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Synthesis and Characterization of Zinc Oxide and Aluminum Doped Zinc Oxide by Sol-Gel Method

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Abstract

In the present Investigation we have used sol-gel and spin coating method to synthesize zinc oxide Nano particles having modified parameters with a comprehensive overview of their structural and experimental study. To evaluate its electrical, structural and optical properties as synthesized particles were analyzed by X-Ray Diffraction (XRD), UV spectroscopy and Particle size analysis. These crystalline size nano particles were utilized in sensors to detect volatile organic compound (Ethanol, Methanol and Formaldehyde) and was found to have high sensitivity and quick response time. In this paper we increase the overall band gap with the increase in concentration of aluminum and at the same time electrical conductivity of ZnO is also changed with the concentration of aluminum.

Keywords: Sol-Gel Method, ZnO2 Nano Particles

1. Introduction

In recent Years, ZnO has been the most focusing area as far as semiconductor materials are concerned and have which shows different properties, because of its optical properties. It looks to be a powder which cannot be dissolving in water. Zno is of the interest today because of its bonding and binding energy (60meV) which combines free electrons to perform leading action (laser) at room temperature. That binding energy is 2.4 times that of room temperature, therefore it is of utmost interest to research community[1]. Besides This property, Zno has an add-on property which is over other materials, its high energy radiation stability and comfortable to etching techniques, which makes it a perfect material to be used in space applications

The Electrical conduction in ZnO above room temperature has been more of interest as it excites the electrons to move from donor levels which actually originated from defects or impurities. We can control the doping level, by doing this insulator material can also be converted semiconductor to metal with the add-on features maintaining the optical transparency that makes it useful for certain applications. In this paper we have reported sol-gel and spin coating method of synthesis of zinc oxide nanoparticles, its characterization and its utilization in sensors to detect harmful gases even in very low range of concentration. There were many methods to synthesize ZnO2 but we preferred sol-gel and spin coating method because, it was easy to use, able to reduce the temperature and time without using any catalyst.

2. Experimental Setup

2.1 Sol-Gel Process

The sol-gel process is a method for creating substances from atoms and is used for creating and making different metal oxide specially the oxides of zinc[2] silicon and aluminum. In this method, a wet chemical route for making the combination of hybrid materials by using the collision dispersion properties of inorganic and organic materials. These methods are used for fabrication of materials from

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a small chemical solution for an integrated network of either discrete particles or network polymers (Figure 1).

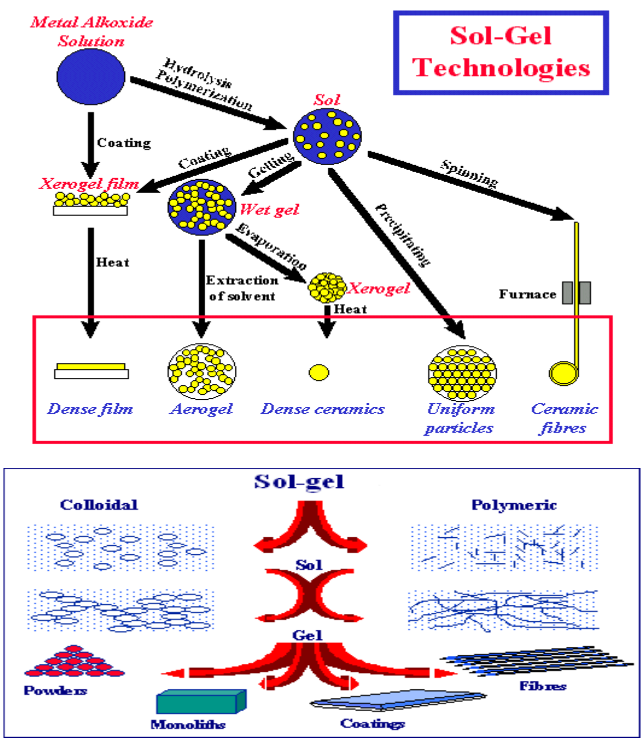

Figure 1. Sol-gel method.

3. Results and Discussion

Zinc oxide thin films are prepared by sol-gel method using zinc acetate as a precursor, diethanolamine (DEA) as a stabilizer and ethanol as a solvent. The concentration of zinc acetate was varied as 0.1M, 0.2M, 0.3M, 0.4M, 0.5M and 0.75M. Zinc acetate was first dissolved in DEA and ethanol, stirred at 60°C for few hours to yield a clear solution. Sol was deposited on glass substrate by using spin coater, at 3000 rpm for 40 seconds. After each coating the film was annealed at 200°C for 10 minutes to evaporate[3] the solvent and to remove the organic residuals. The procedure from coating to drying was repeated 20 times. The second heat treatment was at 500°C for one hour which forms pure zinc oxide. We used 0.5M zinc acetate to investigate the effect of doping Al in ZnO[4]. Aluminum chloride hexahydrate was used as a dopant source. We have prepared ZnO: Al thin films with Zn: Al atomic ratio as 0.5, 1, 1.5, 2 and 2.5%. For the sake of comparison, a ZnO: In (0.5%) film was also deposited to see the effect of the dopant.

3.1 Optical Transmittance Results

The pure and aluminum doped zinc oxide thin films were optically characterized through transmittance measurements in the near UV- Vis region. The optical transmittance shows a good transparency within the visible range with an average transmittance of about 70%-80% as shown in the Figure 2(a,b)

The thin films of ZnO of 0.1M, 0.2M, 0.3M and 0.4M were of poor quality because of the less stabilization of the solution. The solution precipitated at the time of coating or during aging time. In some cases, the solution became highly viscous that spin coating lead to spin-off[5] of the solution[7]. Thin film of 0.75M when annealed at higher temperatures of 500°C resulted in the precipitate formation because of large concentration of ZnO.

Figure 3(a) shows transmittance vs. wavelength plot for undoped and in doped ZnO respectively. The undoped film was found to be transparent with an absorption edge at 376nm, which is close to the intrinsic bandgap of ZnO (3.3eV). These graphs gave the approximate value of band gap. According to these graphs the band gap of undoped O is 3.29eV and band gap of in doped ZnO is 3.43eV (Table 1).

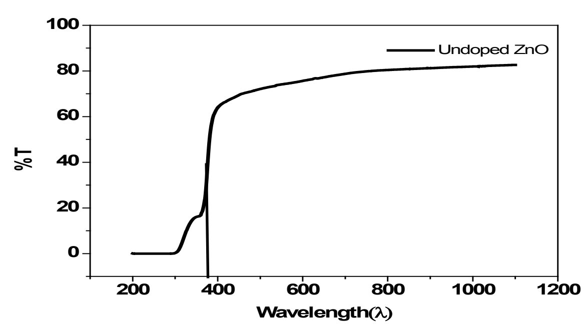

Figure 2(a). Band gap of undoped 0.5M ZnO is 3.29 eV.

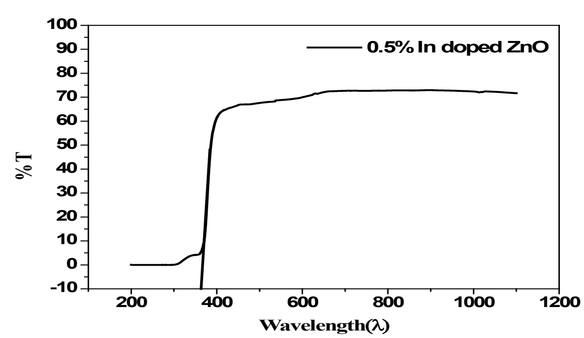

Figure 2(b). Band gap of in doped ZnO is 3.43 eV.

Table 1. Band gaps of undoped and doped ZnO.

Sample	Band gap
A (undoped ZnO)	3.26 eV
B (0.5% Al doped ZnO)	3.240 eV
C (1% Al doped ZnO)	3.236 eV
D (1.5% Al doped ZnO)	3.223 eV
E (2% Al doped ZnO)	3.21 eV
F (2.5% Al doped ZnO)	3.20 eV
G (0.5% In doped ZnO)	3.21eV

It is observed that the band gap of Al doped ZnO varies from 3.24 to 3.20 eV, which indicates that as aluminum concentration increases, the band gap of the thin films decreases with aluminium.

3.2 Electrical Measurements

All the samples exhibit a linear I-V characteristic i.e. an ohmic behavior. It is observed that even a very small percentage of aluminum in ZnO, results in a large decrease[6] in the resistance i.e., by two orders of magnitude. On further increasing the aluminum concentration the resistance tends to decrease in a systematic way.

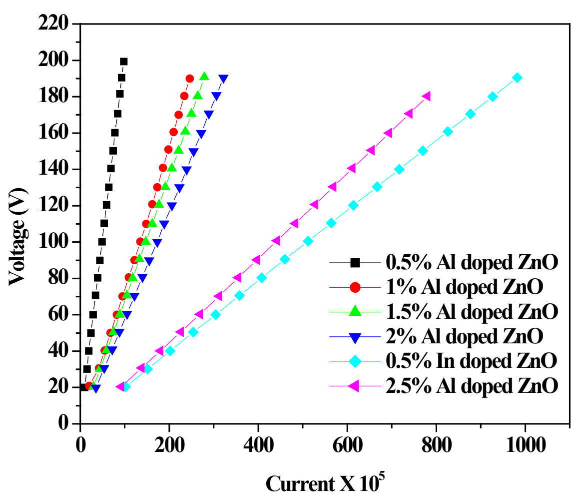

Figure 3. Comparison of the resistance of different percentage of doping ZnO.

The electrical conductivity of ZnO is linked to the number of combinations of electrons which are formed by combination of vacancies and Zinc atoms. Significant improvement in the electrical conductivity was most likely because of properties of oxygen which can combine with other materials from the grain boundaries and hence the grain size increases during the process.

3.3 XRD Results

The crystallinity of ZnO: Al film was determined by X- ray diffraction using an (Bruker) X- ray diffractometer with CuKα radiation source in the range of 30- 700 with glancing angle of 2.50 C. The Figure 4(a,b) shows the XRD patterns of AZO films annealed at 5000C for 1 hr. Three pronounced diffraction[8,9] peaks were observed at 31.79, 34.38 and 56.76. After instrumental broadening correction, an average value of crystallites was found to be 31-50 nm.

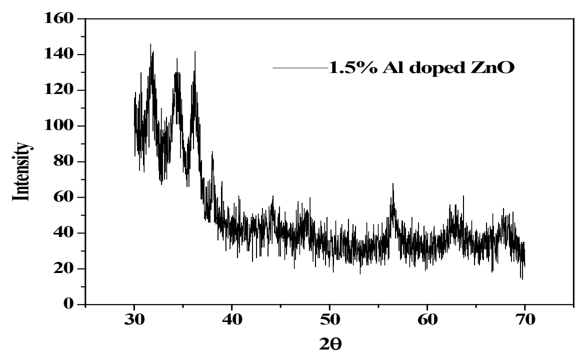

Figure 4(a). X-ray diffraction pattern of 1.5% Al doped ZnO.

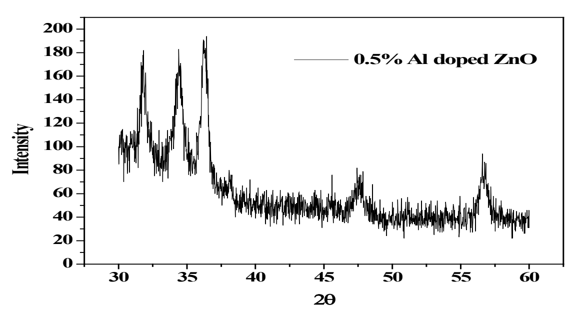

Figure 4(b). X-ray diffraction pattern of 0.5% Al doped ZnO.

It has been reported that with aluminum doping, there is a large decrease in resistivity, of the order of 10-3 Ω cm. Moreover, the band gap shifts to higher values. This is in contrast to the present case. In our case, the change in resistance is not very large. The numbers of electrons that come from doping are not sufficient to cause a Moss Burstein shift. Hence, in the present case, the band gap does not increase. The large resistance in the present case may also be due to the small grain size, 31-50 nm, which results in large number of grain boundaries and hence more resistance. The films were annealed in ambient air, so oxygen absorption from the atmosphere would also result in more stoichiometric ZnO and hence more resistance.

4. Conclusion

ZnO thin Films were manufactured using a sol–gel spin coating techniques. The impact of forerunner quantity was demonstrated and illustrated to clarify the outcomes on ZnO properties, which can be varied by changing the forerunner concentration. These techniques were used to establish the structure and crystallite size of these films and also to study the particle size and morphology. Dielectric studies reveal that dielectric constant and losses decreases with increase in frequency. The dielectric characterization shows the low value of the dielectric constant at higher frequencies. Results which comes out from optical spectrometry depicts that the transmission region for all different samples occurs in visible range and implies that the films are most transparent at forerunner concentration and can be used in certain applications where its properties can be used to increase the strength in thin-film sensors, transistors and solar cells.

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