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Crystal Structure Characterization of Thin Layer Zinc Oxide

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Abstract. In this research the characterization of the crystal structure of a thin layer of ZnO (zinc oxide) were synthesized by sol - gel method and spin coating deposited on a glass substrate. The samples were divided into three sol concentrations of 0.1, 0.3, 0.5 Molar and two deposition temperature is 350 °C, and 550 °C. UV-Vis. spectrophotometer results showed that in the spectrum of visible light (wavelength range 300-800 nm) has a transmittance value of which increases with increasing concentration and temperature deposition of zinc oxide, otherwise the value of the absorption and the band gap energy decreases with the addition of concentration and deposition temperature. The transmittances value of the highest and lowest absorption was 93.5% and 0.03 is at a concentration of 0.1 M and zinc oxide deposition temperature of 550 °C, with a value of band gap energy of 2.98 eV. The XRD results showed that the zinc oxide crystal orientation in the field of 013 with a crystal grain size 14.4472 nm. SEM results showed the surface morphology of zinc oxide such as rod-like.

1. Introduction

Nano-meter sized material has now attracted the attention of researchers science because electrical, physical, chemical, and magnetic properties of potential in the development of technology [1]. One of the material in question is a metallic material. Metal material which widely used in application today is in the form of a thin layer, for example, tungsten oxide (WO₃), indium (In₂O₃), tin (SnO₂), titania (TiO₂), zinc oxide (ZnO) and many other materials. Among the metal materials, metal ZnO has the greatest abundance in nature [2]. Moreover, ZnO metal inexpensive and versatile [3]. A thin layer of zinc oxide (ZnO) pure and doping has been regarded as an excellent semiconductor material for transparent conductive oxide (TCO), because it has a high conductivity, good optical transmittance (> 90%), and low cost of fabrication. A thin layer of ZnO shows the thermal stability, and are not easily change shape if the process of dislocation of material structure [4]. Some of the parameters that can be observed in the manufacture of thin layers by the method of sol-gel spin coating, among other sol solution concentration, the concentration of doping, deposition temperature, the speed of the spin coating, and aging time. Ongoing research to produce the layer characteristics as expected. The characteristics of the optical properties of ZnO thin layers in the range of the visible spectrum (370-800 nm). ZnO thin layer has an optical transmittance values decreasing from 90 to 25 % when the concentration of sol added 0.25 M to 1 M, and a smaller band gap energy values of 3.30 eV to 3.22 eV at a concentration increased from 0.25 M to 0.75



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M. However the band gap energy increased from 0.22 eV to 3.27 eV at a concentration of 0.75 M to 1 M [3]. The optical properties of ZnO seen transmittance 91% to 95% in the range 400 to 800 nm. Crystal size in the range of 18 to 24 nm [5]. Furthermore in this research conducted characterization of thin layer of zinc oxide to variations in the concentration of the sol and the temperature of deposition. The characterizations are the optical properties (transmittance, absorption, energy band gap) of thin layer. Then, determine the crystal structure and surface morphology of the sample of the best of a layer of zinc oxide.

2. Materials and Methods

Substrate materials that used in this research is a glass slide that cut to a size of (10 x 5) mm², and a thickness of 3 mm. Before use of the glass slide is washed in stages. Laundering gradually aims to remove oil and debris on the glass. Leaching the first stage, the glass substrate is inserted into a measuring cup containing a mixture of water and detergent. Furthermore the glass substrate is rinsed with water until clean. Washing at the second stage, the substrate is inserted into a glass beaker containing alcohol. The glass substrate is dried in a furnace at 100 °C for one hour. The glass substrate that is clean, then stored in plastic clip. The process of making sol-gel using basic materials zinc acetate dehydrate (ZnAc), ethanol and MEA (Mono Ethanolamine), each of which serves as the solute, solvent and stabilizer. The sol-gel materials that synthesized divided into three concentrations, namely 0.1 M, 0.3 M, and 0.5 M. The solution ZnAc and ethanol stirrer on a hot plate at a temperature of 70 °C for 10 minutes, or until the solution homogeneously mixed. Layer growth process begins with a drop of sol-gel materials on a glass substrate is approximately 3 drops. Then spin for two minutes with a rotation speed of 2000 rpm. Once the surface evenly coated substrate, then the substrate is dried in a furnace at 100 °C for 10 minutes. The heating process is done in three stages. First heating at a temperature of 150 °C (calcinations) for one hour, aiming to eliminate water and residual solvent content in the coating gradually. A second heating at a temperature of 350 °C for one hour. The increase in the regulated temperature slowly from room temperature to 350 °C. This stage is regarded as pre-heating phase that serves to remove ethanol, water, and MEA, and facilitating change ZnOH into ZnO. The next stage is the post-heating or heating end at 550 °C, for one hour. The same with the process of pre-heating, regulated temperature rise slowly from room temperature to 550 °C. Post-heating function to form ZnO particles with a uniform crystal orientation, and eliminating pores.

The characterization using UV-Vis spectrophotometer is intended to identify how much the value of transmittance and absorption of ZnO thin film for some variations in the concentration (molar sol gel solution). Characterization using X-ray diffraction (XRD) is intended to identify the different forms of the crystal structure in a thin layer of ZnO quantitatively. The identification results obtained by comparing the diffractogram of the ZnO thin layer of the unknown with standard data that contains a reference pattern. The characterization using Scanning Electron Microscopy (SEM) is intended to identify the micro-structure surface of a thin layer of ZnO crystals. The identification results obtained in the form of a thin layer of ZnO surface profiles such as how and where the direction of the slope ramps that can be built using an image processing program in the computer. Determining the value of the energy gap of a semiconductor material needed to know to provide an analysis of the optical absorption coefficient associated with the frequency of photons. Energy band gap of ZnO thin layer (zinc oxide) is calculated by following equation [6].

$$(\alpha h\nu)^{\frac{1}{n}} = A(E - E_g) \quad (1)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant, and E_g is the band gap energy. Exponent n depends on the type of transition, where the transition to direct, indirect, prohibited direct and indirect forbidden row has a value of 1/2, 2, 3/2, and 3.

Results and Discussion

The measurement using equipment UV-Visible, a thin ⁵er of ZnO can transmit visible light at wave number range of 300 nm-800 nm. For a concentration of 0.1 M, 0.3 M, 0.5 M at temperature deposition at 350 °C with the highest and lowest transmittance values are (83.1 % and 21.7 %), (80.4 % and 10.6 %), (78.7 % and 2.7 %), respectively. For temperature deposition 550°C with the highest and lowest transmittance values are (93.5 % and 9.6 %), (87.6 % and 8.4 %), (84.9 % and 5.9 %) respectively as shown in Figure 1 (a) and (b).

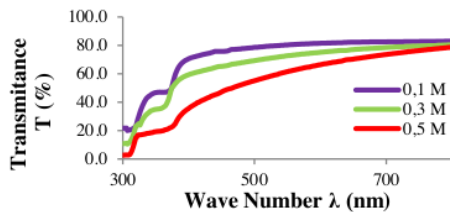


Figure 1(a). Graph the value of optical transmittance T (%) as a function of wavelength λ (nm) thin layer of ZnO temperature deposition at 350 °C

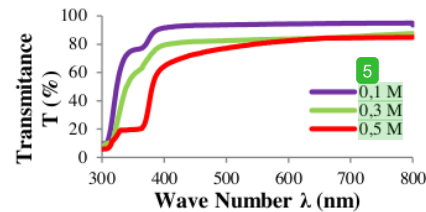


Figure 1(b). Graph the value of optical transmittance T (%) as a function of wavelength λ (nm) thin layer of ZnO temperature deposition at 550 °C

Furthermore, the value of the highest optical transmittance of ZnO thin ²ilm for the wavelength range of 300 nm-800 nm was 93.5%. That value shows the condition of ZnO concentration of 0.1 M and for a deposition temperature of 550 °C. While the lowest value of the optical transmittance of ZnO ²thin layer to the wavelength range of 300 nm-800 nm was 2.7%. That value shows the condition of ZnO concentration of 0.5 M and a deposition temperature of 350 °C. Thus the optical ⁵sorption values (A%) as a function of wavelength (λ) for the variation of the concentration of ZnO is 0.1 M, 0.3 M, 0.5 M respectively and temperature deposition variations was 350 °C and 550 °C as shown in Figure 2 (a) and (b).

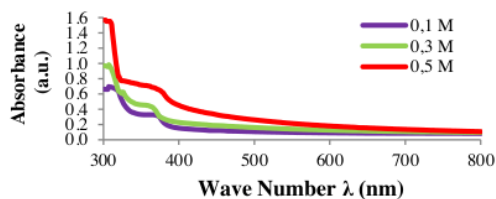


Figure 2(a). Graph the value of a optical absorption (a.u.) as a function of wavelength λ (nm) thin layer of ZnO temperature deposition at 350°C

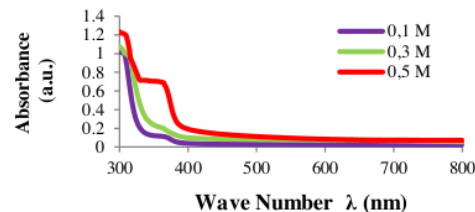


Figure 2(b). Graph the value of a optical absorption (a.u.) as a function of wavelength λ (nm) thin layer of ZnO temperature deposition at 550 °C

Figure 2. shows that for deposition temperature 350 °C and a concentration of 0.1M, 0.3 M, 0.5 M respectively, a thin layer of ZnO can absorption of visible light in range 300nm -800 nm with the lowest and highest values are (8 % and 66 %), (9 % and 97 %), (10 % and 157 %) respectively. Furthermore for

9
deposition temperature 550 °C and a concentration of 0.1M, 0.3 M, 0.5 M respectively, a thin layer of ZnO can absorption of visible light in range 300nm -800 nm with the lowest and highest values are (3 % and 102 %), (6 % and 108 %) (7 % and 124 %) respectively.

Thus, the value of the highest optical absorption of ZnO thin film for the wavelength range of 300 nm to 800 nm is 157 %, i.e on condition ZnO concentration of 0.5 M and temperature deposition at 350 °C. While the lowest value of the optical transmittance of a thin layer of ZnO for the wavelength range of 300 to 800 nm is 3%, that on condition of ZnO concentration of 0.1 M and temperature deposition at 550 °C. The values of gap energy of ZnO thin film obtained for various concentrations (0.1, 0.3, 0.5 M) and the variation of the deposition temperature (350 and 550°C) can be seen in Figure 3 (a) and (b).

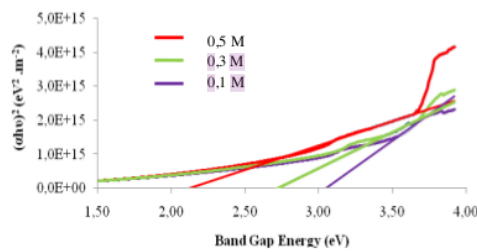


Figure 3(a). Graph The values of gap energy of ZnO thin film obtained for various concentrations (0.1 M, 0.3 M, 0.5 M) and the variation of the temperature deposition at 350°C

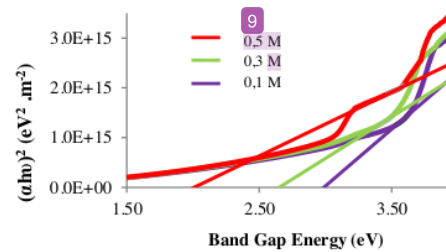


Figure 3(b). Graph The values of gap energy of ZnO thin film obtained for various concentrations (0.1 M, 0.3 M, 0.5 M) and the variation of the temperature deposition at 550°C

Energy gap of thin layer ZnO at different concentration of precursor and temperature deposition was calculated using Equation 1 and shown in Table 1.

Table 1. The value of gap energy for variations in concentration and temperature deposition of thin layer ZnO

The concentration of ZnO (M)	The Value of Gap Energy (eV)	
	Temperature (°C)	
	350°C	550°C
0,1	3,06	2,98
0,3	2,75	2,67
0,5	2,18	2,03

Figure 4 shows the X-Ray Diffraction (XRD) measurement of thin layer ZnO obtained from concentration of precursor 0.1 M and temperature deposition at 550°C. The diffraction pattern of ZnO crystal planes is (013) and hexagonal ZnO crystal, formed at positions 2 theta 64.4415 and the grain size 14.4472 nm. Based on XRD measurement, the grain size and Miller indices could be seen on Table 2

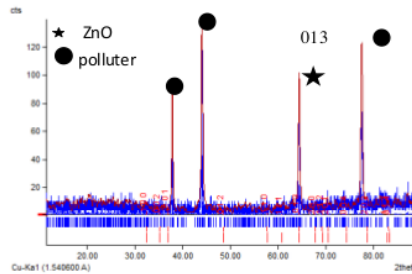


Figure 4. Thin layer ZnO, 0.1 M and temperature deposition at 550 °C of crystal diffraction intensity as a function of 2θ

Table 2. Parameters Crystal Structure of thin layer ZnO, 0.1 M at temperature deposition at 550 °C

No.	2Theta (deg)	The grain size (nm)	FWHM (deg)	Intensity (counts)	Miller indices
1	64,4415	14,4472	0,20860	57	0 1 3

The crystallites sizes (D) of thin layer ZnO is estimated using the Scherer formula [6].

$$D = \frac{k\lambda}{\beta_{2\theta} \cos \theta} \quad (2)$$

3 where k is a constant taken to be 0.94, λ is the wavelength of X-Ray used ($\lambda = 1.54 \text{ \AA}$) and $\beta_{2\theta}$ is the full width at half maximum of (013) peak of XRD pattern, Bragg angle 2θ is around 64.44° . The average value of grain size is found to be 14.44 nm.

Based on Scanning Electron Microscope (SEM) image, surface morphology of thin layer ZnO synthesized with concentration of precursor 0.1 M and temperature deposition at 550°C show in Figure 5.

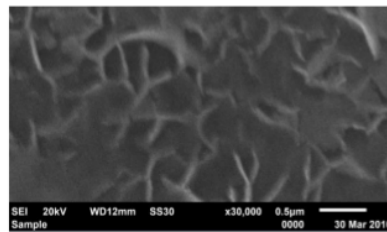


Figure 5. The surface of thin layer of ZnO 0.1 M and temperature deposition at 550 °C

8 The photos of thin layer ZnO crystal surface morphology seen that the growth of ZnO is not symmetrical. The surface morphology obtained in this research when compared to another result [6] are quite similar, meaning that the results of SEM in this study were nearing the properties of ZnO crystals morphology. The growth of ZnO is not symmetrical look like a stalk or stem is commonly known as nano rods. In a

review of the others can be seen as the root of the tree (root like). The results of SEM testing has been done before [7] can be seen in Figure 6.

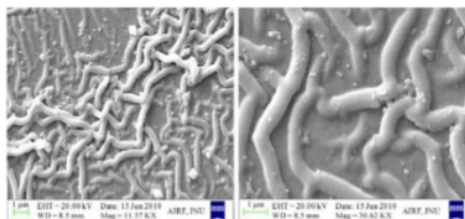


Figure 6. Surface Morphology of thin layer ZnO at concentration of 0.5 M [7]

Conclusion

The characterization of optical structure of thin layer ZnO, obtained that the increasing of concentration thin layer ZnO show that the decreasing the transmittance value, increasing the absorption, and the smaller the value of the gap energy. Furthermore the increasing temperature deposition of thin layer ZnO, show that the increasing of the transmittance value, decreasing the value of absorption, and the smaller the value of the gap energy. Based on field diffraction pattern has a diffraction peak intensity at 14.4472 nm grain size of thin layer ZnO of 0.1 M and temperature deposition at 550 °C. The surface morphology of thin layer ZnO such as rod-like.

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