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The Effect of Pre-heating Temperature on Structural and Optical Properties ZnO Thin Film Synthesized using Sol-Gel spin Coating Method

Nurdin Siregar¹ and Motlan²

Faculty of Mathematics and Natural Science State University of Medan

Email: nurdinfis@gmail.Com

Abstract. The synthesis of ZnO thin film has been successfully carried out using the sol-gel spin coating method. The production of ZnO thin film was grown on a glass substrate with a rotation speed of 5000 rpm and calcined with pre-heating temperature variations of 250, 300 and 350°C and post-heating at a temperature of 550°C. The results of the characterization of ZnO thin films with XRD showed that all crystal structures in the form of hexagonal wurtzite with the largest crystal size for 350°C pre-heating temperature were 35.3 nm. Morphology of ZnO thin films resulting from SEM characterization in the form of crystallite granules and most of them are round and almost uniform and compact. The results of the characterization of ZnO thin film with UV-Vis showed the highest transmittance value for 350°C pre-heating temperature was 53.9%. The highest absorbance value for 250°C pre-heating temperature is 1,192. The biggest energy band gap value for 350°C pre-heating temperature is 3.15 eV.

Keywords: Pre-heating, structure, optical properties of ZnO thin films, Sol-gel Spin Coating Method

1. Introduction

Zinc Oxide (ZnO) which usually appears as a white powder, is almost insoluble in water. ZnO is II-VI type-n semiconductor material with an energy band gap of 3.37 eV and excitation binding energy of 60 meV in room temperature and one type of metal that is in great demand in various types of applications such as sensors, solar cells and nanodivices [1 - 3]

ZnO thin films were synthesized by several methods such as pyrolysis [4], chemical bath deposition [5], physical vapor deposition [6], sol-gel dip coating [3] and sol-gel spin coating [7]. The advantage of the sol-gel spin coating method is that it is very easy and effective, cheap, does not use high vacuum. The other advantageous of the sol-gel spin coating are homogen, good microstructure, and the thickness of the layer can be controlled [8]. The ZnO thin film was also grown using zinc acetate, 2-methoxyethanol and MEA with variations in pre-heating temperature (100 and 300°C). This experiment produced a hexagonal wurtzite and the largest crystal size of 50 nm at a temperature of 300°C [9]. Other studies using zinc nitrate hexahydrate, isopropanol and MEA with variations in pre-heating temperature (300, 400, 500 and 600°C) increased crystallite size from 50-150 nm by increasing pre-heating temperature [10].



2. Experiment

Sol-gel synthesis was carried out by sol-gel method by dissolving Zinc acetate dehydrate 4.0 grams into isopropanol 47.22 ml to obtain a concentration of 0.8 M and then stirring with a magnetic stirrer at a temperature of 60 – 85°C at a rate of 60 - 70 rpm. Diethanolamine (DEA) as much as 1.72 ml as stabilizer was put into solution. The molar ratio between DEA and ZnAc is 1: 1. After the liquid gel is formed, then the solution is left to form a thicker solution that resembles a "gel". Gel-like solution is then dripped on a glass substrate of 5 drops with a 5000 rpm spin coating speed and coatings up to five times. Pre-heating heating was carried out with temperature variations of 250, 300 and 350°C for 5 hours with a holding time of 15 minutes. Post-heating is carried out at a temperature of 500°C with a holding time of 15 minutes.

3. Results and Discussion

3.1 Crystal Structure of ZnO Thin Films

The diffraction pattern of X-ray diffraction (XRD) samples of ZnO thin film synthesized by sol-gel spin coating method by mixing reflux technique for pre-heating temperatures of 250, 300 and 350°C is shown in Figure 1. The figures show that all samples have the same crystal fields, namely fields (100), (002) and (101). All growth peaks are oriented to the plane (101) and hexagonal ZnO crystals. The highest peak intensity (count) for samples with a pre-heating temperature of 300°C is 21846.

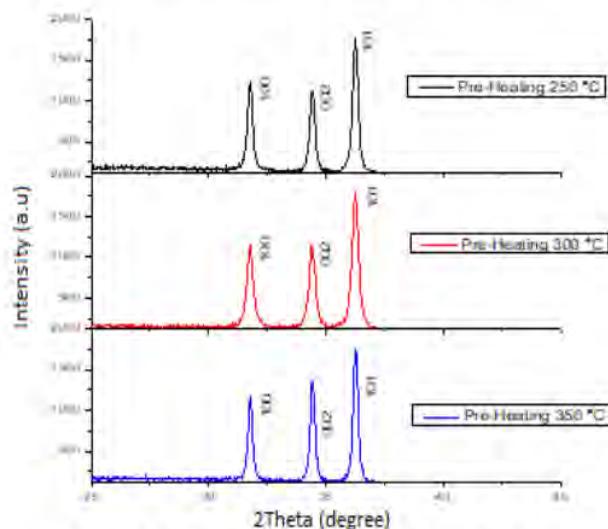


Figure 1. XRD spectrum of ZnO thin films with pre-heating temperature variations

The lattice parameters a and c of ZnO which have hexagonal wurtzite crystal structure are calculated by the equation [11]:

$$a = \sqrt{\frac{1}{3} \frac{\lambda}{\sin \theta}} \quad (1)$$

$$c = \frac{\lambda}{\sin \theta} \quad (2)$$

Crystal lattice parameters for pre-heating temperatures of 250, 300 and 350°C are $a = 3.2490 \text{ \AA}$, $c = 5.2070 \text{ \AA}$; $a = 3.2494 \text{ \AA}$, $c = 5.2038 \text{ \AA}$ and $a = 3.2490 \text{ \AA}$, $c = 5.2038 \text{ \AA}$. The ratio of c / a ZnO thin film crystal to pre-heating temperatures of 250, 300 and 350°C is 1.602; 1.601 and 1.601. The ratio c / a for all pre-heating temperatures has the same value as the ideal value for hexagonal cells $c / a = 1.602$ [12]

The crystal size of ZnO for the pre-heating temperature variance was obtained using the Scherrer equation [13]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (3)$$

where D is the crystal size, λ is wavelength, β is FWHM (full width half maximum), θ is the diffraction angle and a , c is the lattice parameter, the results of which are shown in Table 1

Table 1. ZnO thin film crystal size with pre-heating temperature variations

Pre-heating temperature (°C)	Phase	Peak		Crystal size (nm)
		2θ (degree)	FWHM(degree)	
250	ZnO	36,2293	0,2586	26,0
300	ZnO	36,2242	0,3219	32,4
350	ZnO	36,2380	0,2367	35,3

Based on Table 1 obtained the relationship between temperature pre-heating with crystal size and FWHM value as in Figure 2 and Figure 3.

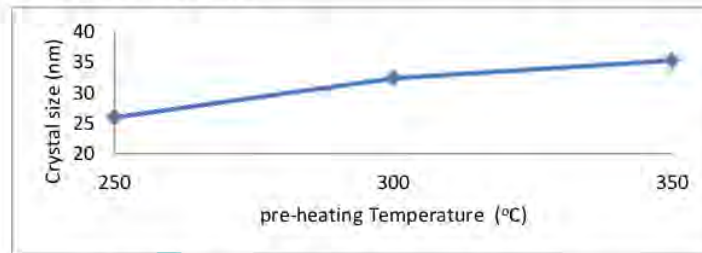


Figure 2. Effect of pre-heating temperature on crystal size

The increase in pre-heating temperature along with the increasing size of ZnO thin film crystals as shown in Figure 2. The figure shows that the heating temperature (pre-heating) affects the properties and size of the crystals. The increase in pre-heating in ZnO thin film growth will accelerate the evaporation process in removing solvents, water and acid groups, and accelerate the process of changing ZnOH to ZnO (formed crystal morphology). According to the results of previous studies that the increase in pre-heating temperature along with the increase in crystal size. Increased pre-heating temperature along with increasing crystal size [9,10]. Figure 3 shows that the value of FWHM (Full width Half Maximum) or width of the XRD spectrum increases at 300°C, and then decreases at 350°C.

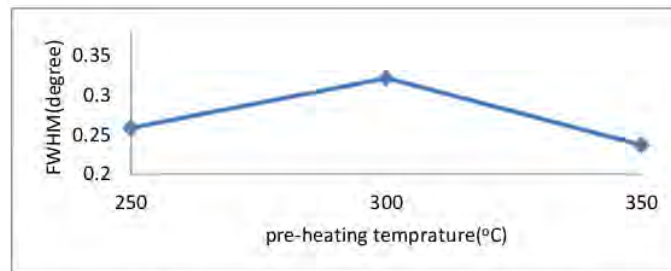


Figure 3. Effect of temperature pre-heating on FWHM values

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 3.2 *Optical Properties of ZnO Thin Films*

The spectra of transmittance and absorbance of ZnO thin film samples with variations in pre-heating temperature from the UV-Vis test are shown in Figure 4 and Figure 5.

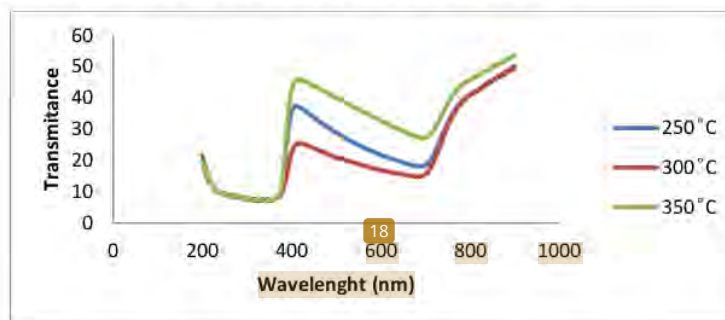


Figure 4. Transmittance spectrum for samples with variations Pre-heating of 250, 300 and 350°C

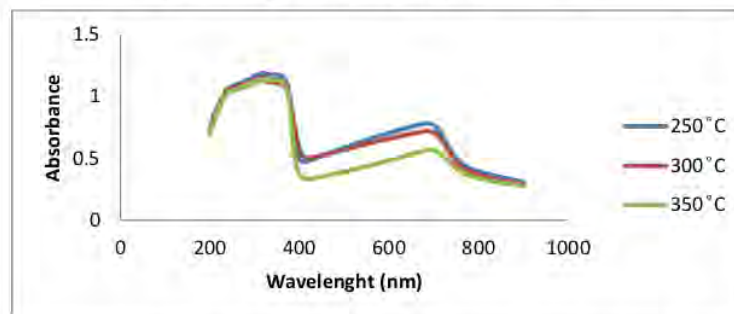


Figure 5 . Absorbance spectrum for samples with variations Pre-heating of 250, 300 and 350°C

Transmittance spectrum curves for samples with pre-heating temperatures of 250, 300 and 350°C shown in Figure 4 show a sharp increase in transmittance values in the wavelength range 350 nm to 400 nm and stable at a wavelength of <350 nm which is in an ultraviolet wavelength region. Figure 5 shows a sharp decrease in the absorbance value for all samples which occurs in the wavelength range 350 nm to 400 nm. In the UV region which is at a wavelength of <350 nm which is an area, the absorbance value is stable.

These results indicate that the increase in pre-heating in ZnO thin film growth, will increase the evaporation speed in removing solvents, water and acid groups, and accelerate the process of changing ZnOH to ZnO (crystalline morphology formed). This will also increase transmittance values [14].

The energy gap width of the ZnO thin film band with variations in pre-heating temperatures of 250, 300 and 350°C is obtained from the slope of the straight line fitting as shown in Figure 6. Figure 6 shows the energy band gap of samples grown at each temperature and also given in Table 2.

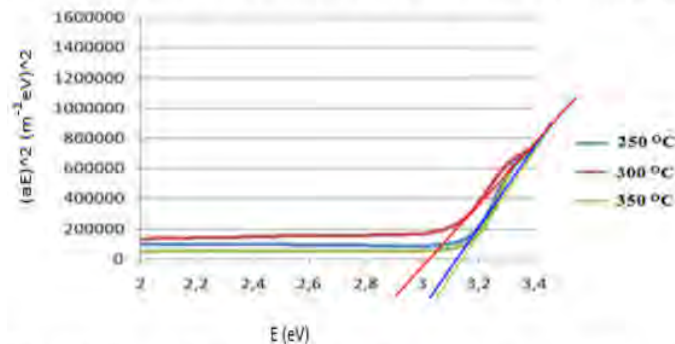


Figure 6. Curve $(\alpha h\nu)^2$ as a function of energy variation in pre-heating temperature

Table 2. Energy band gap of ZnO thin film for pre-heating temperature

Pre-heating temperature (° C)	Energy band gap (eV)
250	3,12
300	3,02
350	3.15

From Table 2, the relationship between pre-heating temperature and energy band gap is shown in Figure 7.

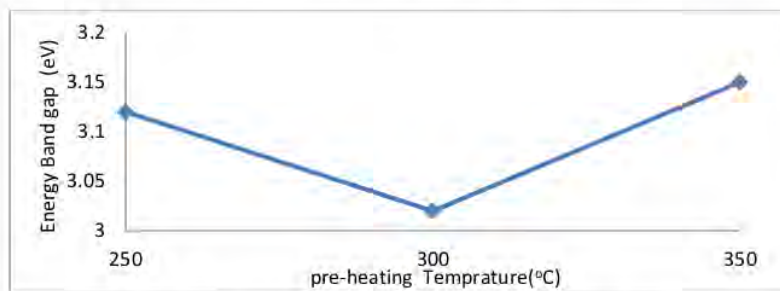


Figure 7. Effect of pre-heating temperature on the width of the energy band gap

The value of energy band gap decreased at 300°C and increased at 350°C pre-heating temperature as shown in Figure 7. The results showed that with increasing heating temperature along with the increase in energy band gap. This is due to an increase in heating temperature which will accelerate the formation of crystals and its band gap.

4. Conclusion

The ZnO thin films on glass substrate synthesized by sol-gel spin coating method and mixing with reflux technique are hexagonal wurtzite, the largest crystal size is 35.4 nm for pre-heating temperature 350°C. The highest transmittance value is 53.9% for the pre-heating temperature of 350°C rotation speed of 3000 rpm. The highest absorbance value is 1.192 for the pre-heating temperature of 250°C, and the smallest band gap is of 3.15 eV for the pre-heating temperature of 350°C. Pre-heating temperature affects the structural properties and optical properties of ZnO thin films

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