

Preparation and Characterization of Zinc Oxide(ZnO) Nanoparticles via Co-precipitation Method

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Abstract:- In the present work, we synthesized Hexagonal Zinc oxide (ZnO) nanoparticles by precipitation method using Zinc nitrate and NaOH as precursors and starch as a stabilizing agent. X-ray Diffraction (XRD) patterns have proved the presence of Zinc oxide nanoparticles. Lattice parameter was derived from XRD data and the particle size of the nanoparticles was calculated by using Sherrer formula. This work aimed at the investigation of the dielectric properties such as dielectric loss and the permittivity of ZnO nanoparticles at various frequencies and temperatures. In addition, the ac conductivity of the Zinc oxide (ZnO) nanoparticles was studied.

Keywords— Zinc Oxide, Nanoparticle, XRD, Dielectric properties, A C Conductivity.

I. INTRODUCTION

Nanoparticles have revolutionized all major industrial areas, from drug delivery to agriculture and food industry [1]. Research on semiconductor nano-crystals has grown tremendously owing to their novel optical, electrical and catalytic properties during the past couple of decades. Unique optical and electronic properties of semiconductor nano-crystals are due to size quantization effect and surface state on the nanometer scale [2]. As the dimensions of semiconductors are of nanometer scale, the optical and electrical properties of these are much different from their bulk properties [3-5]. In recent years, one-dimensional semiconductor nano-materials have been extensively studied because of their potential applications as building blocks for nano-electronics, optoelectronics, electrochemical and sensor devices. Various semiconducting nano-materials of single elements, compound semiconductors and metal oxides have been successfully synthesized and studied [6-8]. Among these, zinc oxide (ZnO) nanoparticles are among the most promising materials for nano scale devices.

Bulk ZnO is cost effective and shows a variety of industrial applications [9-10]. Zinc Oxide is a member of III-V semiconducting compounds and occurs naturally as mineral Zincite. ZnO nanoparticles having a large band gap (3.37eV) with an excitation energy of 60 meV is an attractive material by virtue of its unique properties such as optical transparency, electrical conductivity, piezoelectricity and near UV emission [11-14]. The n-type semiconductor ZnO has numerous applications such as Varistors [15], chemical

sensors [16], and cosmetic materials especially for transparent UV protection [17], photovoltaic solar cells [18], catalysis [19], piezoelectric and luminescent devices [20], pigments [21] and transducers [22]. Moreover, it can also be made conductive by doping [23]. ZnO nanoparticles predominantly show wurtzite construction with lattice parameters $a=0.3296\text{nm}$ and $c=0.52065\text{nm}$. ZnO in its simplest form shows tetrahedron geometry in which each ion is enclosed by four counter ions pointing towards the corners of a tetrahedron. This tetrahedron configuration is responsible for the piezoelectricity and pyroelectricity [24-25]. Piezoelectricity of ZnO arising from its crystal structure makes it applicable for acoustic wave resonators and acoustic-optic modulators. Also, because of its Centro symmetric structure, it is the highest tensor among all semiconductors and gives large electromechanical coupling [26]. It is also inexpensive, nontoxic and environment-friendly. All these applications are based on control of both physical and chemical properties of nanoparticles, which in turn depend upon the size, shape, surface defects and crystal structure of the nanoparticle. Thus, it is important to prepare ZnO by a simple low temperature process that has the potential to yield nanoparticles of different shapes and uniform size.

In general, there are different approaches for synthesis of the ZnO nanoparticles, which are principally divided into three: the vapor phase, the solution and the solid phase [37]. As a result, in recent years different synthesis methods have been devised for ZnO nanoparticles such as vapor transport process [38], spray pyrolysis [39], thermal decomposition

[40], hydrothermal synthesis [41], sol-gel processing [42], direct precipitation etc. Among these methods, several methods like physical vapor deposition (PVD) and chemical vapor deposition (CVD) have been developed to synthesis ZnO nanoparticles into complex structures such as flower-like and web-like agglomerates. However, in order to obtain the final structure, these methods usually require multiple steps, sophisticated equipments and high temperature. In contrast, low-temperature wet chemical processes such as, hydrolysis, precipitation and hydrothermal process are cost effective and scalable and have been used for the preparation of a wide variety of ZnO nanostructures. The precipitation method in particular has been successfully used to design different structures of ZnO [43].

In this work, to prepare ZnO nanoparticles, precipitation method has been used since this method requires fewer instruments, less time and is cheaper. To prepare ZnO nanoparticles in an economical way, one needs to prepare it in single step for large scale production without the unwanted impurities. Low cost precursors such as zinc nitrate hexahydrate and sodium hydroxide are used. ZnO nanoparticles are prepared through a simple precipitation route.

II. EXPERIMENTAL

ZnO nanoparticles are prepared by precipitation method using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. 0.3 g starch is dissolved in 100 ml of distilled water. 10 ml of 0.1 M zinc nitrate is added to starch solution. Solution is stirred constantly using magnetic stirrer until complete dissolution occurs. After complete dissolution of zinc nitrate, 10 ml of 0.2 M sodium hydroxide solution is added drop by drop from the sidewalls. The reaction is allowed to proceed for 2 hrs after the complete addition of sodium hydroxide. The solution then is allowed to settle down overnight and the supernatant solution is then discarded carefully. Then, powder of ZnO nanoparticles is obtained after drying at 100°C.

III. RESULTS AND DISCUSSION

XRD ANALYSIS

The XRD pattern of the ZnO nanoparticles was recorded by using a powder X-ray diffractometer with a diffraction angle between 20° and 80°. Figure 1 and Figure 2 shows the XRD patterns of the prepared ZnO nanoparticles. The patterns were compared with standard data (JCPDS PDF card No. 043-0002) and the formation of hexagonal ZnO nanoparticles in all the samples was confirmed. Crystalline sizes of the samples were calculated using Scherrer formula and Lattice parameters of all samples were calculated using the following formulae and are tabulated.

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

$$a = \frac{\lambda}{\sqrt{3} \sin \theta_{100}} \tag{2}$$

$$c = \frac{\lambda}{\sin \theta_{002}} \tag{3}$$

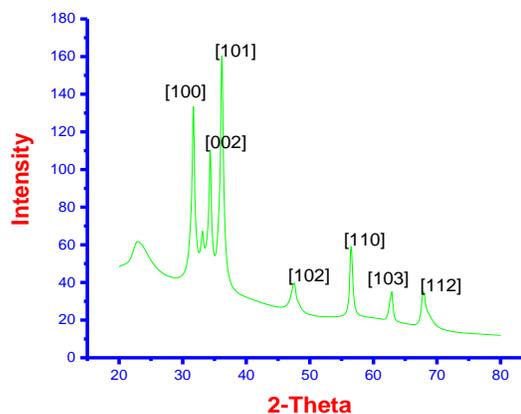


Figure 1: ZnO at 100°C

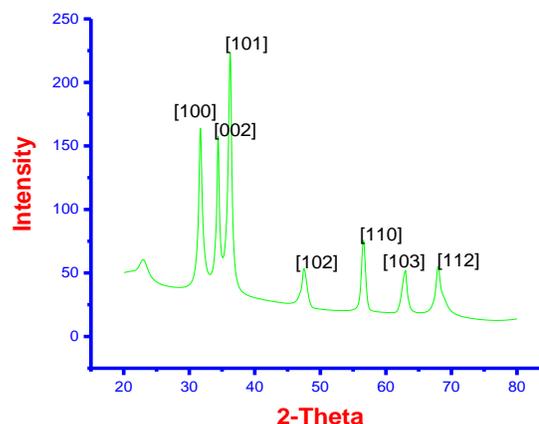


Figure 2: ZnO 200°C

Table 1. Particle size and Lattice parameters of 100 °C and 200°C samples.

Temperature (°C)	Particle Size (nm)	Lattice Parameter	
		a (Å)	c (Å)
100°C	15.87488	3.26223	5.22719
200°C	18.8262	3.20510	5.21686

In co-precipitation method, the crystallite size is observed to be increasing with temperature. Calculated values of lattice parameter of ZnO samples are listed in Table 1 and Table 2 which are in close agreement with standard data. It is clear from the table that the lattice constant is almost constant, and shows only negligible variation with sintering temperature.

DIELECTRIC PROPERTIES

The mechanism for microwave energy transfer into materials is complicated and consists of several contributions to the electric polarizability of molecules. Polarized molecules or atoms can rotate in response to an applied microwave electric field and cause microwave energy to be transferred from the electric field into the material. The dielectric properties of materials are characterized by the complex permittivity [27] given by,

$$\varepsilon = \varepsilon_0(\varepsilon' - j\varepsilon'') \quad (4)$$

Where ε_0 is the permittivity of free space, ε_0 is 8.854×10^{-12} F/m. The real part of the relative dielectric is a measure of the amount of energy stored in the dielectric due to the applied electric field and the imaginary part of the dielectric constant is a measure of an amount of energy dissipated in the dielectric due to the applied electric field. When a material is undergoing a complex series of chemical reactions, the nature of the chemical bonds are constantly changing and this change has a sensitive effect on the molecule's electrical polarizability. Another measure of dielectric loss is $\tan \delta$ or loss tangent, which is defined as $\varepsilon''/\varepsilon'$. It is a derived quantity from the more fundamental electrical parameters ε' and ε'' which are proportional to the amount of energy stored and dissipated respectively in a dielectric due to the presence of an applied electric field.

The dielectric properties of ZnO nanoparticles were analyzed using a Wayne Kerr 6500 B over the frequency range 100Hz-120MHz. The ZnO nanoparticles pellets in disk form were studied at two different temperatures. For measurements, a sample was mounted between copper platforms and electrodes. The capacitance and dissipation factor of the parallel plate capacitor which was formed by the copper plate and the electrode having the sample as the dielectric medium were measured. Frequency-dependence of the dielectric constant and dielectric loss at various temperatures is shown in Figure 3 and Figure 4 respectively.

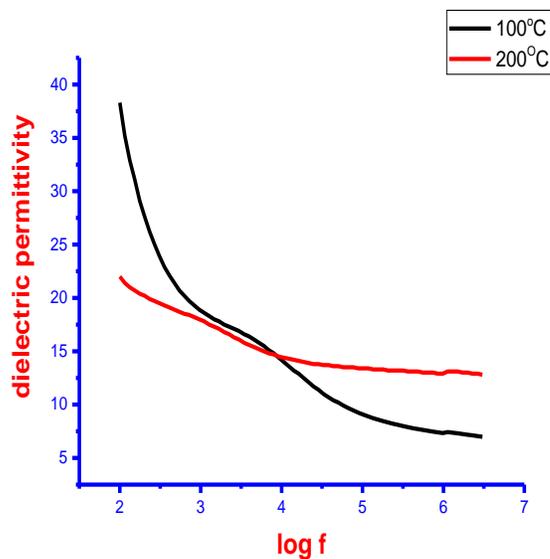


Figure 3

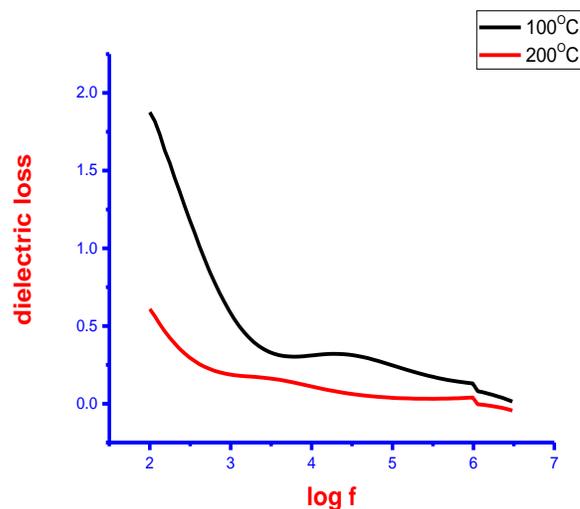


Figure 4

From the Figure 3 it is clear that the dielectric constant initially decreases rapidly with increases in frequency and then decreases slowly at higher frequencies. It is well known that there are two dielectric polarization mechanisms that contribute to the enhanced dielectric behavior of nano-materials: rotation direction polarization (RDP) process and space charge polarization (SCP) process. We suggest that both RDP and SCP process contribute to the enhancement of dielectric response of the ZnO nanoparticles. RDP process contributes for higher ε_r of ZnO nanoparticles. For typical n-type semiconductor, there are a large amount of oxygen vacancies acting as shallow donors in ZnO resulting in a lot of oxygen vacancies existing in the interfaces of ZnO nanoparticles [28-29]. Positive oxygen vacancies together

with negative oxygen ions give a large amount of dipole moments. These dipole moments will rotate in an external electric field, which leads to the rotation direction polarization occurring at the interfaces of n-type ZnO nanoparticles.

On the other hand, SCP can also occur in the sample. Generally, nanostructured materials have about 10^{19} interfaces/cm³, much more than those of bulk solids [30]. The interfaces with a large volume fraction in the nanostructured sample compacted under high pressure (50 MPa) contain a large amount of defects such as micro-porosities, dangling bonds and vacancy clusters. These defects can cause a change of positive and negative space charge distributions in interfaces [31-32]. Negative and positive space charge distribution in interfaces move towards positive and negative poles of the electric field respectively. As they are trapped by defects, dipole moments would be formed and SCP takes place in the sample. Since the volume fraction of the interfaces of nano-size sample is larger than that of bulk materials, SCP is stronger than that in the bulk materials. Thus, ϵ_r of the ZnO nanoparticles is higher than that of the bulk. Nevertheless, in the high frequency range, dielectric response of RDP and SCP cannot keep up with the electrical field frequency variation, resulting in the rapid decrease of ϵ_r in ZnO nanoparticles.

Energy absorption within a dielectric medium is characterized by the loss factor $\tan \delta$. From Figure 4 it is clear that $\tan \delta$ decreases rapidly. As the frequency increases, the rate of decrease slows down and the loss factor becomes almost frequency independent. Due to the high resistivity at the grain boundaries, the value of loss factor is high at lower frequencies. Hence more energy is required for hopping process resulting in high energy loss. However, in the high frequency region, due to the high conductivity of grains, energy required for hopping is less and hence loss factor becomes small.

AC CONDUCTIVITY

The a. c. conductivity of ZnO (σ_{ac}) is calculated with the data available from dielectric measurement and the equation [33].

$$\sigma_{ac} = 2\pi f \tan \delta \epsilon_0 \epsilon_r \quad (5)$$

Where f is the frequency of applied field, $\tan \delta$ is loss tangent available from dielectric measurement, ϵ_r is the relative permittivity of the sample and ϵ_0 is the dielectric permittivity of vacuum. The variations of ac conductivity as a function of frequency for the synthesized ZnO samples are shown in Fig (5).

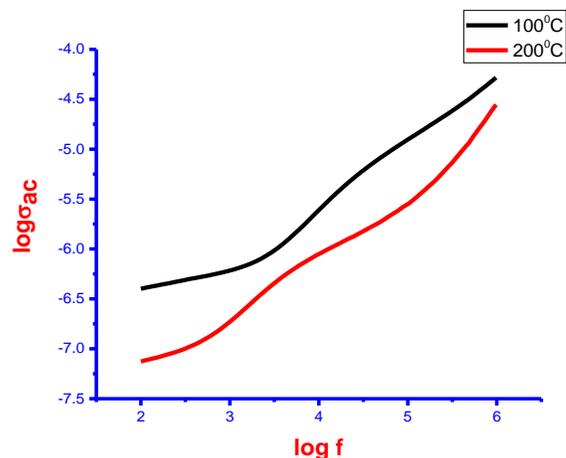


Figure 5

From Figure 5, it is clear that conductivity is almost constant in the low frequency region and at higher frequencies, plots are observed to be linear, confirming the small polaron type conduction in the samples. Small polaron formation may be due to the defects or oxygen vacancies created during sintering. In most of the disordered solids ac conductivity is directly proportional to the frequency, confirming the linear nature [34]. It is observed that, the ac conductivity increases gradually with increase in frequency. The observed slow increase in conductivity at low frequencies becomes rapid at higher frequencies for all samples. At low frequencies, few charge carriers tunnel through the potential barrier that forms the grain boundaries, giving rise to a feeble conductivity. The more charge carriers tunneling with increasing frequency enhances conductivity. Beyond a certain frequency, charge carriers get sufficient energy to overcome the potential barrier leading to a rapid increase in conductivity at higher frequencies [35-36].

IV. CONCLUSION

ZnO nanoparticles with hexagonal structure have been synthesized successfully by precipitation method. Hexagonal Wurtzite structure of ZnO was established by XRD. The average particle size of the samples was calculated using Sherrer formula and it was found to be 15.87nm and 18.82nm for samples sintered at 100°C and 200°C respectively. The impact of frequency and the temperature on the dielectric constant and the dielectric loss for ZnO samples were studied. And also, variations of ac conductivity with frequency were studied. From the dielectric studies it is found that frequency has a negative impact on both dielectric loss and dielectric constant as it decreases with increase in frequency.

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