



e-ISSN: 2319-8753 | p-ISSN: 2320-6710

IJIRSET

International Journal of Innovative Research in
SCIENCE | ENGINEERING | TECHNOLOGY

INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

Volume 10, Issue 5, May 2021

ISSN INTERNATIONAL
STANDARD
SERIAL
NUMBER
INDIA

Impact Factor: 7.512

9940 572 462

6381 907 438

ijirset@gmail.com

www.ijirset.com



Controlled Precipitation of Zinc Oxide by Chemical Bath Deposition

Mohite R.M.¹, Mule S.S.²

Assistant Professor, Department of Physics, S.B.Z. College, Barshi, Maharashtra, India¹

Assistant Professor, Department of Mathematics, S.B.Z. College, Barshi, Maharashtra, India²

ABSTRACT: Zinc oxide is prepared by controlled precipitation method from the zinc nitrate solution at 60 °C. After deposition it is annealed at 500 °C for 5hr. The synthesized zinc oxide is characterized by X-ray diffraction (XRD), UV-Visible and Fourier transform infrared (FTIR) spectrometer. XRD patterns shows preferential orientations of the ZnO along (100), (002) and (101) crystallographic planes. XRD confirms that the morphology of ZnO nanostructures (ZnO_{NS}) is polycrystalline in nature. An FTIR spectrum confirms the formation of ZnO_{NS}. The UV-Vis spectra showed absorbance peaks in the 400-1500 nm region. Optical transmittance spectra gives excellent transmittance (>80%) and a sharp cut-off edge at 385 nm. From the spectra, direct band gap of ZnO is determined which is 3.0 eV at room temperature that makes ZnO useful for optoelectronic devices.

KEYWORDS: Nanostructured ZnO, chemical bath deposition, XRD, FTIR, UV-Vis spectra

I. INTRODUCTION

Nanostructured zinc oxide has attracted attention of researchers and scientist in the last few years because of their useful properties in several applications. It is one of the most used inorganic materials due the flexibility of preparation in different morphologies and tunable properties [1]. It has potential applications in optoelectronic devices due to its direct band gap (3.3eV) [2-4]. It also has some unique and interesting physical and chemical properties such as, large exciton binding energy (60meV) [5-6], chemical [7] and thermal stability [8], radiation hardness [9], piezoelectricity and photoelectricity [10], high conductivity, etc. It has abundant in nature and nontoxic. Due to such intriguing properties zinc oxide became a most attractive semiconducting material for the applications in TCO [11], gas sensor [12], LED's [13], solar cells [14], photonic crystals [15], UV-protectors filters [16], chemical sensors [17] and photodiodes [18]. Zinc oxide is a binary (II-IV) n-type semiconductor because of the Zn interstitials and presence of oxygen vacancies. It has high visible transparency and low electrical conductivity [5]. Zinc oxide thin films have been prepared by a verity of thin film deposition techniques such as Chemical Vapor Deposition (CVD) [18-19], Magnetron sputtering [20], spray pyrolysis [21-22], Pulsed Laser Deposition (PLD) [23], sol-gel method [24-26], electron beam evaporation [27]. Among these methods, the CBD method is one of the most important method and widely used for the preparation of metal oxide nanostructures. This is an attractive technique for fabricating uniform thin films due to the homogeneity of precursor, a large-area deposition and low cost fabrication.

In this study, ZnO is prepared using chemical bath deposition method at 60 °C temperature. High quality ZnO was subjected for different characterizations. Structural characterization of the samples is performed using X-ray diffraction (XRD). Optical characterization is carried out by UV-Visible spectroscopy and FTIR spectroscopy. The structural and optical properties of nanostructured ZnO are discussed in this paper.

II. MATERIALS AND METHOD

Analytical grade Zinc nitrate hexahydrate (Zn(NO₃)₂·6H₂O), EDTA, Ethylenediamine (H₂NCH₂CH₂NH₂) (Merck, AR), PEG-600 (FINAR) and double distilled unionized water was used throughout the experiments. Zinc nitrate hexahydrate (15mM) was dissolved in unionized double distilled water at 60°C. 0.5M% EDTA was added to trap impurities present in the solution. Ethylenediamine (15mM) was added to start formation of ZnO nanoparticles. After the slight stirring of solution it was kept at 60 °C for 6hr using water bath. The solution colour was changed from transparent to white after the addition of ethylenediamine. After 6hr of constant heating solution was left to cool for 42 hr in the bath. ZnO material was synthesized. The ZnO was dried by annealing at 500 °C for 5hr. Good quality ZnO was obtained and subjected for different characterizations.

The crystal orientation of the ZnO thin films was studied by Rigaku, Ultima IV diffractometer. Optical absorption was carried out using Shimadzu UV-Vis spectrometer.

III. RESULTS AND DISCUSSION

Typical XRD pattern of the ZnO is presented in Fig 1. All the diffraction peaks are well indexed to the highly c-axis orientated hexagonal ZnO wurtzite structure (JCPDS no. 36-1451). XRD patterns does not show any diffraction peak corresponding to the impurity which confirming the formation of high purity ZnO. ZnO exhibits some characteristic peaks for (100), (002) and (101) planes indicating the polycrystalline nature. The intensity of the peaks increases with sintering temperature which clearly indicating increased crystallinity. The average crystallite size is observed for the samples calcinated at 500^oC and 600^oC are about 65 nm and 147 nm, respectively. The average grain size (D) of ZnO was determined using Debye-Scherrer formula [28].

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

where, λ is the wavelength of X-ray in nm, β is the FWHM in radians

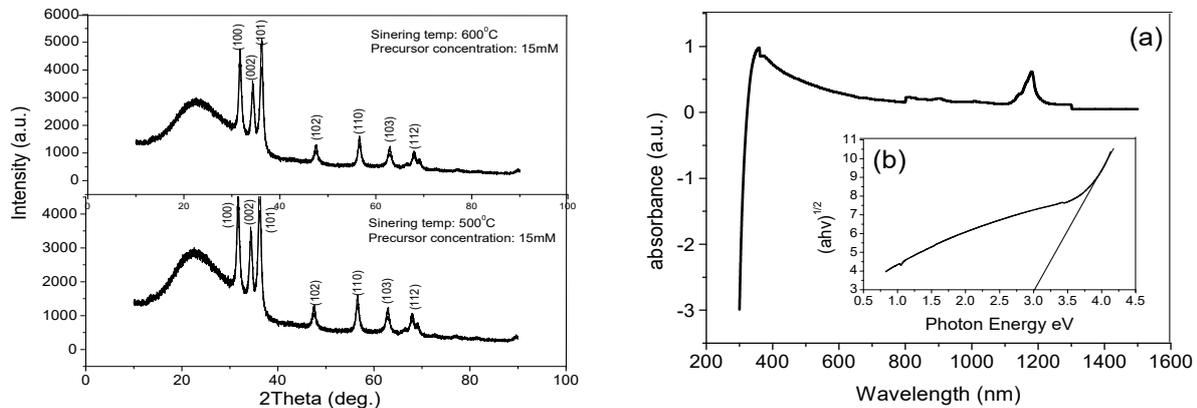


Fig. 1 XRD patterns of ZnO calcined at 500^oC and 600^oC temperatures Fig.2 (a) UV-Vis absorption spectra (b) $(ahv)^{1/2}$ versus photonic energy $h\nu$ plot of ZnO calcined at 500^oC

The relative intensities of the peaks (100), (002) and (101) tend to increase with increase in annealing temperature. It indicates that an improvement in the crystallinity of ZnO with increase in temperature. Crystallization caused by annealing temperature affects the lattice parameters of the material due to oxygen deficiency and strain. Lattice parameters and volume of ZnO primitive cell can be calculated by using Le-Bail and Pawley refinement method,

$$a = \sqrt{\frac{1}{3} \left(\frac{\lambda}{\sin\theta} \right)^2} \tag{2}$$

$$b = \frac{\lambda}{\sin\theta} \tag{3}$$

$$v = 0.866a^2c \tag{4}$$

We also studied the effect of annealing temperature on the bond distance between Zn-O as peak intensities can be affected by annealing temperature we expect the change in bond distance. To see the effect of annealing temperature on Zn-O bond length, Equation (5) is used [29],

$$l = \sqrt{\left[\frac{a^2}{3} + \left(\frac{3}{4} - \frac{a^2}{3c^2} \right)^2 c^2 \right]} \tag{5}$$

where, l is bond length and a & b are lattice parameters.

To obtain the quantitative information about the crystallite orientation along the (002) plane, texture coefficient (T_c) [30] of ZnO annealed at different temperatures was calculated using Equation (6),

$$T_c(hkl) = \frac{[I(hkl)/I_o(hkl)]}{\sum_n [I(hkl)/I_o(hkl)]} \times 100\% \tag{6}$$



where, I is the measured intensity, I_0 is the standard intensity of the plane taken from JCPDS data file.

Table 1. Structural and morphological characterizations of the ZnO films

Sintering Temp. °C	a or b	c	c/a	bond length (nm)	cell volume (nm ³)	Grain size (nm)	Texture Coefficient	Agglomeration size (µm)
500	3.0156	5.2207	1.73	37.6	411.76	65	0.6459	0.5807
600	3.0186	5.2286	1.73	37.7	412.92	147	1.258	0.5824

UV-Vis spectrum is used to determine the optical band gap of nanomaterials. The optical absorption spectrum of ZnO was recorded in the region ranging from 300 nm to 1500 nm using UV-Vis spectrometer (Fig 2 (a)). For the optical device fabrication, the thin film should be highly transparent and conductive. The absorbance of the ZnO is low in the visible wavelength; while high transmission (>80%) obtained in the same region. There is sharp cut off edge near 380 nm due to inter-band transitions. This sharp edge indicates a good structural quality of the synthesized ZnO and makes it a potential candidate for optical device fabrication.

The band gap energy (E_g) of the ZnO can be determined by Tau plot of $(\alpha h\nu)^2$ versus photon energy $h\nu$ (eV). The band gap obtained is found to be 3.0eV as shown in Fig 2(b).

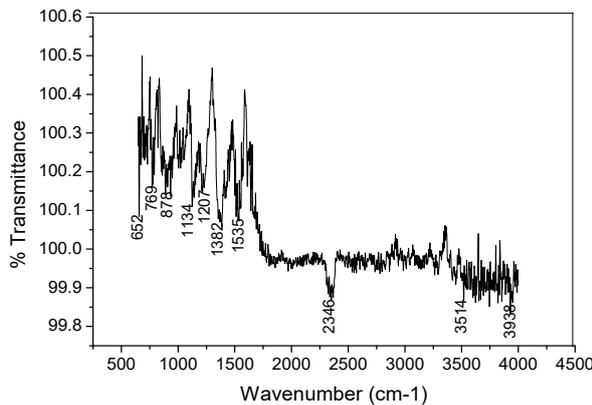


Fig.3 FTIR spectrum of ZnO

Table 2 FTIR spectrum analysis

Bond type	Frequency Range (cm-1)
Zn-O	769
O-H stretching vibration	3000-3600
Oxygen stretching	900-1000
Glass absorption	652
Valence vibrations of the occluded water	3514

The vibration frequencies of functional groups of ZnO have been studied by FTIR spectroscopy. The FTIR spectra were recorded on FTIR spectrometer in the range of 400 cm-1 to 4000 cm-1. Fig 3, shows the FTIR spectrum of the ZnO. FTIR spectra of nanostructured ZnO at room temperature, heated at 500 °C show characteristic peaks at 652, 769, 878, 1134, 2346, 3514 cm-1 and some other associated peaks. The sample heated at 500 °C show the band at 769cm-1 which is assigned to the ZnO band. The band at 3514cm-1 corresponds to valence vibrations of the occluded water. The bands around 3000-3600cm-1 are due to the hydrogen stretching frequency (OH-Stretching) and 900-1000cm-1 are due to the oxygen stretching and bonding frequency. Absorption peaks at 652cm-1 are due to glass substrate. From above FTIR spectrum and its analysis we confirmed the formation of nanostructured ZnO material.

IV. CONCLUSION

In our research work, we have successfully obtained ZnO_{NS} using chemical bath deposition. Structural and optical properties of post-heated ZnO at 500 °C temperature have been studied. XRD pattern reveals that, the synthesized material exhibit hexagonal crystal structure with preferential orientation along (002) crystallographic plane. The structural analysis confirms the polycrystalline nature of ZnO. The change in crystallite size observed with sintering temperature indicating crystallinity increases with sintering temperature. FTIR spectrum confirms the formation pure ZnO nanomaterial. The optical band gap of ZnO material is obtained by UV-Visible study and is found to be 3.0eV. UV-Visible study shows ZnO are applicable for optoelectronic applications.



REFERENCES

- [1] J Rodrigues-Paez, A C Caballero, M Villegas, C Moure, P Duran, J F Fernandez, *J. Eur. Ceram. Soc.* **21**, 925-930 (2001)
- [2] Chopra K L, Major S & Pandya D K, *Thin Solid Films*, **102** 1 (1983).
- [3] S H Jeong, et.al., *Journal of the Korean Physical Society*. **50** 3 622 (2007).
- [4] RM Mohite, RR Kothawale, *Advanced materials research*, 1110, 218-221, (2015)
- [5] T Makino, C H China, T T Nguen, Y Segawa, *Appl. Phys. Lett.* **77** 1632 (2000).
- [6] S J Pearton, D P Norton, K Ip, Y W Hew, and T Steiner, *Superlattices Microstruct.* **34** 3 (2003).
- [7] Min-Chul Jun and Jung-Hyuk Koh, *J. Electr. Eng. Technol.* **8**,1 163 (2013).
- [8] E Shanti, A Banerjee, *Thin Solid Films*. **108** 333 (1983).
- [9] RM Mohite, RR Kothawale, *International Journal of Advanced Scientific and Technical Research*, 2 5 (2012)
- [10] RM Mohite, JN Ansari, AS Roy, RR Kothawale, *International Journal of Nanoscience*, 15(04), 1650011 (2016)
- [11] T T Chen, C L Cheng, S P Fu, Y F Chen, *Nanotechnology*. **18** 225705 (2007).
- [12] Y Y Lin, C W Chen, W C Yen, W F Su., C H Ku., and J J Wu., *Appl. Phys. Lett.* **92**, 233301 (2008)
- [13] RM Mohite, *Studies of doped and undoped nanostructured zinc oxide thin films for solar cell application*, PhD Thesis, Solapur University, 2015.
- [14] D O Dumcenco, et al., *J. Alloys Compd.* **491** 462 (2010).
- [15] R M. Mohite, R R Kothawale, *Indian J Chem.* 54A, 872-876 (2015)
- [16] E W Seelig, B Tang, A Yamilov, H Cao and R P H Chang, *Mater. Chem. Phys.* **80** 257 (2003).
- [17] R M Mohite, *Indian Journal of Applied Research*, 6 10, 248-250 (2016)
- [18] F T Liu, S F Gao, S K Pei, S C Tseng, C H J Liu, *J Taiwan Inst Chem Eng.* **40** 528 (2009).
- [19] S J Chang, et al., *Nanotechnol. IEEE Trans.* **11** 127 (2012).
- [20] Y J Li, R Duan, P B Shi, G G Qin, *J. Cryst. Growth.* **260** 309 (2004).
- [21] S Takeda and M Fukawa, *Thin Solid Films.* **468** 234 (2004).
- [22] A F Aktaruzzaman., G L Sharma., *Thin Solid Films.* **198** 67 (1991).
- [23] A A Yadav, M A Barote, E U Masumdar, *Mater. Chem. Phys.* **121** 53 (2010).
- [24] H S Kang., J S Kang., J W Kim., and S Y Lee., *J. Appl. Phys.* **95** 1246 (2004).
- [25] RM. Mohite, *International Journal of Advanced Scientific Research and Technology*, 2 2 500-505, (2012)
- [26] Mohite RM, Kothawale RR, Roy AS, *Int. Res. Journal of Science & Engineering*, A5, 87-89 (2018)
- [27] A Kuroyangi, *Jpn. J. Appl. Phys.* **28** 219 (1989).
- [28] C C Chen, P Liu, C H Lu, *Chem. Eng. J.* **144** 509-513 (2008).
- [29] B D Cullity, *Elements of X-ray diffractions*, Addison-Wesley, Reading, MA (1978).
- [30] M. Benhaliliba, C E Benouis, M S Aida, F Yakuphanoglu, A S Juarez, *J. Sol-Gel Sci. Technol.* **55** 335 (2010)



INNO  **SPACE**
SJIF Scientific Journal Impact Factor

Impact Factor:
7.512

ISSN INTERNATIONAL
STANDARD
SERIAL
NUMBER
INDIA



INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

 **9940 572 462**  **6381 907 438**  **ijirset@gmail.com**



www.ijirset.com

Scan to save the contact details