International Journal of Sciences: Basic and Applied Research (IJSBAR)

ISSN 2307-4531
(Print & Online)

http://gssrr.org/index.php?journal=JournalOfBasicAndApplied

The Effect of Concentration of Structure and Optical Properties of Thin Films Synthesized by Sol-Gel Methods Spin Coating

Nurdin Siregar[a*], Eddy Marlianto[b], Saharman Gea[c], Motlan[d]

[a,d] *Faculty of Mathematics and Natural Science State University of Medan*
[b,c] *Faculty of Mathematics and Natural Science University of Nort Sumatera*
[a,b] *School of Physics Magister Program University of Nort Sumatera*
Email:nurdinfis@gmail.com

Abstract

ZnO thin films have been successfully synthesized by the method of sol-gel spin coating and mixing processes reflux technique. Materials used Zinc acetate dehydrate Zn $(CH_3COOH)_2$ $2H_2O$, isopropanol and diethanolamine (DEA) are respectively as base material, solvent and stabilizer. Zinc acetate dehydrate Zn $(CH_3COOH)_2$ $2H_2O$ with various concentration of 0.6 M; 0.7 M and 0.8 M dissolved by the solvent isopropanol was then stabilized with diethanolamine (DEA). The molar ratio between the DEA and ZnAc is 1: 1. ZnO thin films on a glass substrate made by spin-coating technique with a rotation speed of 5000 rpm, for 30 seconds and calcination performed by pre-heating temperature of 300^{o}C and 500^{o}C annealing temperature. The results of the characterization of ZnO thin films by XRD indicates all hexagonal shaped crystal structure and the small crystal size of 25.4 nm for a concentration of 0.6 M. The results of the characterization of ZnO thin films by UV-Vis showed the highest transmittance value of 59.0% for concentrations of 0.7 M and the value of the energy band gap of 3.13 eV smallest to the concentration of 0.8 M.

Keywords: Structure; optical properties of ZnO thin films; Concentration.

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* Corresponding author.
E-mail address: nurdinfis@gmail.com.

1. Introduction

Zinc oxide is an inorganic compound with the formula ZnO. In the natural form of the mineral zincite ZnO occurs as a white powder commonly known as zinc oxide which is almost insoluble in water but soluble in alkaline. ZnO change color from white to yellow when heated and in the air turning whitish on cooling. This color change occurs because as the temperature difference is known as thermochromic properties. In materials science in particular, ZnO is one of the candidate materials that have attracted the attention of the material and an n-type semiconductor material group II-VI wide band gap energy of 3.3 eV and a binding energy of excitation of 60 mV at room temperature [1,2].

Thin film is a very thin layer of organic material, inorganic, metal, or metal-organic mixture that can have properties of conductors, semiconductors, and insulators. Thin film technology zinc oxide (ZnO) lately has been much studied and experienced growth, both in terms of how to manufacture, materials used and its application in public life such as light-emitting devices, solar cells, gas sensors, flat panel displays etc. This application is based as pulling characteristics of ZnO as wide band gap energy, direct optical transitions etc. ZnO thin films can be synthesized by various methods, such as molecular beam epitaxy [3], the RF magnetron sputtering [4], pulsed laser deposition [5], spray pyrolysis [6], chemical bath deposition [7], physical vapor deposition [8], sol-gel dip coating [2] and spin coating [9].

Method of sol-gel spin coating is a combination of physical and chemical methods which are used to make thin films of polymer materials deposited on the surface of the photoresist which flat-shaped silicon. After the solution (sol-gel) was dropped on the substrate, the rotational speed is set by the centrifugal force to produce a homogeneous thin film. Synthesis of thin films by sol-gel method of spin coating is very easy and effective simply by adjusting the parameters of time and rotational speed and viscosity of the solution and has several advantages such as low cost, do not use a vacuum chamber with a high, homogeneous composition, coating thickness can be controlled and The microstructure is quite good, so this method is widely used in the manufacture of thin film [10].

2. Experimental

Zinc acetate dehydrate Zn $(CH_3COO)_2$ $2H_2O$, isopropanol (C_3H_8O), and diethanolamine / DEA ($C_4H_{11}NO_2$) respectively as precursor materials, solvents and stabilizers. The molar ratio between the DEA and ZnAc is 1: 1. Zinc acetate dehydrate dissolved by the solvent isopropanol with various concentration (0.6 M, 0.7 and 0.8 M), then stabilized with diethanolamine (DEA). Mixing process using the technique of reflux, the solution was then stirred with a magnetic strierr the rotation speed of 60-70 rpm at a temperature range of 60-85^oC and about 60 minutes longer stirring up in get clear solution and transparent. The solution was then cooled to room temperature to form a gel which is rather thick. Subsequent gel gel dripped on glass slides were placed on a spin coater plate and rotated. The coating is performed for 30 seconds at a speed of 5000 rpm, after subtract surface evenly coated, then dried in a furnace at 100° C and held for 10 minutes. Furthermore, the coating until the fifth. Heating or pre-heating to a temperature of 300^oC for 5 hours, at let stand for 15 minutes and annealing or post-heating at a temperature of 500^oC for 5 hours, allowed to stand for 15 minutes.

3. Results and Discussion

3.1 The crystal structure of ZnO thin films

Characterization using XRD performed to identify the peaks are formed, lattice parameters and crystal grain size. XRD test results for all samples analyzed by search march showed the shape of the diffraction pattern of ZnO in Figure 1. The diffraction pattern of ZnO crystal fields have the same, namely the (100), (002) and (101) and all the peaks oriented growth in the field of (101) and hexagonal-shaped ZnO crystals. The intensity of the peak (chopped) to a concentration of 0.6 M; 0.7 M and 0.8 M is a count of 528, 1457 and 1296 chopped. Crystal lattice parameters for concentration of 0.6 M; 0.7 M and 0.8 M is a = 3.2494 Å, c = 5.2038 Å; a = 3.2490 Å, c = 5.2070 Å and 3.2553 Å a =, c = 5.2073 Å. The ratio c / a crystal films ZnO thin to a concentration of 0.6 M, 0.7 M and 0.8 M is 1.601; 1.603 and 1.601. The ratio c / a for all concentrations having a value equal to the ideal value for the hexagonal cell c/a = 1.602 [11]. These results indicate that all hexagonal wurtzite ZnO crystal form and in accordance with the standards of data ZnO JCPDS card 80-0075.

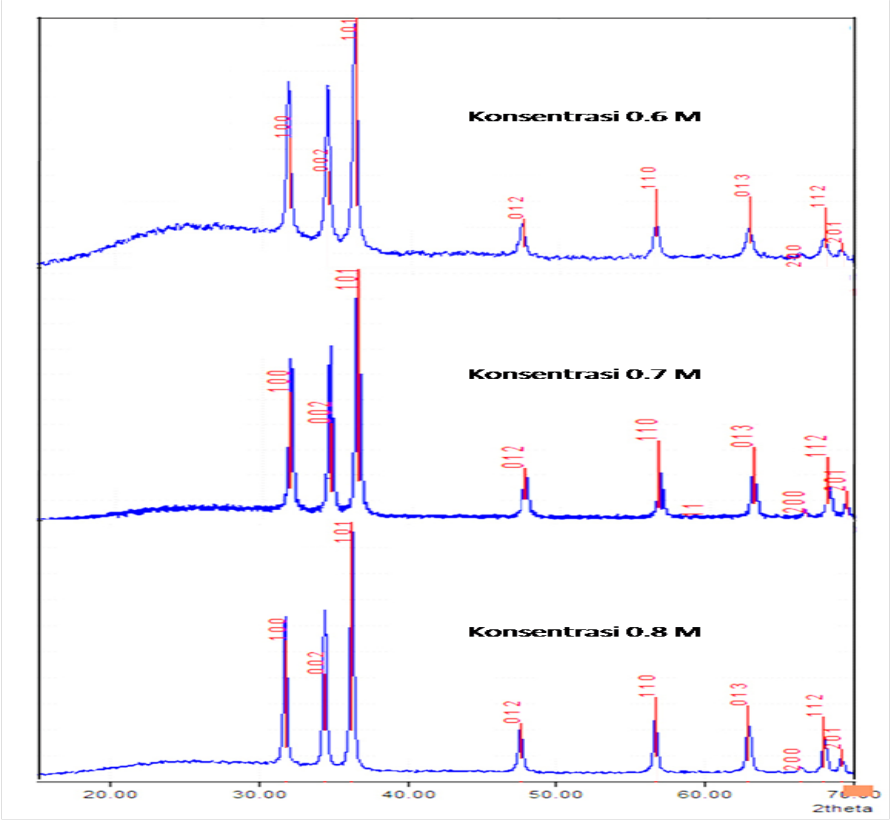

Figure 1: XRD spectra of ZnO thin films with various concentration

ZnO crystal size as shown in Table 1 was obtained by using the Scherrer equation [12], namely:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \qquad (1)$$

where D is the crystal size, λ is the wavelength, β is the FWHM (full width half maximum), θ is the angle of diffraction.

Table 1: The crystallite size of ZnO thin films with various concentration

Concentration	Phase	Peak		Cristal size (nm)
		2θ (degree)	FWHM(degree)	
0.6 M	ZnO	36.2663	0.32990	25.4
0.7 M	ZnO	36.2699	0.29130	30.3
0.8 M	ZnO	36.2377	0.24600	34.1

The crystallite size of ZnO thin film is formed, influenced by the concentration, the greater the concentration, the greater the size of the crystal ZnO thin films were formed and the smaller width of the XRD spectrum or FWHM value. [13, 14, 15].

3.2 Optical properties of ZnO Thin Films

Transmittance and absorbance spectrum of ZnO thin films with various concentration of UV-Vis test results are shown in Figures 2 and 3.

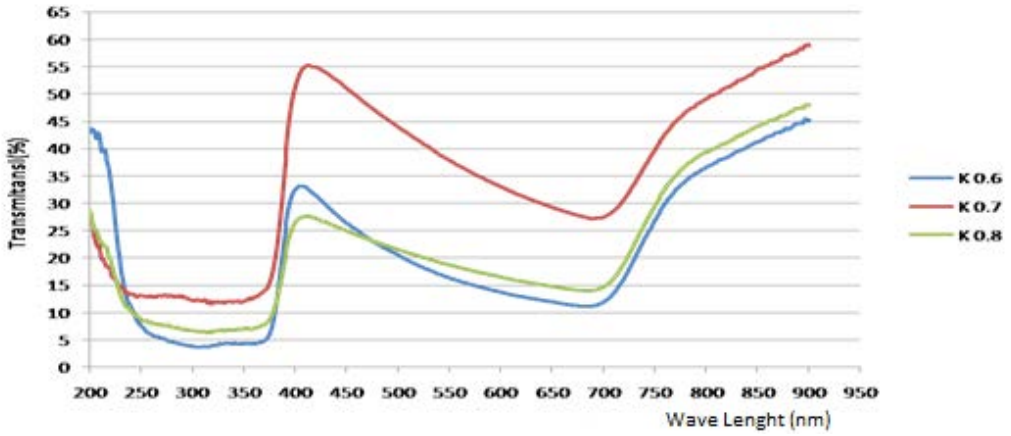

Figure 2: transmittance spectra for samples with various concentrations

Transmittance curve in Figure 2 shows the transmittance value of the lowest and highest for concentrations of 0.6 M; 0.7 M and 0.8 M is (3.6%; 45.2%), (11.8%; 59.0%) and (6.3%; 48.0%). The increase in the value of transmittance sharp enough for all samples ZnO thin films occurs in the wavelength range of 350 nm to 400 nm and stabilized at wavelengths <350 nm which is the ultraviolet wavelength region. The increase in the value of the transmittance of ZnO thin films showed a decrease in the value of a sharp absorbance in the wavelength

range of 350 nm to 400 nm which is the wavelength of the ultraviolet region, as seen in Figure 3. The lowest absorbance value and the highest for a concentration of 0.6 M, 0.7 M and 0.8 M is (0.324; 1.069), (0.217, 0.881) and (0.229; 1.119). The greater the concentration, the constituent atoms will be more and more, as well as the light particle collisions with atoms and the more difficult the more light can pass through it. [13, 14, 16].

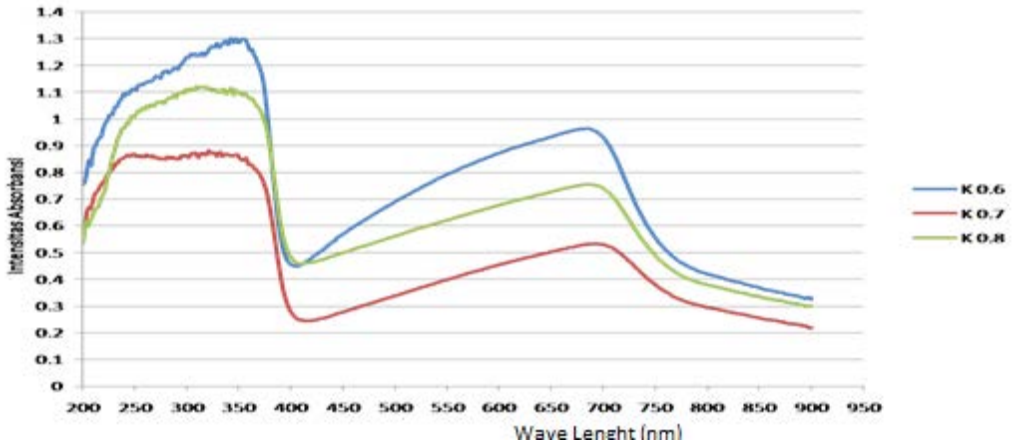

Figure 3: absorbance spectra for samples with various concentrations

For materials with a direct band gap of the optical properties of ZnO thin films studied by correlation coefficient of absorption of the photon frequency satisfies the equation [17]:

$$(\alpha h\nu)^2 = C_D(h\nu - E_{opt}) \qquad (2)$$

the optical absorption coefficient α is, *h* is Planck's constant, ν is the frequency of the incident photon, the C_D is a constant, and E_{opt} is the energy gap of the sample. The energy band gap of ZnO thin films with various concentration obtained from the slope of the straight line fitting the results shown in Figure 4.

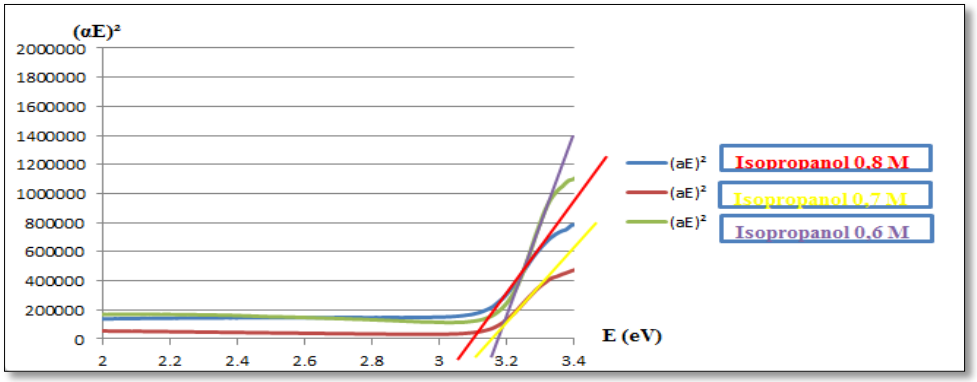

Figure 4: Curve $(\alpha h\upsilon)^2$ as a function of energy with various concentration

Based on Figure 4. gained wide energy band gap of ZnO thin films with various concentration which results are shown in Table 2.

Table 2: The energy band gap of ZnO thin films for various concentration

Concentration	Energy band gap (eV)
0.6 M	3.18
0.7 M	3.16
0.8 M	3.13

In Table 2 shows that the width of the energy band gap decreases with increasing concentration of the solution, it is because the energy absorbed more and more material and cause wide band gap energy decreases.

4. Conclusion

1) Shapped crystal structure of hexagonal wurtzite

2) Crystal size ranges 25.4 s/d 34.1 nm

3) Transmitansi value ranges 45.2% s/d 59.0%

4) The value of the band gap energy ranges 3.13 s/d 3.18 eV.

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