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Structural, UV and IR Study of ZnO Nanoparticles Synthesized by Hydrothermal Process

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Abstract:

Nanocrystalline Zinc oxide was synthesized by hydrothermal process without any calcination at high temperature. In this technique the Zinc Acetate, ethanol and sodium hydroxide is used. The ZnO nannoparticles were characterized by XRD, UV and FTIR. The average grain size, packing fraction, volume density, lattice parameter and Williamson grain size of prepared ZnO nano powder is determined by XRD. Crystalline ZnO with a hexagonal wurtzite is confirmed by XRD results. Absorption spectra of Ultra Violet waves are indicating the smaller size of ZnO nanoparticles by revealing the absorption at wavelength <400 nm.

Keyword: ZnO, Hydrothermal, Particle Size, Band Gap.

Introduction:

By varying the size of particle, the electrical, optical and chemical properties of semiconductors can be tuned [1]. The surface area and morphology (how the crystals are stacked) also plays an important role in many applications (such as photoemitters, transducers, actuators, varistors, sensors and catalysts). Due to a wide range of applications ZnO has become very popular versatile semiconductor [2]. Zinc Oxide (ZnO) is a material with direct band gap (3.37eV at room temperature) and large exciton binding energy of 60meV. Zinc oxide (ZnO), a direct wide bandgap*II-VI* compound n-type semiconductor, has a stable wurtzite structure with lattice spacing a = 0.325 nm and c = 0.521 nm and composed of a number of alternating planes with tetrahedrally-coordinated O2- and Zn2+ ions, stacked alternately along the c-axis [3]. It has attracted exhaustive research effort for its unique properties and versatile applications in transparent electronics, piezoelectric devices, ultraviolet (UV) light emitters and chemical sensors.

There are many existing techniques for preparation of ZnO. Among them, it is well conceived that preparation of ZnO via hydrothermal process provides a promising option for production of better material [4]. There are several advantages of hydrothermal process over other growth processes such as use of catalyst-free growth, low cost, simple equipment, large area uniform production, environmental friendliness and less hazardous.

Experimental Work

Material preparation

The reagents used for the reaction are analytical grade and without further purification before utilization. To prepare ZnO nanoparticles, 100ml solution of zinc acetate dehydrate [Zn $(CH_3COO)_2$. H_2O] (0.1M) and 50ml solution of NaOH (0.5 M) were prepared in ethanol under continues stirring. Then 50ml solution of NaOH is added drop wise in 100ml zinc acetate dehydrate under continues stirring till

obtain 10 pH value. This solution was packed in round bottom flask, kept into stainless steel autoclaves system and maintained at 110°C temperature for 10hr under autogenous pressure. The reaction then kept for cooling naturally at room temperature. Finally the white precursor was collected after the completion of reaction; the resulting white solid products were washed with ethanol, filtered and then dried in air in a laboratory oven at 55°C.

Characterization

The X-ray diffraction data of synthesized ZnO sample were recorded on Bruker D8 Advance Diffractometer with Cu-Kα1 radiation (λ=1.5418 Å) and collected over the range 20°<2θ<80° at room temperature. The optical band gap of synthesized sample is calculated from data recorded by UV-Vis – Spectrophotometer (Shimadzu UV-1800 UV-Vis Spectrophotometer). FTIR spectra of ZnO sample is carried out by using PerkinElmer Spectrum (88522) model.

Result and Discussion:

The X-ray diffraction pattern of ZnO sample shown in figure 1 exhibit a dominant peak at 2θ = 35° corresponding to the (101) plane of ZnO. XRD pattern shows distinct diffraction peaks corresponding to the (100), (002), (102), (110) lattice plane reveals that it has hexagonal wurtzite structure without any impurity phases and confirm that the resulting products have high crystallinity of ZnO sample.

The peak and relative intensities obtained for the ZnOclose to the reported data (JCPDS 01-075-1533).The structural data obtained from X-ray patterns are shown in table 1. The lattice parameters of ZnO sample is obtained a = 3.3278 & c = 5.2498. The values of lattice parameters are close to those reported in JCPDS data (JCPDS 76-0704 a=3.250Å, c=5.207Å). The X-ray density of ZnO sample was calculated using the formula [5].

$$D_x = \frac{nM}{N_A V} \qquad \text{---------- (1)}$$

Where, D_x is X-ray density, n is the number of atoms per unit cell, M is the molecular weight of the sample, N_A is Avagadro's number and V is the volume of the unit cell.

The crystallite size (D) was calculated from line broadening of the major XRD peak using the Scherrer's formula [6].

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad \text{---------- (2)}$$

Where, K is the shape factor, which is a constant taken as 0.9, λ is the wavelength of the X-ray radiation, β is the full-width at half-maximum (FWHM) in radians, θ is the Bragg's angle in degree. The crystallite size of the ZnO sample obtained from eq (2) is listed in table (1). It is found that the sample synthesized by co precipitation route has grain size ~ 20.33 nmand are in good agreement with value reported in literature [7]for pure ZnO. The atomic packing fraction was determined and listed in table 1. The obtained value of atomic packing fraction is in good agreement with the standard wurtzite hexagonal structure.

The plot of W-H analysis for ZnO sample based on the UDM model is shown in figure 2. The obtained crystalline size using UDM model is 24.79 nm which is good agreement with crystalline size obtained by Debye Scherrer formula. The strain produced in the prepared ZnO sample is tabulated in table 1. This lattice strain $\varepsilon = 9.1090 * 10^{-4}$ produced in the sample causes the peak broadening.

UV-vis Spectroscopy:

The optical band gap of synthesized sample is calculated from data recorded by UV-vis – Spectrophotometer (Shimadzu UV-1800 UV-vis Spectrophotometer). The UV-vis spectra of ZnO sample recorded in the wavelength range 200-800nm at room temperature as shown in figure 3. The band gap was calculated by plotting the absorption plot $(\alpha h\upsilon)^2$versus (Energy, E) shown in figure 3. The energy bandgap is found through figure 4 is 3.32 eV.

FTIR:

FTIR spectra of ZnO sample is carried out by using PerkinElmer Spectrum (88522) model. The FTIR spectra for grown ZnO sample assigned at room temperature shown in figure 5. The broad peak in higher energy region at 3271.36 cm^{-1} is due to O-H stretching and peak in the lower range at 1682.23 C=N stretch, and 1639.67 due to C=O stretch. All other peaks are attributed to the characteristic of the prepared ZnO nanoparticle. The FTIR spectrum of the main transmission band is due to Zn-O stretching of ZnO in the range of 574.29 – 589.77 cm^{-1}.

Table. 1-Lattice parameter, volume of unit cell, x-ray density, c/a ratio, grain size, atomic packing fraction, strain and energy ban gap of ZnO.

Sample	Lattice parameters		Volume cell $(A^o)^3$	X-ray density (g/cm^3)	c/a Ration	Atomic packing fraction	Scherrers Formula D (nm)	UDM		Bandgap (eV)
	a (A^o)	c (A^o)						D(nm)	$\varepsilon^* 10^{-4}$	
ZnO	3.3278	5.2498	152.46	5.32	1.577	0.76	20.33	24.79	9.1090	3.27

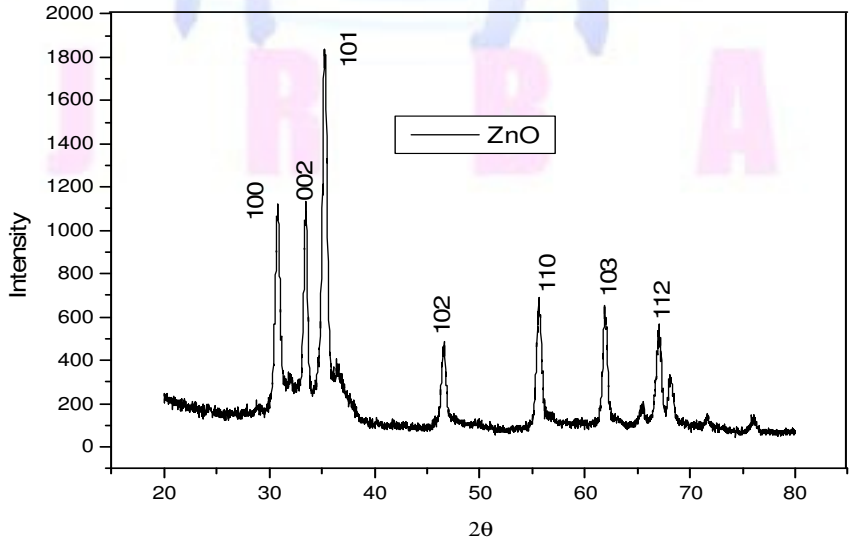

Figure. 1- XRD pattern of ZnO

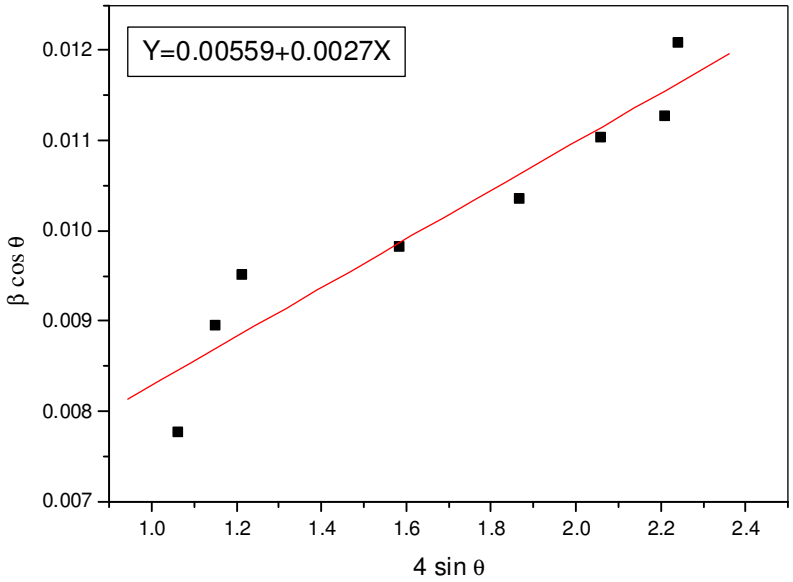

Figure 2: The W-H plot of $\beta_{hkl}cos\theta$ versus 4 $sin\theta$ for ZnO samples

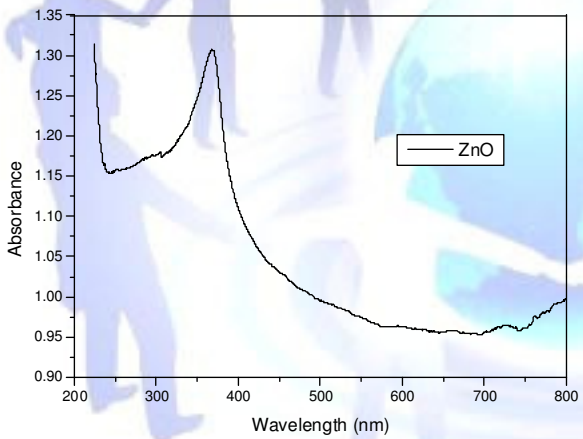

Figure 3: UV graph of ZnO

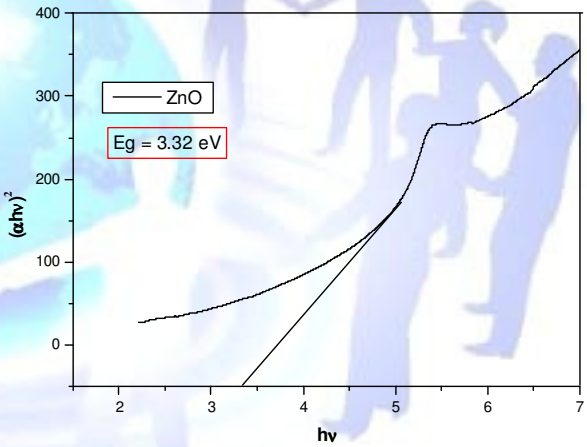

Figure 4: (αhυ)²vshυ of ZnO

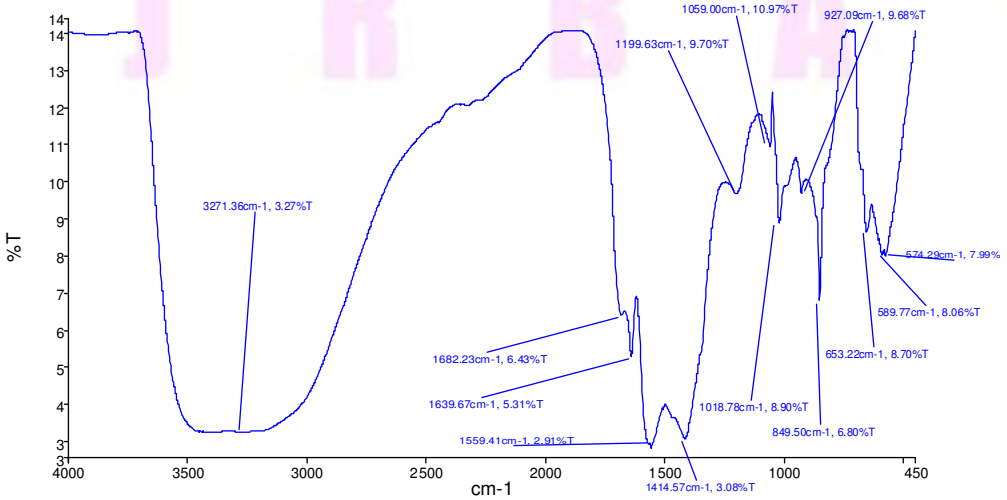

Figure 5: FTIR spectra of ZnO

Conclusions:

Hydrothermal method is very simple and low cost technique for synthesis ZnO. The crystal structure, lattice parameters, atomic packing fraction, strains and phase purity of the sample are confirmed through XRD analysis. The energy bandgap of prepared material is found nearly equal to bandgap of ZnO mention in literature. The Zn-O bonding in the ZnO nanoparticle is confirmed by FTIR analysis.

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