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STRUCTURAL AND OPTICAL PROPERTIES OF TITANIUM DIOXIDE THIN FILM DEPOSITED BYSPIN-COATING TECHNIQUE

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ABSTRACT

This research investigates the effects of thermal annealing on the optical, morphological and structural properties of Tio_2 thin film. Tio_2 was deposited on glass substrate by spin –coating technique using spin –coater model laurel WS-650Hz-23NPP. Surface morphology of thin film was studied using Scanning Electron Microscope (SEM) model ASPEX 3020. The optical absorbance and transmittance measurements were recorded by using a single beam spectrophotometer Avantes model Avalight-DH-5-BAL. After annealing at different temperature for one hour from 100^0C to 550^0C with Carbolite tubular oven model Srw 21-501042 Type-CT17, the morphology, optical properties of TiO_2 thin film were obtained. Observation showed that as the annealing temperature increases there is increase in absorption of photon energy and as the wavelength increases the absorption decreases. It also shows that transmittance increases as the wavelength increases and decreases with increases in annealing temperature. SEM reveal as annealing temperature increases, the grain size of Tio_2 gradually increases. The energy gap was observed and determined, the band gap energy decreases from 3.3 eV to 3.05eV as the annealing temperature increases. The impact of heat treatment of TiO_2 thin-film on the microstructure, surface roughness, photon absorption, and the band gap energy was obtained.

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INTRODUCTION

Titanium dioxide (TiO_2) is the most investigated crystalline oxide in the surface science of metal oxides. Its physical and chemical properties are dominantly determined by its surface condition. Titanium dioxide has been studied extensively in the field of surface science due to the wide range of its applications and the expectation for insights into surface properties on the fundamental level (Diebold, 2003 and Liu, 2003). TiO_2 is a photocatalyst with relatively high efficiency, high transparency, excellent mechanically and chemically durable in the visible and near infrared region of spectrum. Titanium dioxide (TiO_2) is a white solid inorganic substance that is thermally stable and non-flammable. It is chemically inert. Titanium dioxide has high corrosive resistance, excellent optical transparency in visible and near infrared regions as well as high refractive index (Bakar, 2014; Abdullah, 2015 and Wu, 2014). Kurniawan Foe *et al.*, (2014) fabricated a poly [3-hexylthiophene] (P3HT) and [6,6]-phenyl-C61-butyric acid methyl ester ($PC_{61}BM$) organic photovoltaic cells (OPCs) using TiOx interfacial layer. Improvement in the photon absorption of the active layer was reported using TiO_xlayer coated organic photovoltaic solar cells (Kurniawan, 2014; Ourahmoun, 2010; Ilican, 2008).

EXPERIMENTAL PROCEDURES

Substrate preparation

Glass slides of dimensions25.4mm by76.2mm was rinsed in de-ionized (DI) water. They were then placed in a beaker containing isopropyl (IPA) before cleaning in VWR ultrasonic cleaner with Digital Timer and heater 97043-986 at room temperature for30 min. The cleaned glass slides were dipped in ethanol and dried in a stream of nitrogen gas (N_2). All glass slides received the same cleaning process.

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Preparation of Tio$_2$ solution

25ml Ethanol solvent was added into 3g of Tio$_2$ powder from Sigma Aldrich. The solution then underwent ageing process for 3 hours upon sonication at room temperature without heat to allow homogeneous mixture and Tio$_2$ powder to fully dilute into solvent. Ethanol was chosen as a solvent because it has no characteristic absorption and emission in the visible range.

Film processing and Deposition

Thin films were obtained by spin coatingthe solution of Tio$_2$ dropped onto the pre-cleaned glass substrate placed on the stub of the spin –coater model laurel WS-650Hz-23NPP.Which was rotated at 4000 rpm for 30 sec atroom temperature. After depositing by spin coating, the films were dried at 60°C for 30 min in an oven model Uniscope SM 9063 Laboratory oven SURGIFRIEND MEDICALS, England to evaporate the solvent. The process from coating to drying was repeated to obtain the desired thickness of the film. Filmswere annealed at different temperatures ranging from 100°C to 550°C for 1hr with Carbolite tubular oven model Srw 21-501042 Type-CT17 and quenched to room temperature in argon gasto study the effect of annealing. One sample was not subjected to heat treatment after the initial pre-heating at 60^{0}C for 30 min which depict control.

Optical Characterization

The optical transmittance of the samples was measured and recorded with Avantes UV-VIS spectrophotometer model Avalight-DH-5-BAL.The spectrophotometer operates by providing a light source towards an aperture using an optical fiber and uses another aperture to measure the amount of light transmitted or reflected by the sample under test. The transmittance spectrum is as shown in figure 1&2 The absorption spectra for Tio$_2$ thin films annealed at different temperatures is as shown in figure 3 It is deduced that as the annealing temperature increases there is increase in absorption of photon energy. It also shows that transmission increases as the wavelength increases and decreases with increases in annealing temperature. As the annealing temperature increases the absorption increases and as the wavelength increases the absorption decreases. To convert between the absorbance and transmittance, equation (1)was used.

$$\text{Absorbance (A)} = 2 - \log_{10\,(\%T)} \qquad (1)$$

The absorption coefficient of thin film is calculated from the formula (2) [9]

$$\alpha = 2.303(A/t) \qquad (2)$$

Where, A is absorbance and t is the thickness (Shakti, 2010). The absorption coefficient α and the extinction coefficient k are related by the formula (3)

$$K=\alpha\lambda/4\pi,\ K=2.303(A/t)\ \lambda/4\pi \qquad (3)$$

Extinction coefficient k is calculated with this formula $K=2.303(A/t)\ \lambda/4\pi$

Where, λ is the wavelength. The variation of extinction coefficient k with wavelength is shown in figure 4. Tio$_2$ is a direct –gap semiconductor. The optical band gap energy Eg and absorption coefficient α is related by the equation (4) (Tauc, 1970; Hussein, 2011; Balogun, 2017).

$$(\alpha hv)^2 = hv — Eg \qquad (4)$$

Where,α is the absorption coefficient and hv is the incident photon energy. Band Gap Energy (E) = hc/ λ where h = planks constant =6.626 x 10^{-34} joules sec. C= speed of light =3.0 x 10^8 meter/sec. where1ev =1.6 x 10^{-19} joules (conversion factor). For calculation of the optical band gap of films, the curve of $(\alpha h\upsilon)^2$ vs. hʋ was plotted. The energy band gap was obtained from straight line plot of $(\alpha h\upsilon)^{2\,/}$ vs. hʋ by extrapolating of the line to base line in Figure 7 to Figure 10.

Optical transmittance measurement

The optical transmittance of the samples was measured with spectrophotometer Avantes model Avalight-DH-5-BAL.

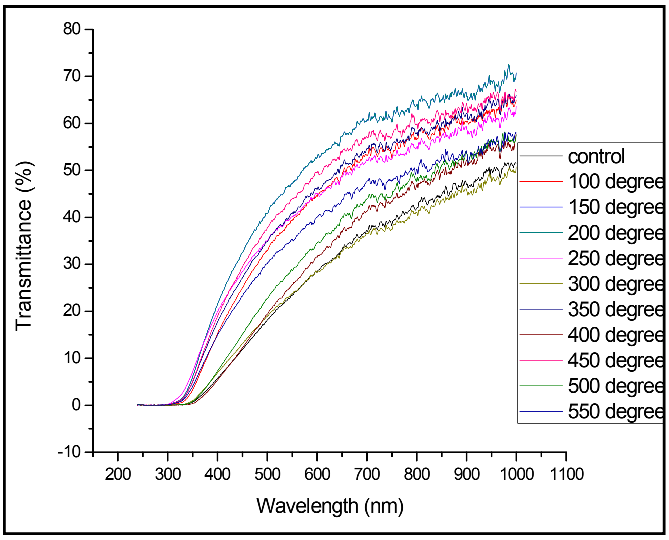

Figure 1. Plot of Transmittance vs Wavelength graph

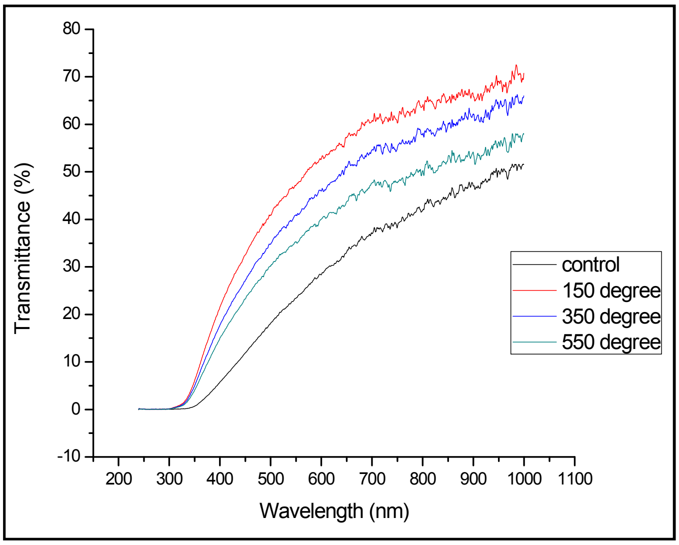

Figure 2. Plot of selected Temperatures of Transmittance vs Wavelength graph

Optical reflectance measurement

The opticalreflectance of the samples was measured with Avantes UV-VIS spectrophotometer model Avalight-DH-5-BAL. The plot of reflectance versus wavelength is shown in Figure 5 and Figure 6.

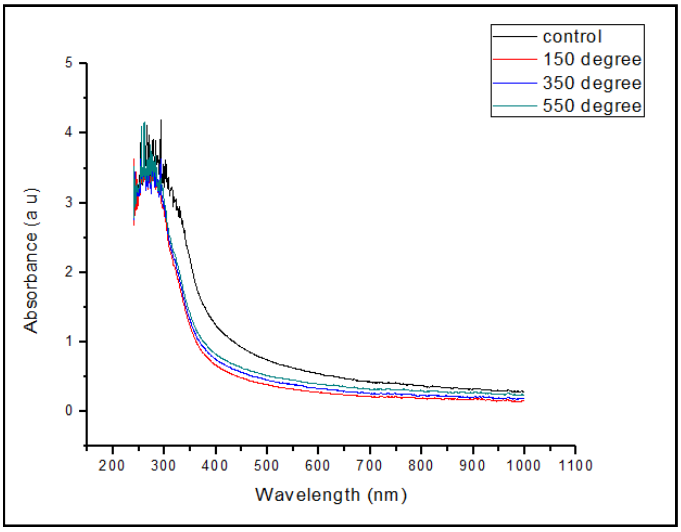

Figure 3. Plot of selected Temperatures of Absorbance vs Wavelength graph

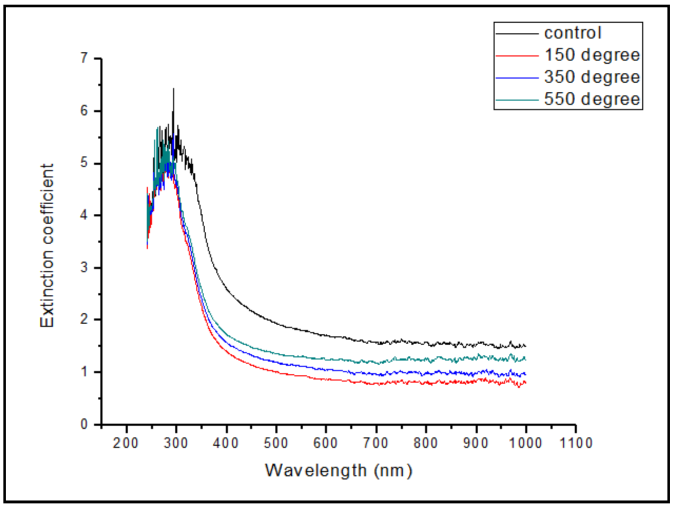

Figure 4. Plot of Extinction coefficient vs Wavelength graph

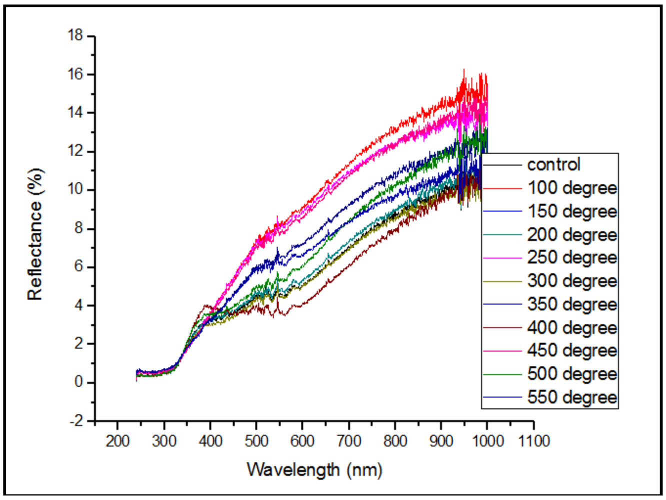

Figure 5. Plot of Reflectance vs Wavelength graph

Morphological Characterization

Scanning electron microscope (SEM) model ASPEX 3020 was employed to image the surface of the samples. All samples were appropriately cut to a size that can fit on the specimen stub of the SEM machine. SEM provides a useful means for investigating the morphological properties of the samples surface at desired magnification. SEM morphology study of samples was carried out to analyze the surface behavior.

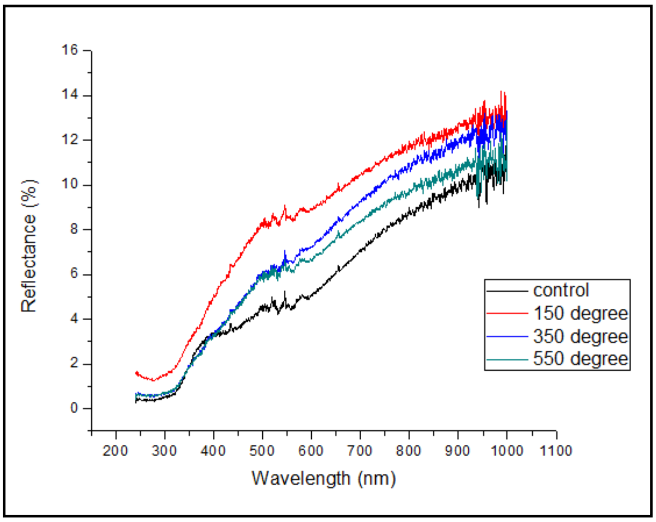

Figure 6. Plot of selected Temperatures of Reflectance vs Wavelength graph

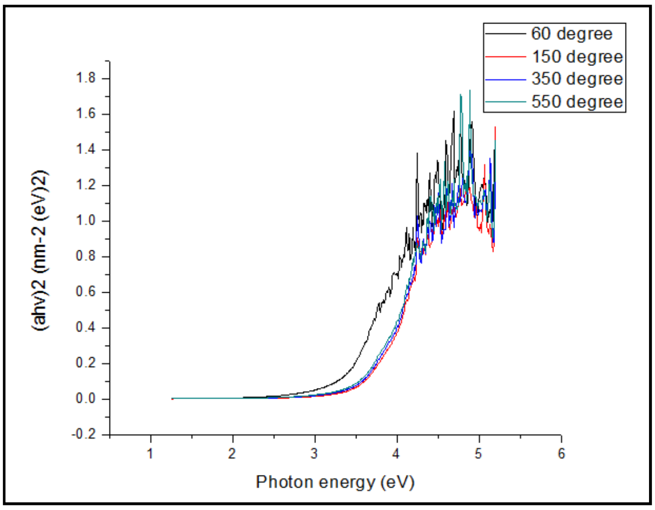

Figure 7. Plot of $(\alpha h\nu)^2$versus $h\nu$ for Tio_2 thin films annealed at different temperature

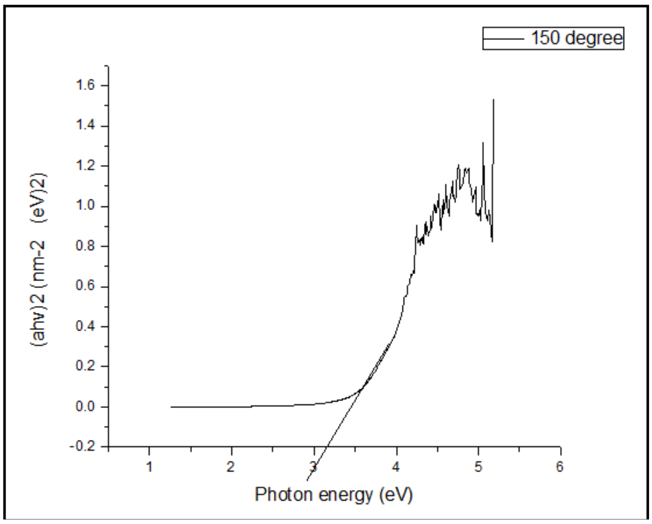

Figure 8. Plot of $(\alpha h\nu)^2$versus $h\nu$ for Tio_2 thin film annealed at 150^0c

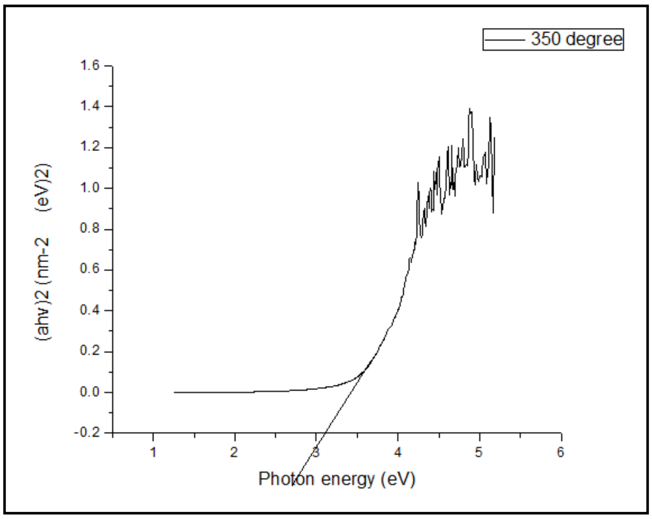

Figure 9. Plot of $(\alpha h\nu)^2$versus hν for Tio_2 thin film annealed at 350^0c

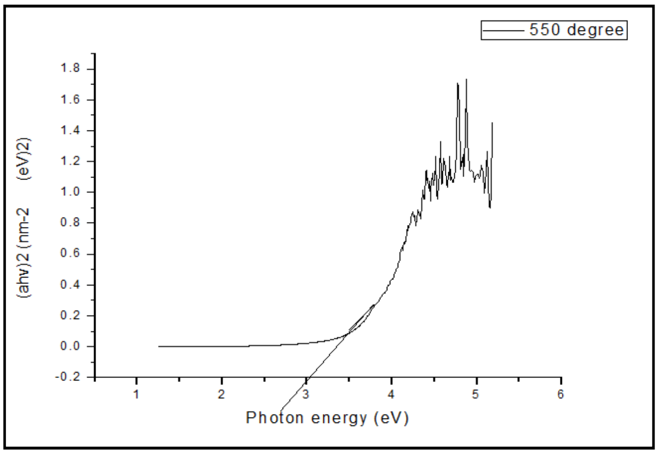

Figure 10. Plot of $(\alpha h\nu)^2$versus hν for Tio_2 thin film annealed at 550^0c

Figure 11 to Figure 14 shows images at different temperature in order to compare their surface morphologies with respect to the temperature variation. Observation showed that as the annealing temperature is increased the surface roughness increases. This may be due to increase in grain size with increase in annealing temperature. Figure 11 to figure 14 shows the morphological images at control, 150^0C, 350^0C, 550^0C

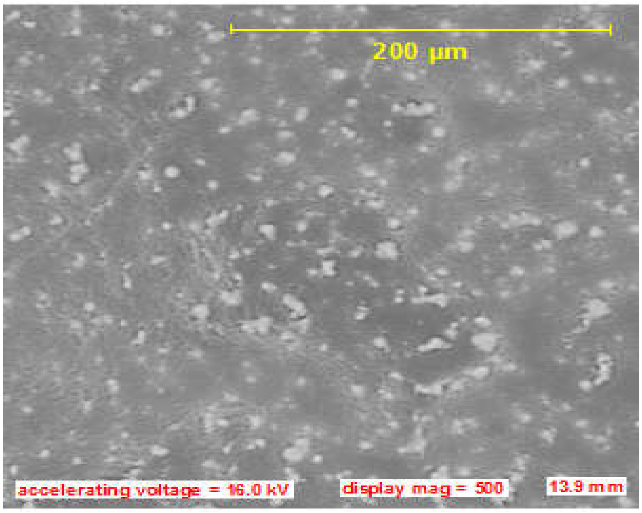

Figure 11. SEM image of Tio_2 control

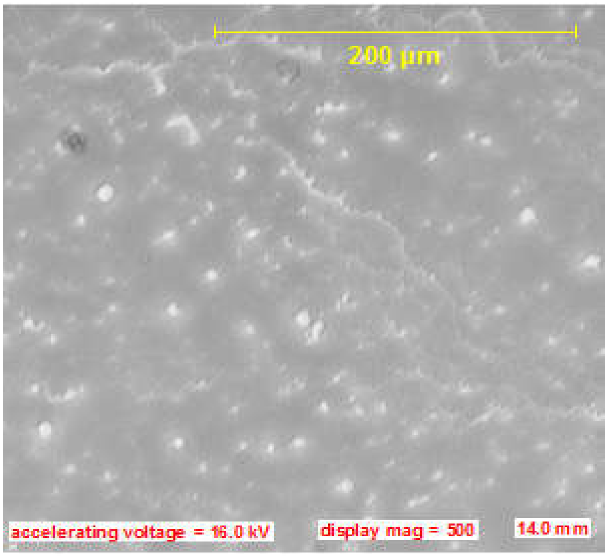

Figure 12. SEM image of Tio_2 at 150^0C

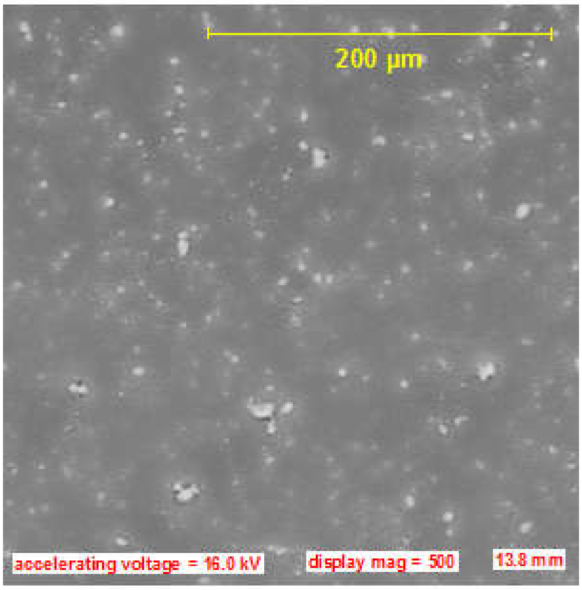

Figure 13. SEM image of Tio_2 at 350^0C

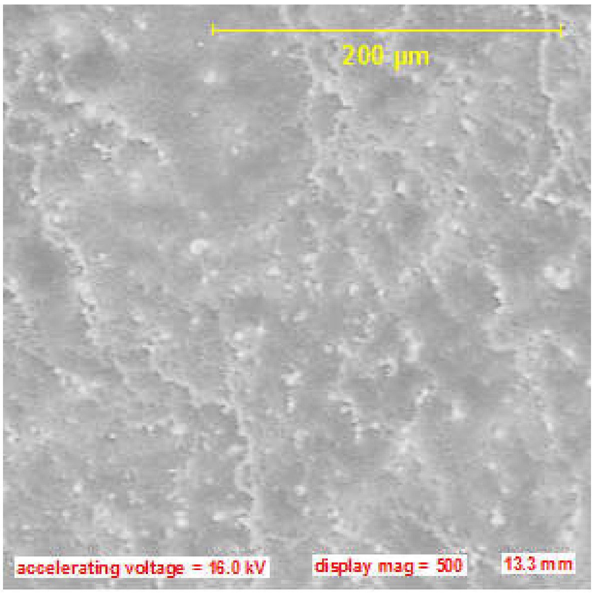

Figure 14. SEM image of Tio_2 at 550^0C

RESULTS

It was deduced that as the annealing temperature increases there is increase in absorption of photon energy. It also shows that transmission increases as the wavelength increases and decreases with increases in annealing temperature. As the annealing temperature increases the absorption increases and as the wavelength increases the absorption decreases. Titanium dioxide thin films prepared on glass substrate by spin coating process and annealed at different temperatures from 100ºC to 550ºC step of 50^0C. It was deduced that annealing temperature affects the morphological and optical properties of the Tio_2 thin films. The transmission spectrums of the films were recorded by UV-VIS Spectrophotometer. Extinction coefficients were calculated. The extinction coefficient showed some variation with rise in annealing temperature of Tio_2 films. The Optical energy band gap values were obtained by plot of $(\alpha h\nu)^2$ versus hν. The value of band gaps agrees approximately with that of bulk Tio_2. As the annealing temperature increases the band gap value decreases. SEM image of the samples revealed that the roughness of the surface of the sampleincreasesas annealing temperature increases, this is attributed to increase in grain size, so that band gap energydecreases.

DISCUSSION

Unheated sample (control) has the least percent transmittance compared to the heated samples at 100^0C to 550^0C. Un-annealed sample has the highest absorbance in the visible spectrum and near infrared spectrum compared to the thermally annealed samples and it also has the highest extinction coefficient value. But has the least band gap energy approximately 2.8eV compared to annealed samples. SEM analysis showed that surface roughness of unheated sample is the highest.

Conclusion

The Titanium dioxide thin film was deposited and annealed at 100ºC to 550ºC in step of 50^0C, respectively. The result shows that the film annealed at 550°C has the highest absorption in the visible region. The unheated sample (control) has the highest extinction coefficient value but has the least band gap energy. The band gap energy of the thin filmdecreases as the annealing temperature increases. As the annealing temperature increases the band gap energy value decreases from 3.3 eV to 3.05eV. SEM image of the samples revealed that the roughness of the surface of the thinfilmincreases as annealing temperature increases, this is attributed to increase in grain size, so that band gap energy decreases. Heat treatment will improve the stability or efficiency of Tio_2 thin film.

REFERENCES

Abdullah, S. M., Ahmad, Z., Sulaimana, K. 2015. The Impact of Thermal Annealing to the Efficiency and Stability of Organic Solar cellsProcedia - *Social and Behavioral Sciences*., 195 2135 – 2142, 2015.

Bakar, N. A. Supangat, K. Sulaiman, Controlling the morphological, structural, and optical properties of one-dimensional PCDTBT nanotubes by templatewetting, Nanoscale Research Letters, 9,1-6, 2014.

Balogun, S. W., Ezike, S. C., Sanusi, Y.K., Aina. A. O. 2017. Effects of Thermal Annealing on Optical Properties of Poly (3-Hexyithiophene): [6,6]-Phenyl C6o-Butyric Acid Methyl Ester Blend Thin Film. *Journal of Photonic Materials and Technology.,* Vol. 3, No. 2, 2017, pp. 14-19. doi: 10.11648/j.jmpt.20170302.12

Diebold, U. 2003. “The surface science of titanium dioxide” Surface Science Reports, vol. 48, no. 5–8, pp. 53–229, 2003.

Hussein, H.F., Shabeeb, G. M., Sh. Hashim. S. 2011. Preparation ZnO Thin Film by using Sol-gel-processed and determination of thickness and study optical properties. J. Mater. Environ. Sci. 2 (4) 423-426, 2011. http://www.jmaterenvironsci.com

Ilican, S., Caglar, Y. Caglar, M. 2008. Preparation and characterization of ZnO thin films deposited by sol-gel spin coating method. *Journal of optoelectronics and advanced materials* Vol. 10, No. 10. 2578 – 2583.

Kumar, K. Balachandr, P. Raji, Synthesis and Characterization of Nano Zinc Oxide by sol gel Spin Coating. *Recent Research in Science and Technology*, 3(3): 48-52, 2011 www.recent-science.com

Kurniawan, F., Namkoong, G., Samson, M, Younes, E. M., Nam, I., Abdel-Fattah, T.M. 2014. Stability of High Band Gap P3HT: PCBM Organic Solar Cells Using TiOx Interfacial Layer Journal of Photoenergy (2014) Volume 2014, Article ID 784724, 6pages http://dx.doi.org/10.1155/2014/784724

Liu, H., Ma, H. T., Li, X. Z., Li, W. Z., Wu, M. and Bao, X. H. “Theenhancement of TiO2 photocatalytic activity by hydrogen thermal treatment,” Chemosphere, vol.50, no.1, pp. 39–46, 2003.

Ourahmoun, O., Belkaïd M.S. 2010. Dependence of the characteristics of organic solar cells on cathode polymer interface Revue des *Energies Renouvelables* Vol. 13 N°4, 583 – 590.

Shakti, Nanda , P. S. Gupta, Structural and Optical Properties of Sol-gel Prepared ZnO Thin Film. *Applied Physics Research*., Vol. 2, No. 1. 19-28, 2010.

Tauc, J. 1970. The optical properties of solids (North-Holland, Amsterdam, 1970).

Wu, Q., Bhattacharya, M., L.M.J. Moore, S.E. Morgan, 2014. Air processed P3HT:PCBM photovoltaic cells: Morphology correlation to annealing, degradation, and recovery, *Journal of Polymer Science Part B: Polymer Physics,* 1511–1520, 2014.

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