

EFFECT OF ANNEALING ON THE STRUCTURAL AND ELECTRICAL PARAMETERS OF SOL-GEL DERIVED ZnO NANOPARTICLES

Abstract

In this paper, we report the synthesis of ZnO nanoparticles prepared via. Sol-gel method. The prepared samples were annealed at 200⁰C and 800⁰C for 1hr. The influence of annealing temperature on the particle size and morphology was examined by X ray diffraction. Electrical parameters such as dielectric constant, dielectric loss factor and AC electrical conductivity with respect to frequency and temperature were measured and the results have been analysed. As the process is simple and low-cost, it has the potential to be produced on a large scale.

Keywords: Sol-gel method, dielectric loss factor, dielectric constant, Electrical conductivity

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I. INTRODUCTION

Zinc Oxide is one of the II-VI semiconducting compounds which occurs naturally as mineral zincite. It belongs to hexagonal wurtzite type crystal exhibiting anisotropy. ZnO is a well-known n-type semiconductor and having a wide band gap of 3.3 eV at 300 K [1-5]. It is transparent to the visible light [6]. Moreover it is considered as a major candidate for UV and blue light-emitting devices such as blue LED and Lasers because of its large exciton binding energy [7, 8]. Even though, several NPs are showing their applications in different sectors of technology, zinc oxide (ZnO) NPs have showed much more importance in the recent years due to their attractive and outstanding properties such as high chemical stability, high photostability, high electrochemical coupling coefficient and a wide range of radiation absorption [9]. It exhibits piezoelectricity, meaning it can generate an electrical charge when subjected to mechanical stress. Additionally, ZnO has antibacterial and UV-protective properties, which make it useful in various industries such as cosmetics, healthcare, and textiles [10,11]. Recent years, ZnO NPs are predominantly used as antimicrobial agents, delivering systems vaccines and anti-cancer systems, photocatalyst, biosensors, energy generators and bio-imaging materials [12–15]. In addition to their fundamental importance in solid-state physics, the phonon properties of ZnO are also of practical significance. For example, they play a vital role in determining the thermal conductivity and thermal transport properties of ZnO, which are important for applications such as thermoelectric devices and heat dissipation in electronic devices [16].

II. EXPERIMENTAL DETAIL

ZnO NPs were prepared by an easy route sol-gel method. The reactants used here for the synthesis of ZnO nanoparticles were zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), Sodium hydroxide (NaOH) and PVA were used for the preparation of ZnO nano particles. Distilled water was used as the solvent. In this process Zinc acetate and NaOH were taken in the 1:3 molecular ratio and was dissolved in 100ml doubly distilled water then 2g of PVA was added to the modified Zinc acetate solution. Now a white precipitate was obtained. Annealing was carried out for the samples at 200⁰C, 800⁰C. The annealed samples were characterized structurally and electrically.

III. CHARACTERIZATION

The structural characteristics of pure and annealed samples were identified by the powder x-ray diffraction analysis. The AC electrical measurements such as dielectric constant, dielectric loss factor and electrical conductivity were carried out by the conventional parallel plate capacitor method using an Agilent 4284A LCR meter at various temperatures for different frequencies.

IV. RESULTS & DISCUSSION

Fig 1-3 shows the PXRD pattern of as prepared and annealed samples and the variation of structural morphology for different annealing temperatures was analysed. In order, to understand the ZnO phase evolution, calcinations was performed at 200⁰C and 800⁰C. The results showed that ZnO thus formed is of hexagonal phase (JCPDS no. 36-1451 with P63 mc space group, $a=3.249 \text{ \AA}$, $c=5.206 \text{ \AA}$) The major peaks were indexed as (1 0 0),

(0 0 2),(1 0 1), (1 0 2), (1 1 0),(1 0 3),(2 0 0 and (1 1 2). It is observed that there is an improvement in the crystallinity of ZnO nanoparticles with the increase in the annealing temperature [17]. Table.1 shows the effect of annealing temperature on the crystallite size. When the annealing temperature is increased to 200°C, the crystallite size of 26.66 was obtained. Further increase in the annealing temperature results in the increase of the size to 31.96 nm. The colour of pure ZnO was white, at 800°C it turns to pale yellow. The yield percentage of various temperatures were calculated and given in the table 1.

Table1: Preparation time, Sample Used , Colour and Yield Percentage for the Prepared Samples

Sample	Reaction time (min)	Colour	Yield (%)
Pure ZnO	60	white	30.34
Annealed 200°C	60	white	18.6
Annealed 800°C	60	Pale yellow	9.7

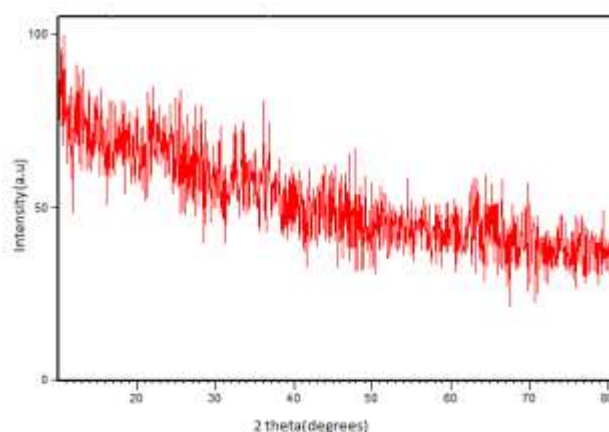


Figure 1: Un-annealed ZnO nanoparticles

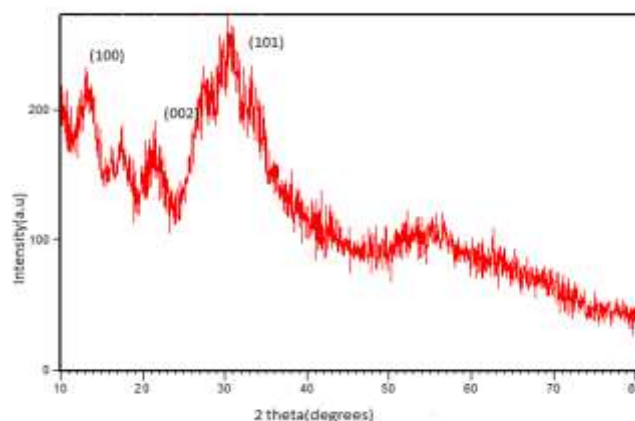


Figure 2: 200°C annealed ZnO nanoparticles

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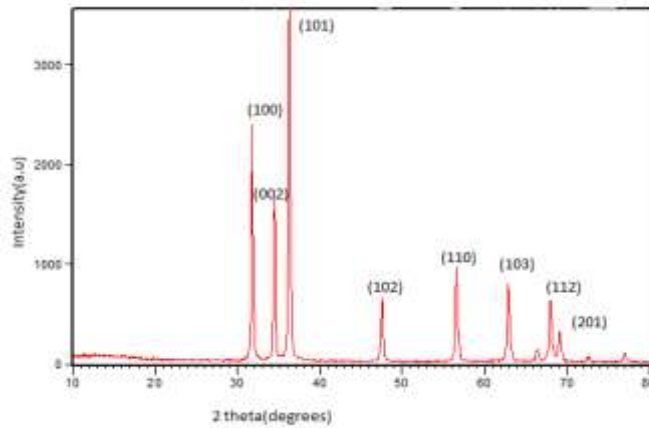


Figure 3: 800⁰C annealed ZnO nanoparticles

The dielectric and the AC conductivity of the prepared samples were analyzed for the temperature range 40 - 150°C for the range of frequency 100 Hz – 1 MHz using Two-probe method. Variation of dielectric constant for the as prepared and annealed samples with temperature for various frequencies are depicted in Figs. 4 - 6. From the Figure it was clear that for all frequencies, with increasing the temperature, the dielectric constant increases. This may be due to change in available number of free electrons in a material with increasing temperature in turn, changes the conductive part and hence the capacitive part [18]. In Fig7-9 at low frequencies the dielectric loss is high. Higher dielectric loss factor ($\tan\delta$) at lower frequency may be attributed to DC conduction losses [19]. Typically, the dielectric losses are attributed to the crystal structure and the imperfections in the crystal system such as impurities, micro-structural defects, grain boundaries, porosity, micro-cracks and random crystallite orientation.

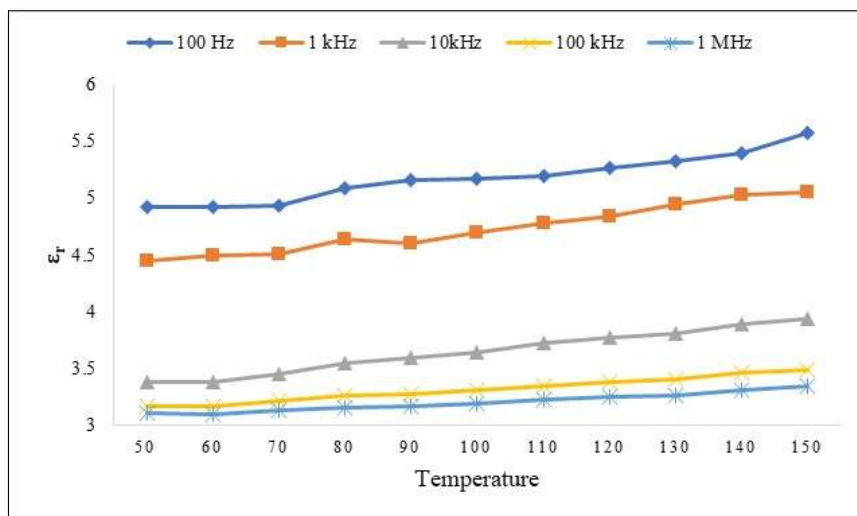


Figure 4: Dielectric Constant for Un-annealed ZnO

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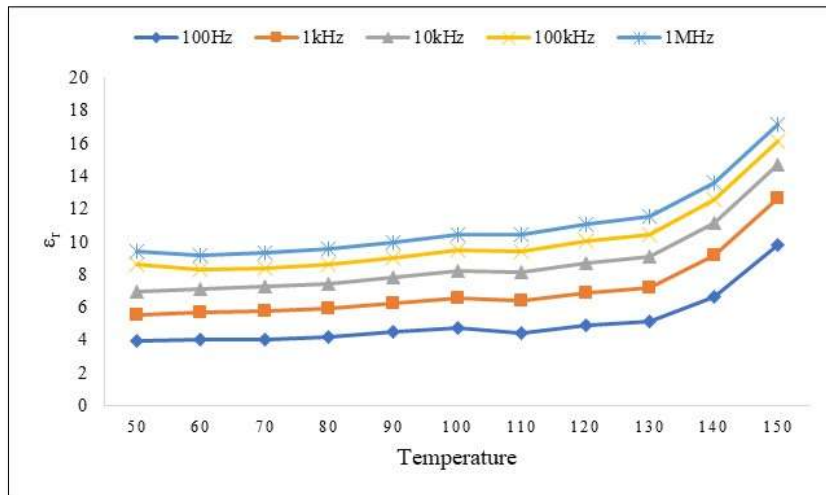


Figure 5: Dielectric Constant for ZnO annealed at 200°C

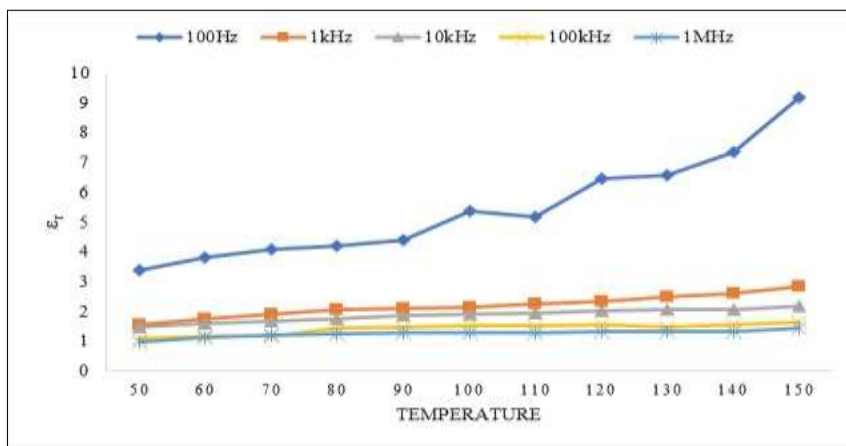


Figure 6: Dielectric Constant for ZnO annealed at 800°C

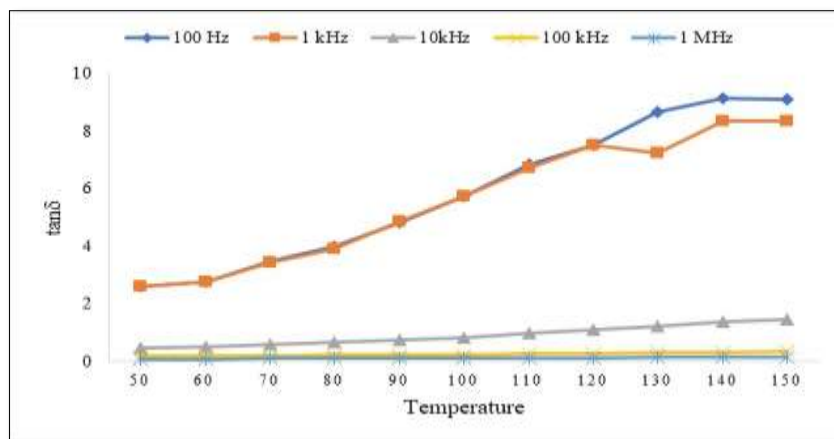


Figure 7: Dielectric loss for Un-annealed ZnO

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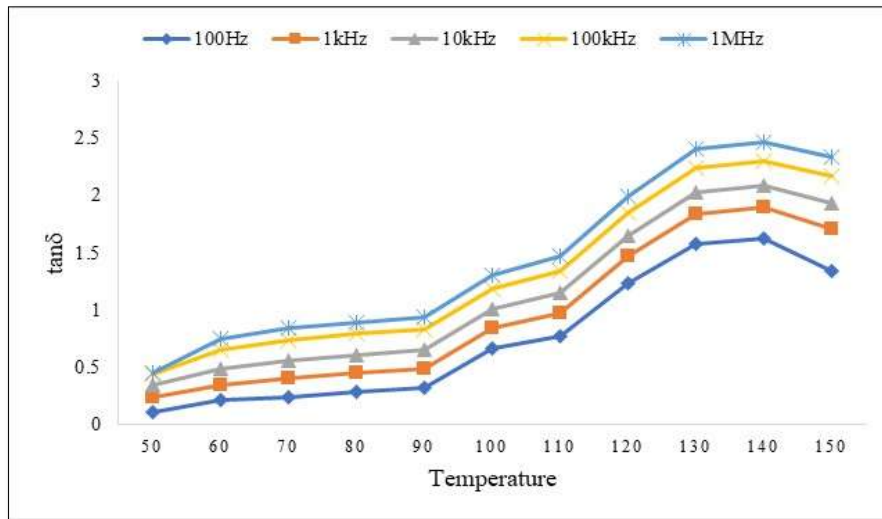


Figure 8: Dielectric loss for ZnO annealed at 200°C

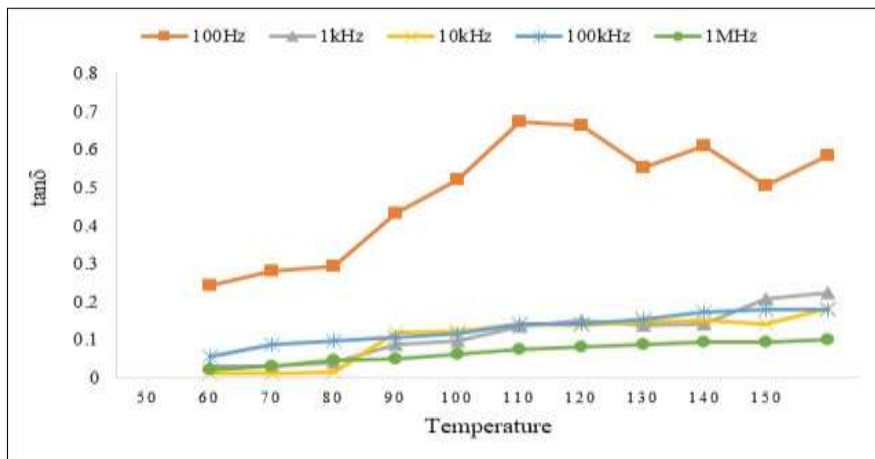


Figure 9: Dielectric loss for ZnO annealed at 800°C

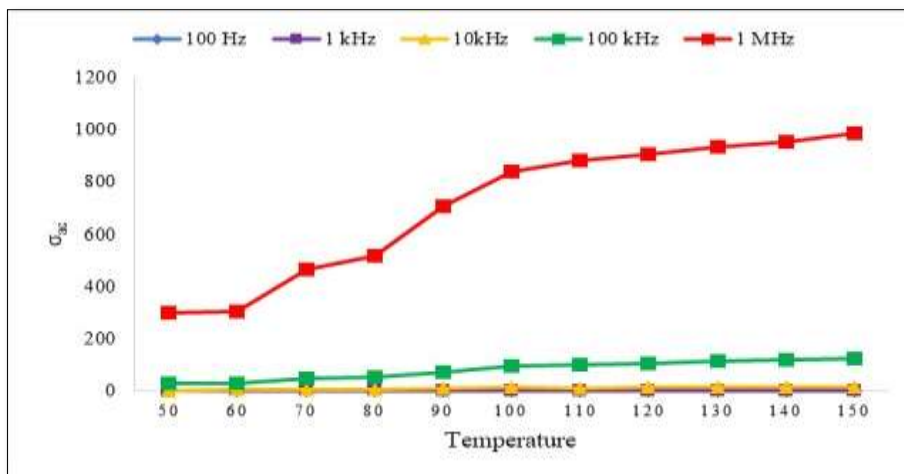


Figure 10: AC Conductivity for Un-annealed ZnO

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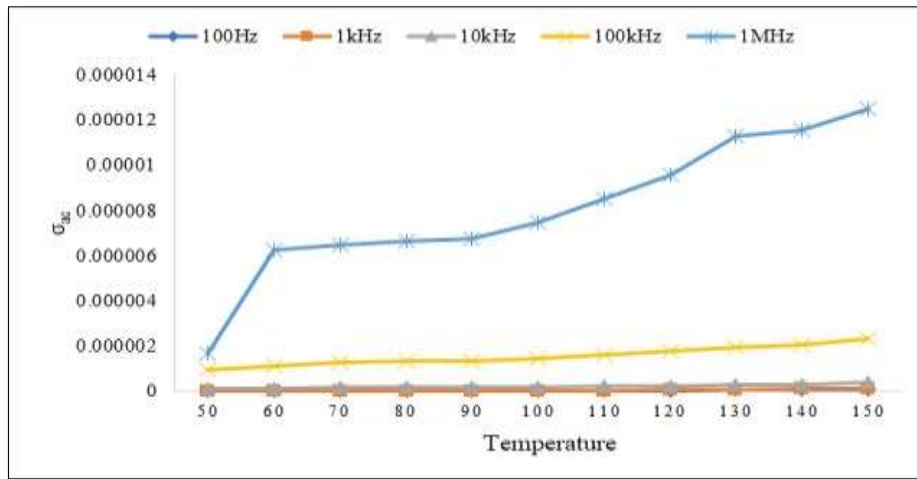


Figure 11: AC Conductivity for ZnO annealed at 200°C

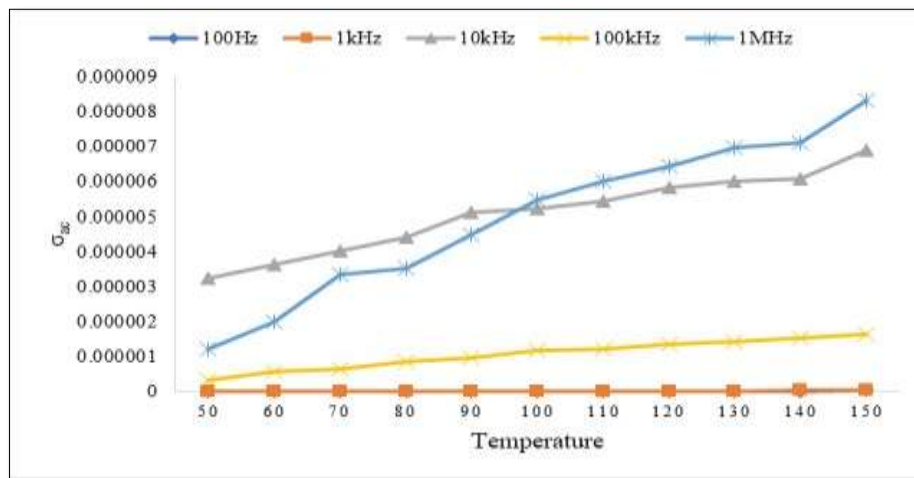


Figure 12: AC Conductivity for ZnO annealed at 800°C

The plotted graph from Fig 10-12 explains the frequency independent conductivity in the low frequency region and frequency dependent conductivity in the mid and high frequency regions respectively. In the low frequency region, nearly uniform value of conductivity represents the continuation of DC conductivity. In the mid and high frequency regions, conductivity increases monotonically with frequency and represents the AC conductivity following the frequency power law [20].

V. CONCLUSION

The ZnO nanoparticles were successfully synthesized through simple sol-gel method and annealed at 200°C and 800°C. The X-ray diffraction pattern confirmed the formation of ZnO with hexagonal structure. The heating technique also influences the size and it shows that there is an improvement in the crystallinity of ZnO nanoparticles which leads to fine quantum confinement with the increase in annealing temperature. The electrical parameters

are measured and the variations with respect to temperature and frequency was evidently analysed.

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