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Research Article

Thermal effect on TiO_2-doped ZnO thin film and its evaluation as a transparent electrode material

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Abstract: There is a search for cleaner and sustainable lower cost transparent conductive oxide as an alternative to high cost and toxic indium tin oxide. This work examines post- deposition heat treatment properties of Titanium dioxide doped Zinc oxide as transparent conducting oxide. Titanium dioxide doped Zinc oxide solution was deposited via spin -coating deposition technique and annealed to investigate annealing temperature effect on the properties. Characterization was done with UV -Vis spectroscopy, Scanning Electron Microscopy, and X-ray diffraction. X-ray diffraction revealed that the films grow preferentially oriented in the (014) crystallographic direction of the Zinc Oxide grains. Optical transmittance of thin film annealed at 400 ºC was approximately 80% in the visible solar spectrum which suggests a highly suitable material for transparent conducting oxide applications. Optical band gap energies varies between 3.74 and 3.78 eV and a low electrical resistivity values 2.04 x 10 - 3 Ω-cm to 3.12 x 10 - 3 Ω-cm. Scanning Electron Microscopy images show that the roughness and grain size of thin film increases with increase in temperature.

Keywords: Optical Properties; ZnO:TiO2 Composite; Transparent Electrode; Spin-coating; Electrical Properties

1. INTRODUCTION

This research examines the properties of Titanium dioxide (TiO2) doped zinc oxide (ZnO) after heat treatment as transparent electrode material. Optical, morphology, structural, and electrical properties are analyzed for possible application as transparent conductive oxide (TCO). Transparent conductive oxides (TCOs) are conducting electrode on which active components materials are deposited. According to literature, a good transparent conductive oxide for electrode and photovoltaic applications must have the quality of low electrical resistivity (10-3 to 10-4 Ω-cm), high visible light transparency between 80 and 91%, and band –gap energy < 3.35 eV in the solar spectrum as reported (Hirata *et al.,* 1996; Qin et al., 2021; Sato *et al.,*1994; Seki *et al.,* 2001; Wang *et al.*, 2021). The commercial transparent conductive oxides in the market are fluorine doped tin oxide (FTO) and indium-doped Oxide (ITO) thin films. There are flaws and shortcomings associated with the current TCOs namely, high cost of production, deposition at very high temperature, toxicity, and indium scarcity. Research on low cost TCO is justifiable due to ever increasing production cost of electronic devices based on current commercial ITOs.

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These flaws and shortcomings call for more research for alternatives and substitutes materials to these commercially based TCOs. Reducing the cost of production of TCO commercially without compromise of standard solar parameters needed remain a challenge and a topic for research. Zinc oxide is being considered as a possible alternative because of its properties such as band-gap energy of 3.37eV, higher breakdown voltage to withstand high electric field, high power and high temperature operations but there some flaws associated with the use of ZnO, such as intrinsic defects, oxygen vacancies (VO), and zinc interstitials (Zni) as reported in literature by (Afrina *et al.,* 2019; Rwenyagila *et al.,* 2014; Schmidt-Mende et al., 2007; Sumit, 2020). Doping ZnO with Titanium dioxide (TiO2) a group B element may modify the properties of ZnO and reduce or eliminate flaws associated with ZnO because TiO2 have high conductivity, high optically transparency, large energy gap, good mechanical, electrical, optical and thermal properties. Moreover, band gap of zinc oxide (ZnO) and titanium dioxide (TiO2) are very close to each other. Doping of a material with another material can modify properties of a material. Zinc oxide and titanium dioxide applications are found in the field of engineering and technology. The drawbacks associated with ZnO may be possibly overcome by doping ZnO with TiO2 (Tian *et al.*, 2009).

There are several techniques that can be used to deposit, dope and synthesis thin films such as, chemical vapor deposition (CVD) (Mohammed *et al.*, 2015), sol-gel deposition (Ahmad *et al.*,2010; Bakri *et al.,* 2017 Bessekhouad et al., 2003; Cenovar, et al., 2012; Haripriya *et al.,* 2009), spin coating (Balogun *et al.,* 2017, 2018; Vasuki et al., 2014; Sandeep & Dhananjaya, 2015; Vasuki *et al.,* 2014), spray pyrolysis, simple chemical method (Ali *et al,* 2018), radio frequency magnetron sputtering and DC magnetron sputtering and magnetron deposition (Su-shia *et al,* 2005), pulse laser deposition (Kadhim et al,2015), hydrothermal process, thermal decomposition, combustion method, laser ablation technique, electrochemical process, etc. Among the different techniques of thin film deposition, spin-coating technique was chosen in comparison to the other methods. Spin-coating route is considered as very promising one for the synthesis because of its relative advantage of uniform deposition unto flat substrate, is cheaper and easier to use. Some studies reported that electrical properties of the TiO2 thin film increase with increase in the annealing temperature and resistivity decrease with increase in the annealing temperature. Also the grain size of the TiO2 increases with increase in the annealing temperatures by different method of synthesis.

Literature indicates that annealing temperature influences the surface porosity, surface morphology, structural, and electrical properties thin film. Hassan et al., (2008) reported that TiO2 films produced at room temperatures exhibited high visible transmittance. Balogun *et al.,* (2021) investigated the influence of annealing temperature on the optical and structural proper-ties of zinc oxide (ZnO) thin film as a transparent conductive oxide electrode prepared by spin- coating method. They showed that ZnO annealed at different temperature had minimum sheet resistance of 2.1 X 105 (Ω / sq), resistivity of 4.20 (Ω-cm), highest optical transmittance value of 95% in the visible region and optical band gap energy that lies between 3.25 and 3.6 eV. Rwenyagila et al (2015) investigated structural and optical properties of ZnO film precursors for multilayered transparent solar cell electrodes and reported very high resistivity values for a single layered ZnO films for transparent electrode and recorded maximum transmittance value of 90% in the visible region of wavelength. Muslih and Kim (2017) reported zinc oxide thin film deposited by spin- coater and annealed at 500 OC having resistivity of 2.35 x 10-3 Ω.cm.

This present research focus on effect of post-deposition heat treatment on the TiO2-doped ZnO thin film and evaluation of the properties as a transparent conductive oxide as electrode material. The thin films were subjected to temperature annealing from 400 ^{0}C to 1000 ^{0}C to evaluate the optical, morphological, structural, electrical properties and to ascertain the suitability as a transparent conducting oxide. The reported results shows that TiO2-doped ZnO thin film have highest transparency in the visible region which occurred at annealing temperature of 400 ^{0}C. The resistivity of ZnO reduced as a result of TiO2 doping making TiO2- doped ZnO thin film as an alternative to expensive commercial ITO and FTO.

2. MATERIALS AND METHODS

2.1. Materials

This research was carried out with the following materials, Zinc Oxide purchased from Sigma-Aldrich with a molecular weight of 81.39gm Mol. Nano powder < 50nm particle (>97%) and Titanium dioxide, Microscope glass slide, Ethanol (99.8%), Isopropanol alcohol (IPA), and deionized water. All reagent were used as received.

2.2 Method

The experiment was done under ambient temperature.

2.2.1 Preparation of glass slide substrate

The microscope glass slides to be used were washed with detergent solution in a beaker placed inside ultrasonic bath and then rinsed with deionized water. Further cleaning was done with ethanol and IPA. Thorough washing of glass slides was done to remove organic and inorganic impurities embedded on the surface of the microscope glass slides. The glass slides dried with nitrogen gas and used.

2.2.2 TiO2- doped ZnO solution preparation

250 mg of 99.99% pure ZnO powder and 250 mg of 99.8% TiO2 powder was dissolved into 10 mL ethanol of 99.9% purity separately. The solutions were stirred separately on magnetic stirrer for 2hr for homogenous mixture. These two solutions were then mixed in the volume ratio (1:1; 1:09; 1:08; 1:07; 1:06; 1:05; 1:04; 1:03; 1:02; 1:01) of ZnO to TiO2 and stirred for another 2 hours more in a beaker on magnetic stirrer. Then the solution was deposited on glass slides and without heat treatment optimized for the highest transmittance value. The sample with the highest value of % transmittance (1: 07) was chosen for further work of deposition

2.2.3 Deposition of TiO2- doped ZnO Solution

Deposition of the solution on glass slide substrate was done with a spin –coater (model WS-650MZ-23NPP). Spin-coating machine is used to deposit solution processed materials onto flat glass substrates. Spin- coating is a procedure used to deposit uniform thin film to flat substrates. Usually, a small amount of coating material is applied on the center of the substrate placed on the stub of the spin –coater, which is either spinning at low speed or not spinning at all. The substrate is then rotated at desired set speed (rpm) and at set time (seconds) in order to spread the coating material by centrifugal force. Rotation is continued while the fluid spins off the edges of the substrate, until the desired thickness of the film is achieved. Spin coating involves acceleration of drops of liquid on a rotating substrate. Spin –coater was set at 2000 rpm for 30 sec. with vacuum created by vacuum pump. Surface profiler was used to measure thin film thickness. Surface profiler measurement of thin film indicated a formation of 100 nm thickness on glass substrate under ambient condition. Post- deposition heat treatment of fabricated thin film was carried out in temperature range of 400 ºC to 1000 ºC in a step size of 100 ºC. Carbolite tubular furnace (model Srw21-501042 Type-CT17) was used for heat treatment under Argon gas. All samples were quenched at room temperature

2.3 Characterization of TiO2- doped ZnO thin film Samples

The fabricated thin films were characterized with Ultraviolet-Visible Spectrophotometer (UV-Vis), X-ray Diffractometer (XRD), Scanning Electron Microscope (SEM), and I-V measurement taken with Keithley source-measure unit (SMU). UV-Vis measurement was recorded with UV-Vis Spectrophotometer (model Avalight-DH-5-BAL) which gives the value of transmittance against wavelength to enables us calculate the absorbance and band gap energy (Eg) of the thin film. XRD gives the knowledge about the crystal structure of the material and the crystallinity. Scanning Electron Microscopy (model ASPEX 3020) reveals the surface morphology of thin film and the electrical resistivity of the thin film was calculated using the recorded values obtained from Four- Point Probe (FFP) system.

2.3.1 Optical Properties Calculations

The recorded value obtained from UV-Vis Spectroscopy was used to calculate the Absorbance using Eq. (1)

$$A = 2 - \log(\%T)$$

(1)

Where A is the optical absorbance in arbitrary unit (a u) and % T is the optical transmittance.

Energy band gap calculation was obtained with Eq (2)

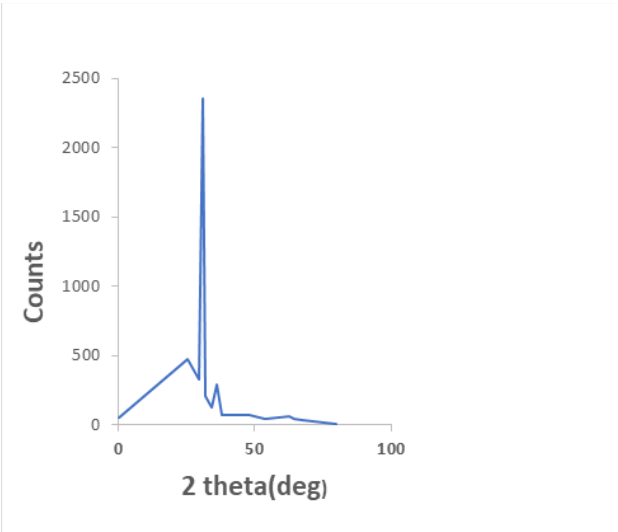
$$(\alpha h\nu)^{1/n} = A(h\nu - Eg) \text{ or } (\alpha h\nu)^2 = h\nu - Eg$$

Where (hv) is the photon energy in electron volts (eV), Eg is optical band gap, A is a constant and n is 1 or 4.

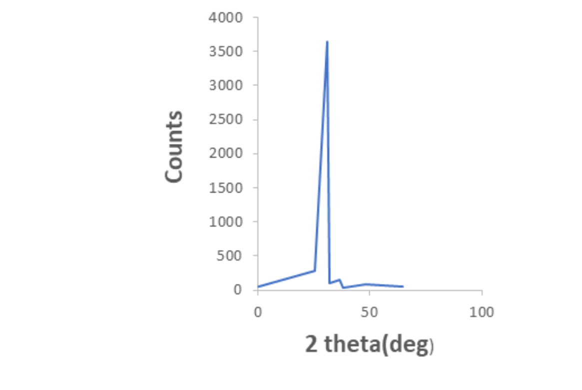
3. RESULTS

3.1 Structural Properties

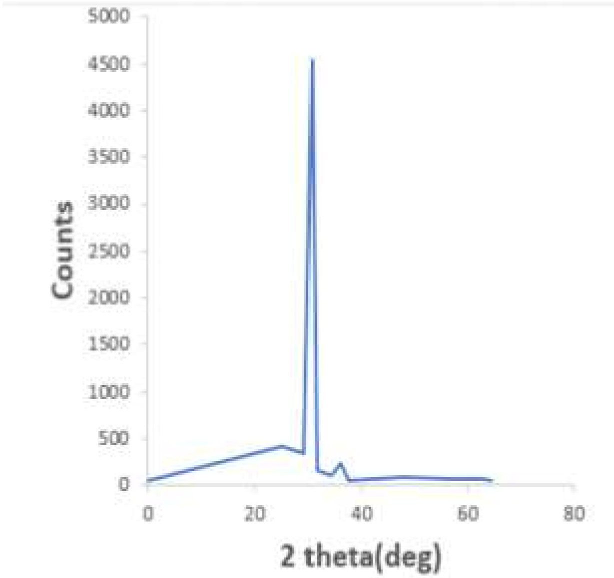
XRD patterns analysis was obtained from diffactometer system EMPYREAN, Cu Kα radiation (λ = 1.54060 A°) at 45 kV/40 mA. The XRD diffraction patterns of the TiO2- doped ZnO thin films obtained at annealed temperature of 400º C, 500º C, and 600º C are shown in figure 1(a-c). All annealed samples have a dominant peak at 2θ = 30.880º with sharpness which suggests high crystallinity. The crystallographic parameters and the structural properties shows a hexagonal wurzite structure for ZnO and Tetragonal structure for TiO2. The X-ray composition analysis confirmed presence of ZnO phase based on the reflection from the plane at 400° C, (101), (104), (014), (100), (002), (101), (004), (200), (105), (103), (203), (112), and TiO2. At annealed temperature of 500° C composition analysis confirmed presence of ZnO phase based on the reflection from the plane (101), (014), (100), (101), (004), (200), (028) and TiO2. Sample annealed at 600° C confirmed the presence of ZnO based on the reflection from the plane (101), (104), 014), (020), (110), (004), (200), (222), and TiO2. Exhibition of strong peak occurred at plane (014) which indicates preferred growth orientation in the crystallographic direction of the ZnO.



(a)



(b)



(C)

Fig. 1(a-c): X-ray diffraction patterns of ZnO: TiO_2 annealed at (a) 400° C, (b) 500° C, and (c) 600° C

3.2 Optical Properties

Optical evaluation or measurement was carried out under ambient conditions. Absorption peak occurred in the visible region. Optical measurement helps to evaluate the optical transmittance, the reflectance, calculate absorbance, the refractive index, the extinction coefficient, the absorption coefficient (α), and band gap energy. Optical properties optimization of the ZnO: TiO_2 composite without temperature annealing shows molar ratio 1:07 is stable throughout the visible range of wavelength spectral (400 to 900 nm) as shown in figure 2. Optical transmittance was above 85%. The composite molar ratio 1: 07 was then subjected to thermal annealing at different temperature as shown in figure 3.

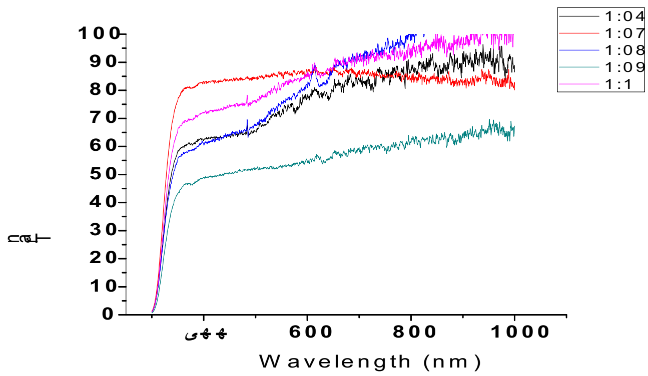


Fig. 2: Transmittance spectra of ZnO: TiO_2 at different volume ratio

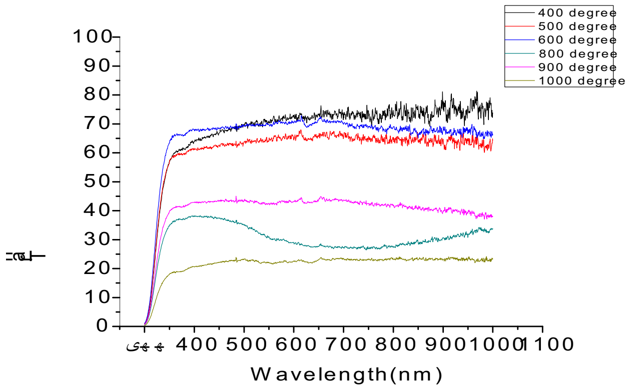


Fig. 3: Composite solution (ZnO: TiO_2) deposition at 1: 07 and thermally annealed

Optical transparency in the visible wavelengths in figure 3 was approximately 80% for thin film annealed at 400 ºC, the sample annealed at 600 ºC, fall within 70% and the rest annealed samples fall below 70%, therefore the thin film annealed at 400 ºC is highly suitable for transparent conducting oxide applications.

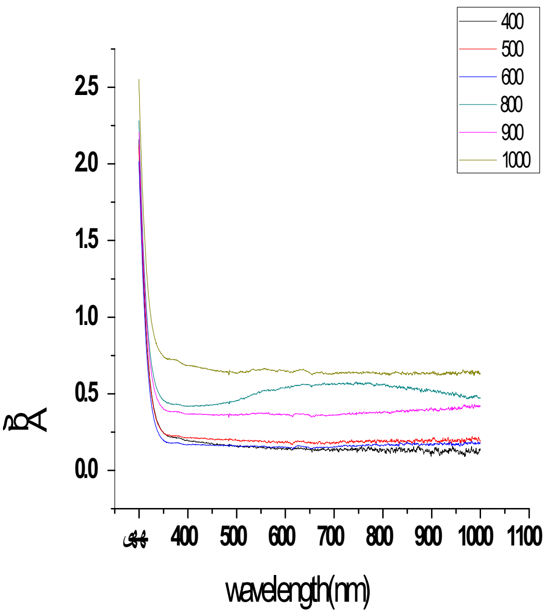


Fig. 4: Plot of UV-Vis absorbance spectra of composite

The plot of absorbance as a function of wavelength of the thin films is shown in figure 4. Thin film composite annealed at 400 ºC have the least absorbance among the remaining annealed samples. For application as TCO electrode the absorption must be low for high transmission of photon energy.

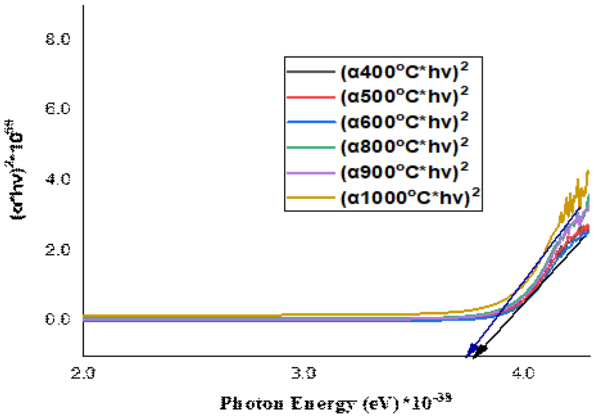
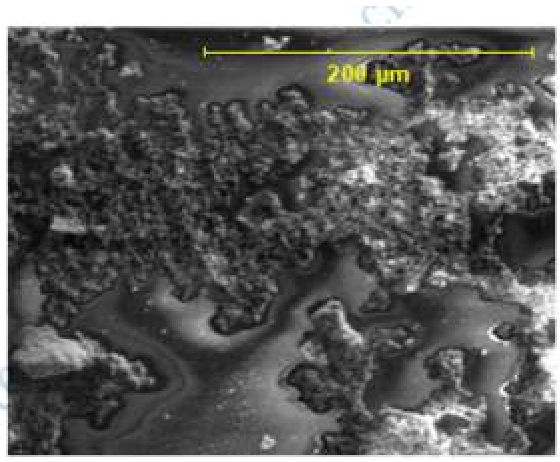


Fig. 5: Tauc Plot of $(\alpha hv)^2$ as a function of photon energy (hv) at different Annealing Temperature.

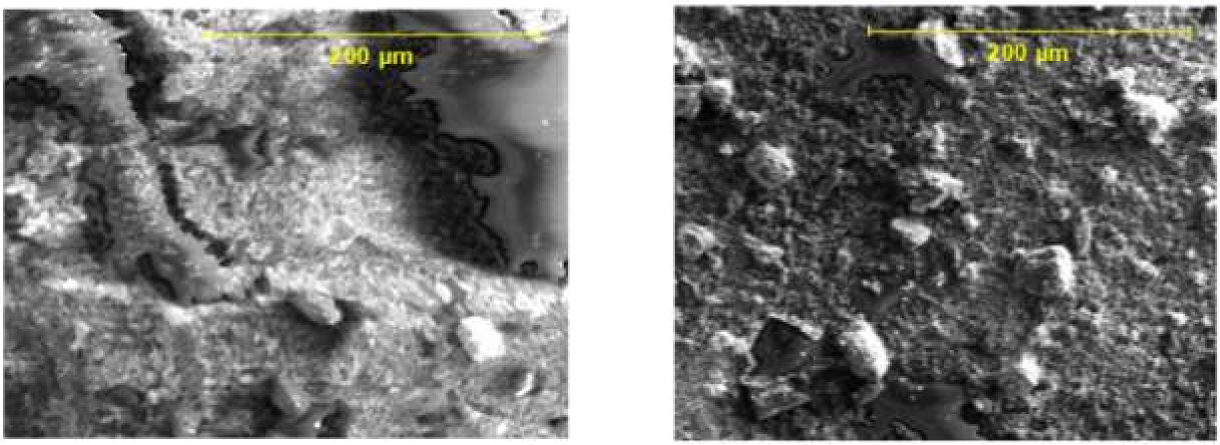
TiO_2 doped ZnO had optical band gap energies between 3.74 and 3.78 eV which concur with the result reported by (Rwenyagila et al, 2015) shown in figure 5.

3.3 Morphological Properties

Morphology study of TiO_2 doped ZnO thin film was done using Scanning Electron Microscope (SEM) at 200 µm, the surface morphology and roughness of sample obtained as shown in Figure 6 (a-c). The changes on surface morphology clearly can be observed. Upon thin film subjected to high temperature, the roughness and grain size increases.



(a)



(b) (c)

Fig. 6 (a-c). SEM images of TiO_2 -doped ZnO thin film at (a) 400 °C (b) 500 °C (c) 600 °C

3.4 Electrical Properties

Four- point probe (4PP) system was used to evaluate resistance of thin film. Current –Voltage (I-V) characteristic measurement obtained was used to calculate the resistance. The corresponding values of resistivity was calculated at 400 °C, 500 °C, and 600 °C annealing temperature using Eq. (3).

Where ρ is the electrical resistivity in Ω-cm, R is the electrical resistance measured in Ω, a = area (sq cm), and l is the length measured in cm.

Table 1. Electrical properties of thin film at 400 °C, 500 °C, and 6/00 °C annealing temperatu

Annealed Temperature (°C)	Average electrical resistivity ρ measured in Ω- cm
400 °C	2.04×10^{-3}
500 °C	2.38×10^{-3}
600 °C	3.12×10^{-3}

4. DISCUSSION

4.1. X-Ray Diffraction Analysis

Samples annealed at temperature 400 °C, 500 °C, and 600 °C were subjected to crystal structure analyses via XRD and the results are as shown in figure 1(a-c). Structural properties showed a hexagonal wurzite structure for ZnO and Tetragonal structure for TiO_2. The X-ray composition analysis confirmed presence of ZnO and TiO_2 phase based on the reflection from the plane at 400° C. At annealed temperature of 500° C composition analysis confirmed presence of ZnO and TiO_2. Sample annealed at 600° C confirmed the presence of ZnO and TiO_2 based on the reflection from the plane. Exhibition of strong peak occurred at plane (014) which indicates preferred growth orientation in the crystallographic direction of the ZnO: TiO_2 grains. X-ray diffraction measurements revealed that the films grow preferentially oriented in the (014) crystallographic direction of the ZnO grains

4.2. Optical Properties Analysis

Figure 2 shows optical properties of TiO_2 doped ZnO thin film optimization without heat or annealing. The sample of molar ratio (1:07) was chosen because it has stable transmittance values in visible wavelength spectrum. Sample annealed at temperature 400 °C had transmittance value of 80% in Figure 3 among samples annealed. Sample annealed at 400 °C will compete favorably with commercial transparent electrode material in terms of optical properties required of a good TCO. Thin film annealed at 400 °C have the least absorbance in figure 4, the absorbance of transparent electrode material must be low for high efficiency in application in electronic devices. Figure 5 shows the Tauc Plot of $(\alpha h\nu)^2$ as a function of photon energy (hv) of annealed samples. TiO_2 doped ZnOhad optical band gap energies between 3.74 and 3.78 eV. Band gap decreases with increase in thermal temperature. This shows that annealing temperature modified the optical properties of the fabricated thin film.

4.3. Morphology of Synthesized TiO_2 doped ZnO thin film analysis

With respect to figures 6 (a-c), the roughness of the thin film increases with increases in annealing temperature. The changes on surface morphology of thin film can be observed clearly in figures 6 (a-c). Upon thin film subjected to high temperature, the roughness and grain size increases. Probably increase in grain size could be attributed to the roughness of the thin film. It can be deduced that decrease in band gap is as a result of increase in annealing temperature.

4.4. Electrical Properties analysis

Electrical properties of a transparent electrode material (TEM) is function of sheet resistance, resistivity, Figure of Merit for transparent (FOM) and efficiency. It is expected that a good TCO should have high transmittance and a low resistivity for good conductivity. Current –Voltage (I-V) characteristics measurement was obtained with Four- point probe (4PP) system this values were used to calculate the value of the resistivity in table 1. From the calculation at temperature 400 ºC, 500 ºC, and 600 ºC. TCO must have the quality of low electrical resistivity between 10^{-3} and 10^{-4} Ω- cm, Resistivity in Table 1 lies between 10^{-3} and 10^{-4} Ω- cm which confirms the fabricated thin film as a suitable possible alternative to substitute current available transparent electrodes.

5. CONCLUSIONS AND RECOMMENDATION FOR FURTHER WORK

We studied the thermal effect on the properties of TiO_2 doped ZnO thin films. The XRD analysis revealed hexagonal wurzite crystal structure. Observation shows that the structure and crystallinity of thin film contain hexagonal wurzite (014). The transmittance (% T) of TiO_{2-} doped ZnO thin film at 400 ºC is in agreement for the standard transparent conducting oxides and band gap of the TiO_2 doped ZnO is larger than that of bulk ZnO. Electrical properties of ZnO improved by TiO_2 doping. Thin film annealed at 400 ºC will be highly suitable for transparent conducting oxide applications. The summary of the results show that TiO_2 doped ZnO is a transparent material and has potentials for electronic devices application as transparent conducting electrode. Further research work on solar cell device fabrication with the use of fabricated TiO_2 doped ZnO transparent electrode material should be a challenge to future researcher to ascertain the TiO_2 doped ZnO performance in active cell.

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7. COMPETING INTEREST

The authors declare no competing Interest.

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