SYNTHESIS AND FABRICATION OF ZINC OXIDE ON FTO SUBSTRATES BY SPIN COATING METHOD

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Abstract

ZnO plays an important role in many semiconductors technological aspects. In the present work zinc oxide particles were prepared by wet chemical method, using ($Zn(NO_3)_2.6H_2O$) and (NaOH) with different concentrations (0.25,0.5,0.75, and,1)M and study the effect of temperature on the structures and particles sizes. ZnO nanoparticles synthesized with 1 M of NaOH at 80 ºC. ZnO particles were characterized by using X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

Key words: $Zn(NO_3)_2.6H_2O$, NaOH, ZnO, Nanoparticles, XRD SEM and EDS

Introduction

Zinc Oxide (ZnO) is a wide band gap semiconductor with wurtzite structure. The physical and chemical properties of nanoscale particles are different when they are compared with the bulk materials. The ZnO is known as a wide band gap (3.37 eV) with a high exciton binding energy (60 meV) and exhibit the most sheeny and numerous configuration of nanostructure that one material can form. Also, ZnO as an important semiconducting material has a wide range of applications in gas sensors, chemical absorbent, nanogenerators, electrical and optical devices, electrostatic dissipative coating, and advanced ceramics. ZnO nanoparticles have been prepared by various methods such as thermal decomposition, solvothermal reaction, reactive electron beam, evaporation technique, chemical vapor deposition, hydrothermal method. Due to the simplicity, versatility and low cost of this route, the solochemical method is a process extremely viable for industrial production of zinc oxide. Among control of the particle size is one main concerned for nanostructured material synthesis because electrical and optical properties of nanomaterials depend on both size and shape of the particles. Therefore, it is desired to synthesize nanomaterial in a controllable size by a simple approach.

Materials and Methods

Starting Materials

The materials used in this study were Zinc nitrate hexahydrate, Zn $(NO_3)_2.6H_2O$ with different concentration (0.25 ,0.5 ,0.75 .and 1) M of Sodium hydroxide, NaOH and Fluorine doped Tin Oxide (FTO) glass.

Preparation of particle

The wet chemical method was used to prepare zinc oxide (ZnO) particles. using zinc nitrate hexahydrate and sodium hydroxide as precursors. (0.25, 0.5, 0.75 and 1) M of NaOH which prepared by dissolving (40gm) of NaOH in 1liter (1000 ml) of deionized water. The solution was stirred at 70ºC. 0.5M of $Zn(NO_3)_2.6H_2O$ was prepared by dissolving (37.175 gm) $Zn(NO_3)_2.6H_2O$ in (250 ml) of deionized water, and then added to the basic solution drop by drop, the solution was keeping stirring and heating for 3h at the temperature of 80 ºC .After the completion of reaction, the solution was allowed to settle for over night and after that the supernatant solution was

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discarded carefully. Thus produced particles were washed five times using deionized water. The solution was coated on the Fluorine doped Tin Oxide (FTO) glass by using spin coating machine with 500 rpm for 10 minutes. Then, the fabricated sample was dried in oven at 80°C for overnight, and calcined at 200 ºC, 300 ºC and 400ºC for 2h in a muffle furnace. Finally, the last sample was prepared with 1 M of NaOH at temperature 80 °C. The flow diagram of preparation of zinc oxide (ZnO) was shown in Figure 1.

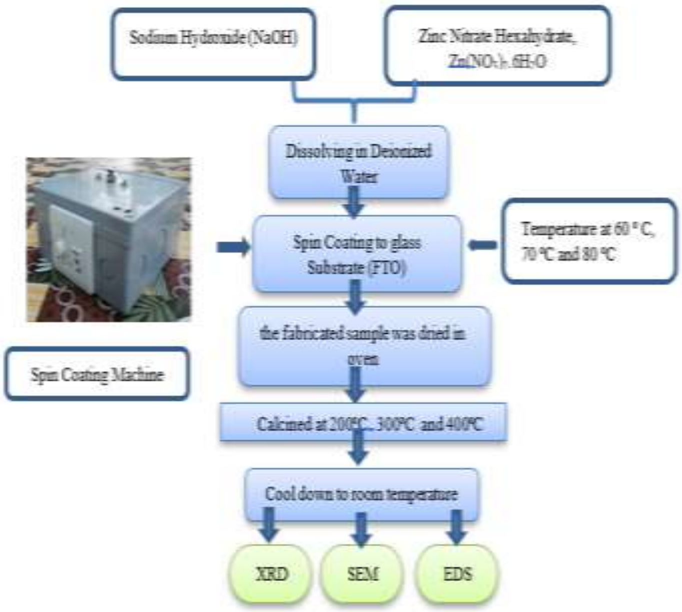


Figure 1 Block Diagram of experiment procedure

Methods of Characterization

X-ray diffraction (XRD)

The crystallinity and phases of the ZnO nanostructures were characterized by X-ray Diffractometer, XRD-6000, Shimazu, Japan with Cu-k_α radiation (λ= 1.5412 A), 40kV, 30mA) in the 2° range of 10°-80° with 2° /min scanning rate. The crystallize size (D) of selected samples were estimated using the Scherer's equation,

$$G = \frac{\beta\lambda}{\mathrm{FWHM}\cos\theta} \qquad (1)$$

where β = Scherrer constant ≅ 0.899

G = crystallite size (nm)

λ = wavelength (λ= 1.5412 Å)

FWHM = full width at half maximum (rad)

θ = Bragg angle (deg)

Scanning Electron Microscopy (SEM)

The morphological feature of the nanostructures was observed by a scanning electron microscope SEM-EDS. JCM-6000, JEOL. The surface morphology of Zinc Oxide samples and the size of particles can be magnified from 10 x to 600,00 x for secondary electron images and from 10 x to 300,00 x for backscattered electron images. The samples for SEM were coated with gold.

Energy-dispersive spectroscopy (EDS)

Energy-dispersive X-ray spectroscopy (EDS, also abbreviated EDX or XEDS) is an analytical technique that enables the chemical characterization/elemental analysis of materials. SEM-EDS analysis is a non-destructive analytical technique (to the sample), but unlike XRF that can be undertaken in-situ without sample removal, SEM-EDS does require sample removal. The sample material is irradiated with electrons resulting in the emission of x-rays characteristic to the elements present. The EDX detector detects the X-rays which are produced by the material when electrons interact with its surface during SEM imaging.

Results and Discussion

X-ray Diffraction (XRD) Measurement

The structural study was determined by the X- ray diffraction performed with a Multiplex's Diffractometer (model: RIGAKU-RINT 2000). XRD pattern was utilized to explore and recorded as the crystalline structures. According to Figures, the XRD patterns of the ZnO films deposited at different temperatures of 60 ºC, 70 ºC and 80 ºC. It is observed that the nanostructures of zinc oxide (ZnO) thin films exhibit hexagonal structure with c/a ratio of about 1.63. It was observed that increasing in substrate temperature shows slightly increase in crystallite sizes and higher peak intensities, due to high crystallinity. The ZnO films exhibit highly (002) oriented nanocrystalline structure. The average crystalline size of ZnO was calculated according to Scherer's equation and the result was found to be 30.996 nm. Table 1 shows the XRD analysis result for ZnO thin films.

Table 1 XRD analysis result for ZnO thin films

Temperature (°C)	Plane (hkl)	FWHM Peak (degree)	2-Theta Value	d (Å)	Crystallite Size (nm)
200	002	0.271	34.685	2.611	30.677
300	002	0.272	34.673	2.621	30.875
400	002	0.276	34.669	2.675	31.436

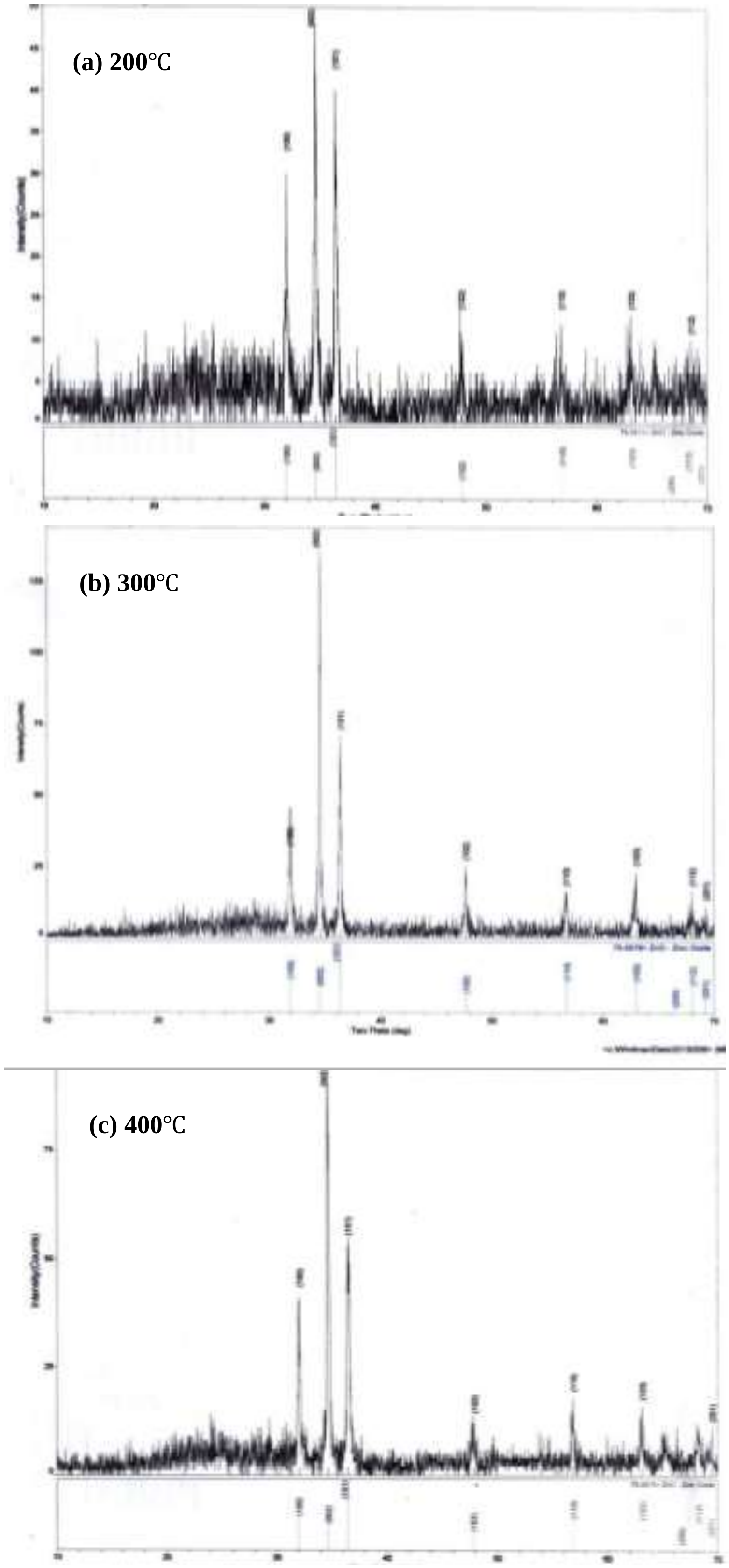
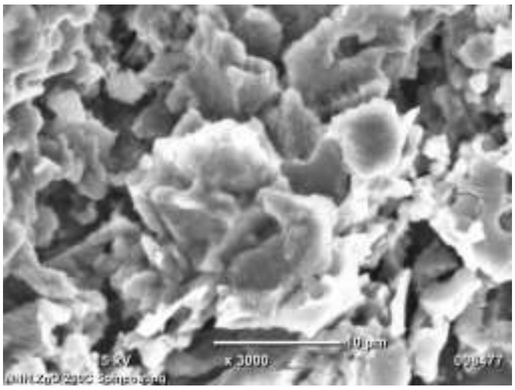


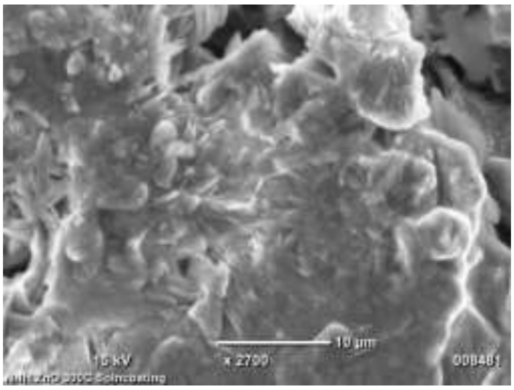
Figure 2 XRD spectra of ZnO thin films at (a) 200℃ (b) 300℃ (c) 400 ℃

SEM Analysis of Zinc Oxide (ZnO) Nanoparticles

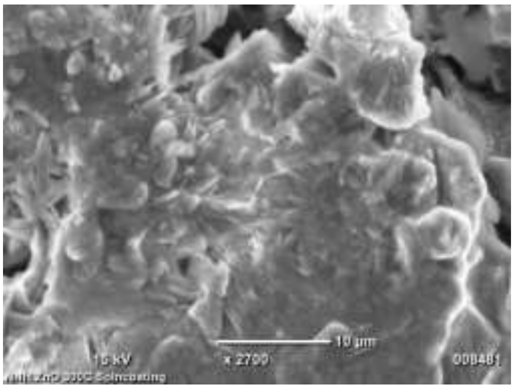
The surface morphologies by SEM observation have been characterized using JEOL Scanning Electron Microscope model JCM-6000, SEM-EDS type. In spite of such imperfection, some zinc oxide (ZnO) were well preserved and could be able to identify under SEM. According to Figures, ZnO have been successfully prepared by Spin Coating method and the shape and size are well uniformed.



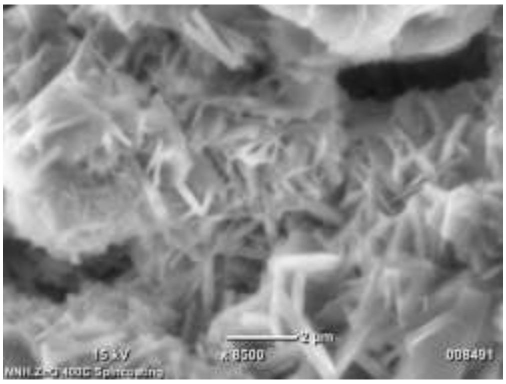
Magnified (x 3000) at 200°C



Magnified (x 2700) at 300°C



Magnified (x 2200) at 400°C



Magnified (x8500) at 400°C

Figure 3 SEM Images of ZnO at different magnification

SEM imaging to EDS Analysis

Energy Dispersive Spectroscopy (EDS) is capable of obtaining rapid qualitative chemical information, semi-quantitative composition determinations, maps showing lateral distribution of chemical elements, and compositional profiles across a surface. All stable elements can be detected with the exception of hydrogen, helium, and lithium. According to figure, the mass percentage of oxygen and zinc element in zinc oxide compound were observed by EDS analysis.

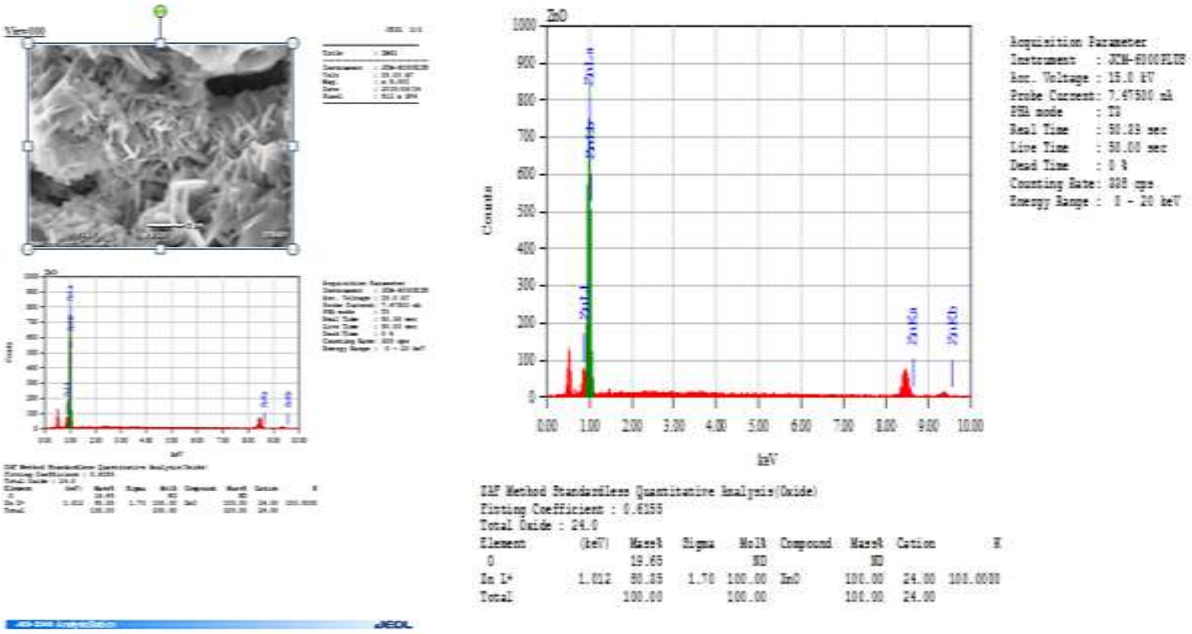


Figure 4 SEM-EDS Images of ZnO

Conclusion

Zinc oxide nanostructures were prepared by the combination of Zinc Nitrate Hexahydrate with sodium hydroxide. The XRD analysis confirmed that the ZnO nanoparticles have the hexagonal wurtzite structure. The ZnO films exhibit highly (002) oriented nanocrystalline structure. The average crystalline size of ZnO was calculated according to Scherer's equation and the result was found to be 30.996 nm. The morphology was evaluated using SEM. At 200°C, the SEM image of ZnO thin film was homogenous. The topography of the surface shows the microstructure which looks like a cluster of thread. At 80°C, the crystals are well in order and the grain sizes of the films were found to be within the range for 2 µm to 10 µm. In SEM-EDS image, the mass percentage of oxygen element was found in 9.65% and zinc element in 80.35%. Zinc Oxide compound was found in 100%. The crystallized sizes of ZnO nanoparticles presented in this study leads to the possibility of several applications in nanotechnology, such as advanced ceramics, solar cells and antimicrobial agent.

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