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PURE ZINC OXIDE BASED NONO THICK FILM FOR WATER VAPOUR SENSING APPLICATION

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Abstract
In this paper pristine Zinc Oxide (ZnO) nanoparticles was synthesized by a co precipitation method. The structural and compositional characterization has been studied by using X-ray powder diffraction (XRD). The sensors are made in the form of thick film. The Surface morphologies of the prepared samples were analyzed using Field Emission Scanning electron microscopy (FE-SEM) for thick film. Further, Water vapour or humidity sensing investigations of these sensing materials were done. Our result indicate that ZnO in form of thick film sensor is most sensitive for humidity under same conditions. The hysteresis plot between increasing and decreasing the RH range from 30–90% RH and vice versa has been studied. The samples resistance decreases from 10^{11} Ω to 10^{8} Ω respectively. The similar change was also observed in sensitivity. The results were re- producible up to ± 77% after 2 months of observations.

Keywords: ZnO, Humidity chamber, XRD.

Introduction

Humidity plays a very important role in every part of the earth surface especially in various biology and automated industrial area. For the required surrounding atmosphere, it is most important to monitor, detect and control the ambient humidity under different conditions. Recently, in the field of material science, nanocrystalline materials divert much interest due to their novel structural, electrical and optical properties which are significantly differ from the bulk solid state [1].The fundamental properties of ZnO, like growth, electrical and optical properties, since the potential for optoelectronic devices based on ZnO is also one of the main motivations for the present work. ZnO is a direct wide band-gap 3.37 eVand II-VI binary compound semiconductor and crystallizes inthree forms such as hexagonal wurtzite, cubic zinc blende and the rarely observed cubic rock salt [2]. A tendency of different fast growth directions of ZnO could result in growth of a diverse group of hierarchical and complex nanostructures. This is partly reflected by the various structural morphologies of ZnO nanomaterials such as nanorods, nanotubes, nanocorals, nanoflowers, and nanowalls[3].

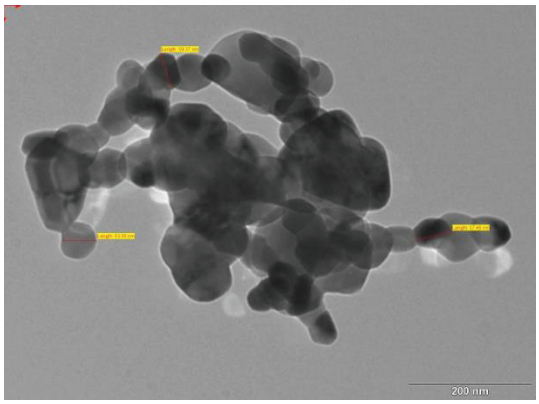
Experimental

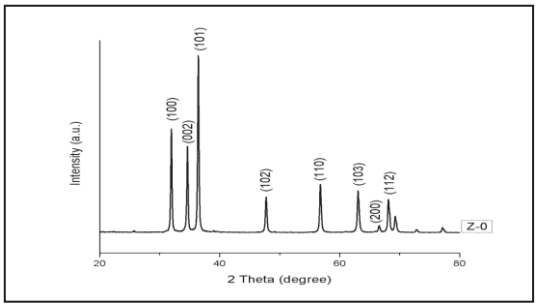
In preparation Zinc Oxide (ZnO) 0.2M Zinc Acetate dehydrates was dissolved in 100 ml deionised water was ground for 15 min and then mixed with 0.02 M solution of NaOH with the help of glass rod. After the mixing the solution was kept under constant magnetic stirring for 15 min. and then again it was 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 ^{o}C for 4 hrs. so the moisture will remove from the final product and we will get 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 ^{o}C for 6 hrs. in the auto-controlled muffle furnace *(Gayatri Scientific, Mumbai, India.)* and get a final product of ZnO nanoparticles.

Preparation of thick films

The thick films were prepared by using screen printing techniques. Initially, for the screen printing the thixotropic paste was formulated by mixing the sintered fine powder of pure nano powder of Zinc Oxide (ZnO) a with a solution of ethyl cellulose (as 10% temporary binder) in a mixture of organic solvent such as butyl cellulose, butyl carbitol acetate and turpineol. The ratio of inorganic to organic part was kept as 75:25 in formulating the paste. The paste was then used to prepare thick films of pure nano materials of ZnO and it was screen printed on a glass substrate in the desire pattern. The prepared films were dried at 80-110^{o}C in oven for 1hrs then the dried films are kept for fired at 500^{o}C for 25 min in muffle furnace (Kumar make Mumbai), so that all the organic materials (in the form of binders) and organic impurities can be evaporated form the sensor material. For the surface conductance measurement, the electrodes of silver paint were formed on adjacent sides of the films.





Characterization

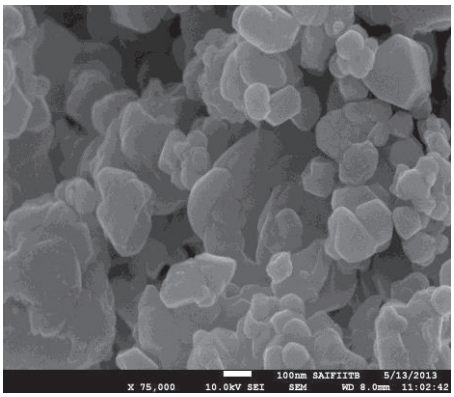
X-Ray Diffraction

The XRD pattern of pristine zinc oxide (ZnO) nanostructure synthesized by liquid phase method via co-precipitation method calcinated at 800^{o}C.The crystalline nature with 2θ peak lying at (100), (002), (101), (102), (110) and (103) planes. All the peaks match well the standard hexagonal wurtize structure of zinc oxide (ZnO) 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 [7].

TEM

TEM images for pristine ZnO which shows the formation of highly crystalline zinc oxide (ZnO) nanoparticle by using liquid phase method via solid state method. The shape of ZnO nanoparticles is like to dumbel, nanoroads and their average crystalline size is found to be 56.78 nm [8].

FE-SEM



The micrograph of pure ZnO thick films. In these the 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 using TEM and XRD measurement.

Result and Discussion

It has observed that there is the influence of the humidity on the transport properties of various metal oxides. The materials which used for the study were found to be more sensitive to the humidity. The properties such as electrical conductivity, sensitivityand response time and activation energy were studied in presence of humidity**.**

Hysteresis characteristics

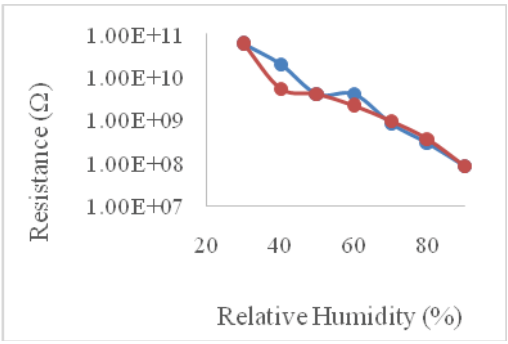


Figure (1.1)

Figure1.1 shows the hysteresis plot forpristine Zinc Oxide (ZnO) at constant temperature 30^{o}C. Hysteresis plot shows the variation between resistances of sample with respect to the relative humidity in increasing and decreasing order (30 to 90 % RH and 90 to 30 % RH) in steps of 5 % RH. Particularly, it is corresponding to resistance measurement by Keithley voltage source meter 2400 at constant temperature.Overall, in all the samples hysteresis plot measurement carried by Keithley voltage source meter (2400) at different constant temperature relative humidity varies from 30 to 90 % RH in steps of 5 % RH and vice versa also.

In all the samples the resistance of sensors decreases by increasing the relative humidity from 30 to 90% RH and the resistance of sensors increases by decreasing the relative humidity from 90 to 30% RH at respective constant temperature. The presences of hysteresis show the processes of adsorption and deadsorption are not so faster at particular

humidity. In this process the adsorption would not be efficient which cause a small change in the resistance. The physiosorbed water molecules is converted into chemisorbed by donating the surface electron at the constant temperature [10].The samples resistance decreases from 10^{11} Ω to 10^{8} Ω respectively.

Sensitivity

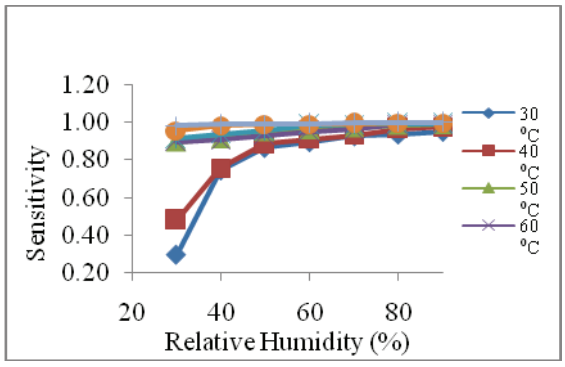


Figure (1.2)

Figure (1.2) shows the variation of sensitivity with increasing and decreasing (30 to 90 %RH and 90 to 30% RH) for pure Zinc Oxide (ZnO) respectively at constant temperature (30ºC to 90ºC).The pristine Zinc oxide (ZnO) based sensors exhibits significantly higher sensitivity than sensor constructed specially frompure ZnO nanoparticles itself due to the formation of heterogeneous interface between them and more adsorption site was created to absorbed more water vapuors. 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. 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. Initially, at low humidity levels the adsorbed water molecules get ionized on the surface and the hydronium ions are produced by the assistance of high electric charge density in the neighbourhood of the hydroxyl (OH-) sites resulting in the protonic conduction to the adjacent sites [11].

In these samples 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. For higher RH thesensitivity is found to be higher in case of all samples of thick films. The sensitivity of pristine Zinc oxide (ZnO) is more.

Conclusion

Nanostructured ZnO was successfully prepared via co- precipitation method. Minimum crystallite size was found to be for ZnO is 37.32 nm by using Debye Scherrer formula and it is also confirm by TEM. The nano particles size crystalline i.e. the grain size are in the range of nanometer surface morphology was confirmed by using FE-SEM analysis and which is responsible for humidity sensing mechanism. Surface morphology of pure ZnO shows that most particles are spherical in shape and hexagonal shape leaving more space as pores and hence it will more sensitive for water vapour sensing. The Hysteresis plot shows very significant average change in the value of the resistance from 10^{11}□ to 10^{8}□ during forward and reversed cycles. The sensitivity is found tobe increasing with the RH for all the samples of thick films sensor and it is increasing up to some particular limit of RH and then afterward it remains constant.This nano composites carries a good scope for the development of moisture sensor in the range of relative humidity 30% to 90% RH.The results were re- producible up to ± 77% after 2 months of observations respectively.

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