observed in the value of resistance of composite sample ZS7. In all the prepared sample the hysteresis is present which shows processes of regeneration is quite slower as compare to the pristine samples [14]. Apart from these a sample ZS5 (70 SnO_2 - 30 ZnO) shows comparable decrease in resistance with an increase in % RH which specifies that the conduction occurred at the grain surface by release of electron from the water molecule as compare to the pristine material. However, the sample shows the notable change in the resistance values in between the humidity range 30-90 % RH and possessed a high sensitivity factor due to large surface area and porosity in the form of thick films.



3.2 Sensitivity

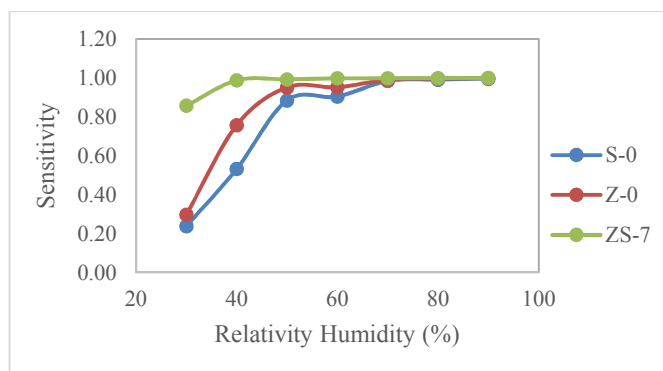


Figure (4) Sensitivity curve

In the above prepared nano composite samples, the sensitivity is found to be increasing with the RH for the samples of thick films and it is increasing up to some particular RH and then afterward it remains constant as shown in figure (4). As the RH of the sample increases the sensitivity is found to be higher in case of thick films sensor. The sensitivity of ZS7 (70 SnO₂- 30 ZnO) is more than pristine samples. By adding SnO₂ to ZnO as a nano-composites which shows that the sensitivity remains constant. As the change in conductivity is more in ZnO-SnO₂ nano composite samples the similar change is observed in sensitivity also. Hence, by the addition of SnO₂ to the pristine ZnO stabilized the sensitivity of all the samples. The nano composite sensors exhibit significantly higher sensitivity than sensor constructed specially from pure ZnO, nanoparticles itself due to the formation of heterogeneous interface between them and more adsorption site was created to absorb more water vapours [15]. The fall in resistance is mainly due to the increased amount of conduction electron or charge carrier upon adsorption of water vapours by the surface layer of the thick films.

IV. CONCLUSION

Nanostructured ZnO, and SnO₂ was successfully synthesized via chemical precipitation method. Minimum crystallite size was found to be for ZnO is 37.32 nm, SnO₂ it is 23.19 nm. Surface morphology of ZS7 shows that most particles are spherical in shape leaving more space as pores and hence it was most sensitive among all the prepared samples. The Hysteresis curve shows very significant average change in the value of the resistance from 10¹⁰ Ω to 10⁷ Ω during forward and reversed cycles of sample ZS7. The sensitivity is found to be increasing with the RH for all the samples of thick films and it is increasing up to some particular RH and then afterward it remains constant. As compare to the pristine the nano composite of ZS7 (70 SnO₂- 30 ZnO) sample is more sensitivity. This nano composites carries a good scope for the development of moisture sensor in the range of relative humidity 30% to 90% RH.

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