TEMPERATURE DEPENDENCE OF HETEROGENEOUS CdO-NiO NANOCOMPOSITE THIN FILMS

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Abstract

This research aims to synthesize the CdO-NiO nanocomposite thin films deposited on fluorine tin oxide (FTO) glass substrates were prepared by spin coating method. Cadmium sulphate, nickel sulphate, ammonia and polyethylene glycol were used as the starting materials to obtain CdO-NiO nanocomposite powders by sol-gel method at 400 ºC, 500 ºC, 600 ºC and 700 ºC for 2 hrs. Then the precursor solution of CdO-NiO nanocomposite were coated on FTO glass substrates. The phase formation, structural properties and surface morphology of nanocomposite were characterized by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The absorption wavelengths and energy band gap of CdO-NiO nanocomposite thin films were measured with the help of UV-Vis Spectroscopy. CdO-NiO nanocomposite thin films can be promoted further experimental studies such as thin films solar cells, memory devices and optoelectronic materials.

Keywords: CdO, NiO, XRD, SEM and UV-Vis Spectroscopy

Introduction

Thin film technology is the development of very thin layers of material to deposit on the surface of objects, ranging from semiconductors to plate glass. Thin film offers a number of benefits to manufacturers including reduced production costs, smaller and more lightweight finished products and increased flexibility [Ashfari. B, et al, 2001]. From variety of materials including silicon and ceramic can make thin film. Thin film technologies are process for depositing and processing thin layers from a micron thick down individual atomic layer. One use of thin film technology is in semiconductor production, including development of new photovoltaic cells [Al-Juaid.F et al, 2012]. A nanocomposite is a multiphase material in which, in contrast to micro composites, one of the phases has one, two or three dimensions of less than 100 nm or the composite phases have nanoscale distances between them [Abou-Helal M.O, 2002]. Based on the matrix material, nanocomposites are classified into polymer matrix composites, metal matrix composites, and ceramic matrix composites. Nanocomposites are materials that have a solid structure in which the distance between the phases is least wise formed of dimension with nanoscale size and general form of an organic phase, or vice versa, form an organic matrix set in the organic phase [Feinle, M. S. et al, 2016]. Nanocomposite materials can exhibit individual properties such as mechanical, chemical, electrical, optical and catalytic properties [Liao.Y, Xu.Y, et al., 2013]. Nanocomposites are the heterogeneous/ hybrid materials that are produced by the mixtures of polymers with inorganic scale. Their structures are more complicated than that of micro composites [Niederberger.M, et al., 2009]. A nanocomposite combines two or more materials of which at least one is a nanomaterial with different physical and chemical properties. Nanocomposites are prepared by chemically oriented synthetic methods: soft lithography, lamination, spin coating or solution casting [Owens. G.J, et al., 2016]. The primary challenge in the processing of nanocomposite is achieving a homogeneous dispersion of the nanoparticles.The

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application areas for nanocomposites involves medical areas and spans many industries from electronics to automotive to sport equipment [Sule.E.E, 2013].

Materials and Methods

Sample Preparation of CdO-NiO Nanocomposite Powder

Materials

For the preparation of experimental details, the main starting chemicals cadmium sulphate ($CdSO_4$) and nickel sulphate ($NiSO_4$) are reagents used for the preparation of CdO-NiO nanocomposite. Starting materials were purchased from Academy chemical store in the downtown of Yangon. This chemical was analytically pure and directly used as received without further purification.

Methods

The synthesis method consisted of one step (1) the preparation of CdO-NiO nanocomposite powder.

Sample Preparation of CdO-NiO Nanocomposite Powder

In this work, starting chemicals such as cadmium sulphate ($CdSO_4$), nickel sulphate ($NiSO_4$), polyethylene glycol ($C_{2n}H_{4n+2}O_{n+1}$) and ammonia (NH_3) were of analytical grade and used without further purification. The CdO-NiO nanocomposites were synthesized by using sol-gel method and distilled water was used as the dissolving solvents throughout in the study. The molar ratio of 1:1 for cadmium sulphate dehydrate and nickel sulphate dehydrate were firstly dissolved in distilled water to form 0.1 M molar solution for preparing the nanocomposite. Secondly, the resulting mixed solutions were stirred vigorously onto the magnetic stirrer (1000 rpm) at room temperature for 4 hrs. While the stirring process, the reducing agents of ammonia and poly ethylene glycol were added drop by drop into the mixed solution for adjusting the pH level until 7 to obtain the CdO-NiO nanocomposite. Thirdly, the homogeneous solution was cooled to settle down for 24 hrs at room temperature. The next day upper parts of the solutions were decanted to the beaker and the obtained sludge was filtered through filter papers. Finally, the paste of CdO-NiO composite was dried at 100 ºC for 24 hrs and then calcined at 400 ºC, 500 ºC, 600 ºC and 700 ºC for 2 hrs. The structure and crystallization of CdO-NiO nanocomposites were examined by XRD machine, the surface morphological characteristics were determined by SEM and the optical properties and energy band gaps were also detected by UV-Vis Spectroscopy.

Thin Films Preparation of CdO-NiO Nanocomposite

Firstly, the 1 cm × 1 cm FTO /glass substrates were firstly cleaned. The precursor solution of CdO-NiO nanocomposites were coated onto the fluorine doped tin oxide (FTO)/glass substrates by spin coating method. The cleaning glass substrates were tested the conducting layers with digital meter. The prepared CdO-NiO nanocomposite powders were dissolved with 2-methoxyethanol ($C_3H_8O_2$) as solvent to form viscous paste at 80 °C for 3 h. This viscous paste was then coated onto fluorine doped tin oxide (FTO) / glass substrates at 2500 rpm for 30 s by spin coating technique. CdO-NiO solution was deposited onto chemically cleaned FTO conductive glass substrates. The nanocomposite layer coated films were annealed at 100 °C for 1h for diffusion films. Then, the films were cooled slowly at room temperature. The distribution and morphological characterizations of films were examined by Scanning Electron Microscopy (SEM). Optical absorption spectra of the nanocomposite films were examined using a UV- Vis spectroscopy. Figure 1(a~d) showed the photos of preparation of CdO-NiO nanocomposite thin films. The block diagram of preparation of CdO-NiO nanocomposite thin films was shown in Figure 2.

Figure 1 (a) Testing Conducting Layer

Figure 1 (b) Spinning Process

Figure 1 (c) Annealing Process

Figure 1 (d) CdO-NiO Thin Film

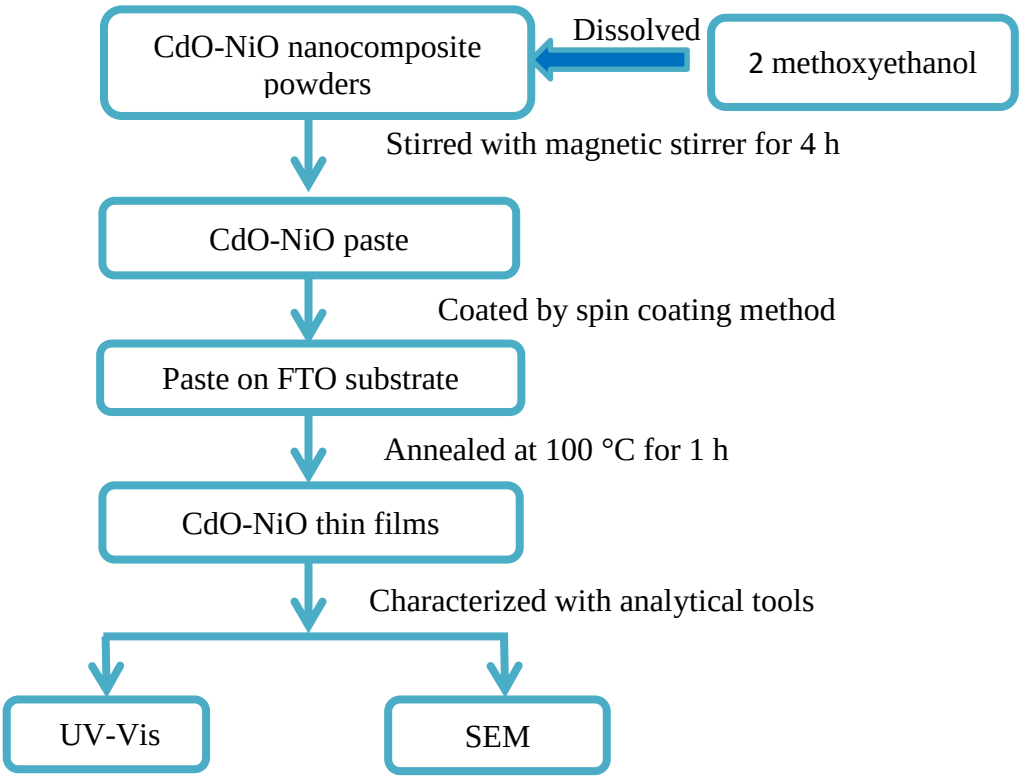

Figure 2 The Block Diagram of Preparation of CdO-NiO Nanocomposite Thin Films

Results and Discussions

Characterization Techniques

The nanocomposite synthesis of CdO-NiO was characterized by XRD, SEM, and UV-Vis spectroscopy for structural, morphological, and optical properties.

XRD Analysis of CdO-NiO Nanocomposite Powder

The structural analysis of CdO-NiO nanocomposite powders was examined by the XRD technique. To examine the crystal structure and phase formation of CdO-NiO nanocomposite powders at 400 °C, 500 °C, 600 °C and 700 °C, it was performed using monochromatic CuK_{α} radiation (λ=1.54056Å) operated at 40 kV (tube voltage) and 40 mA (tube current). The results showed that all the diffraction peaks of CdO-NiO powder can be perfectly matched with JCPDS library file for cubic structure. The distinct peaks were found to be (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) crystal planes for CdO and (1 1 1), (2 0 0), (2 2 0) planes for NiO respectively. The average crystalline sizes of CdO-NiO nanocomposites powders at 400 °C, 500 ºC, 600 ºC, and 700 ºC were 34.2118 nm, 36.3477 nm, 41.6114 nm and 48.5123 nm respectively. The crystalline size was calculated by using Debye Scherrer's formula, ($G=\frac{0.899 \times \lambda}{B \times \cos\theta}$). The XRD pattern of CdO-NiO nanocomposite powders at different temperatures was shown in Figure 3.

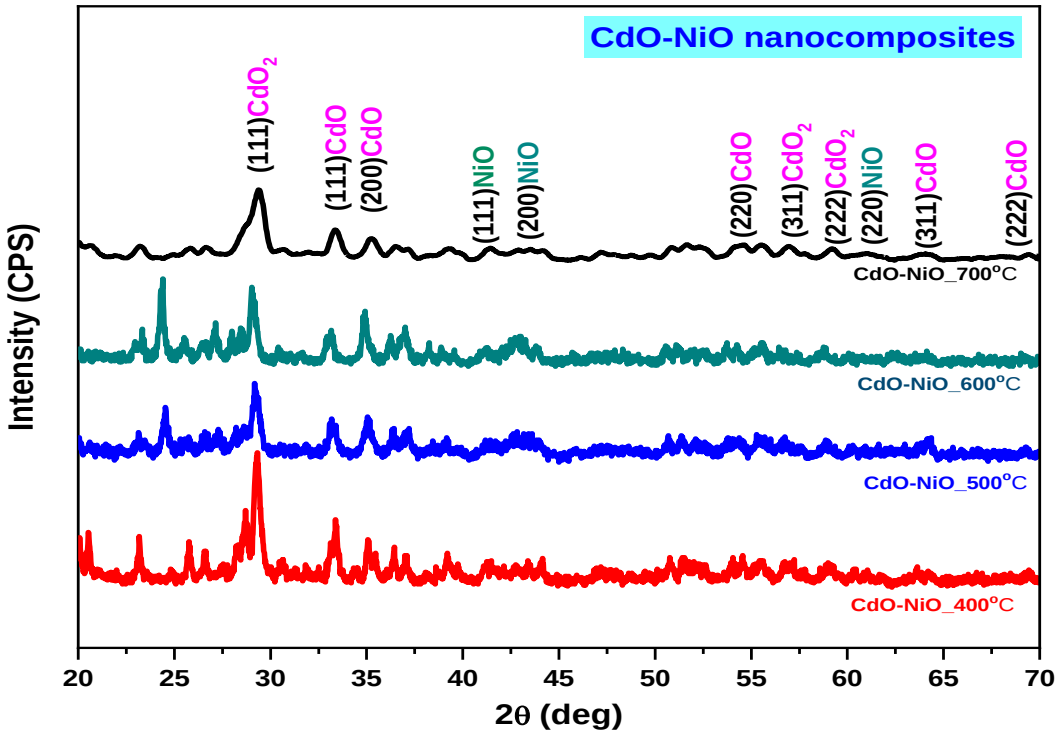

Figure 3 The XRD Pattern of CdO-NiO Nanocomposite Powders at Different Temperatures

SEM Analysis of CdO-NiO Nanocomposite Films

Surface morphology of CdO-NiO nanocomposite thin films on FTO/glass substrates were carried out by SEM analysis. SEM images of CdO-NiO nanocomposite thin films for the different temperatures at 400 ºC, 500 ºC, 600 ºC and 700 ºC were shown in Figure 4(a~d). As the detail analysis of SEM image, it was found that flat and a little crack and more uniform particle size than powder images. These images showed smaller particles features in microstructure. The grain sizes of CdO-NiO nanocomposite thin films were found to be 1.27 µm, 1.18 µm, 1.11 µm and 1.03 µm,

respectively. From SEM analysis, the surface of the films became rough, but the sizes are smaller after annealing different temperatures. On the other side, the achieving relatively films cracking was decreased with the increasing temperature.

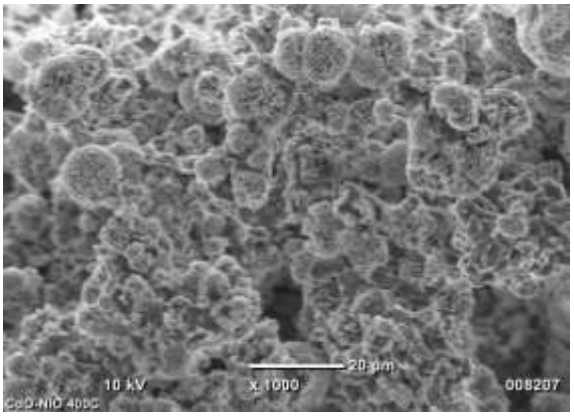

Figure 4 (a) SEM Micrograph of CdO-NiO Nanocomposite Film at 400 ºC

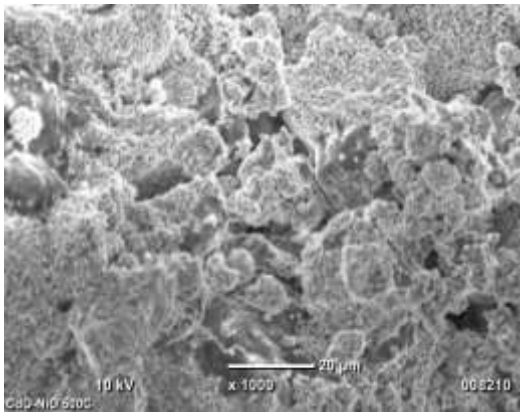

Figure 4 (b) SEM Micrograph of CdO-NiO Nanocomposite Film at 500 ºC

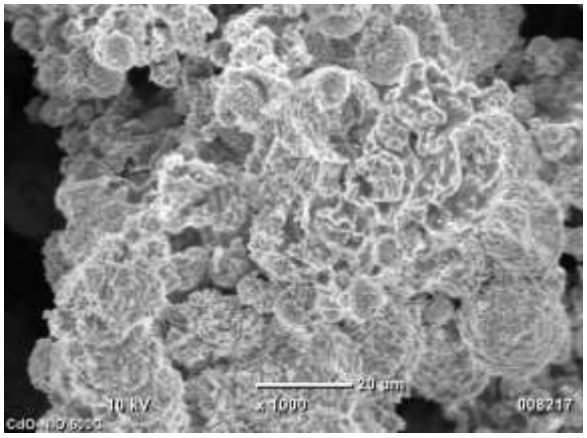

Figure 4 (c) SEM Micrograph of CdO-NiO Nanocomposite Film at 600 ºC

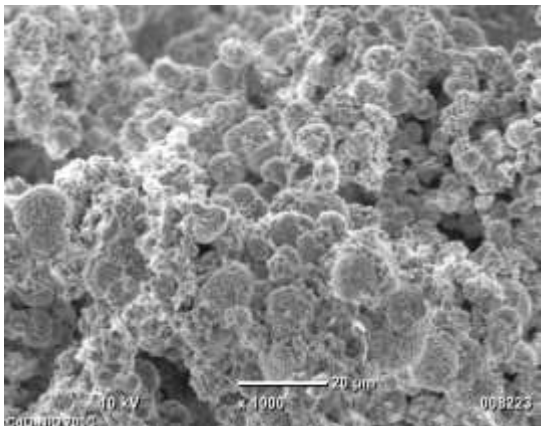

Figure 4 (d) SEM Micrograph of CdO-NiO Nanocomposite Film at 700 ºC

UV-Vis Analysis of CdO-NiO Nanocomposite Thin Films

The absorption spectrum and the energy band gap of CdO-NiO nanocomposite thin films were performed using UV-Vis spectrometer. The wavelength ranges of spectrum laid between 200 nm to 700 nm as shown in Figure 5 (a~d). UV-Vis analysis determined the variation of the absorption coefficient (α) as a function of the photon energy at room temperature. The optical band gap (E_g) was evaluated from the absorption spectrum and the optical absorption coefficient (α) near the absorption edge. The optical band gap energy in absorption edge of CdO-NiO nanocomposite thin films were given in Table 1. The alternative method to observe the band gap is calculated by Tau's relation. The $(\alpha h\upsilon)^2$ and $h\upsilon$ characteristic curve of CdO-NiO nanocomposite thin films at 400 ˚C, 500 ˚C, 600 ˚C and 700 ˚C were shown in Figure 6 (a~d). On the characteristic curve, the extrapolating the straight line onto horizontal axis ($(\alpha h\upsilon)^2 = 0$), give the values of band gap energy obtained CdO-NiO nanocomposite thin films had a direct band gap 2.69 eV at 400 ˚C, 3.83 eV at 500 ˚C, 4.05 eV at 600 ˚C and 4.29 eV at 700 ˚C respectively.

Table 1 The observed energy band gap of CdO-NiO nanocomposite thin films at different temperatures

Sr No.	Temperature (°C)	Observed energy band gap (eV)
1	400	2.69
2	500	3.83
3	600	4.05
4	700	4.29

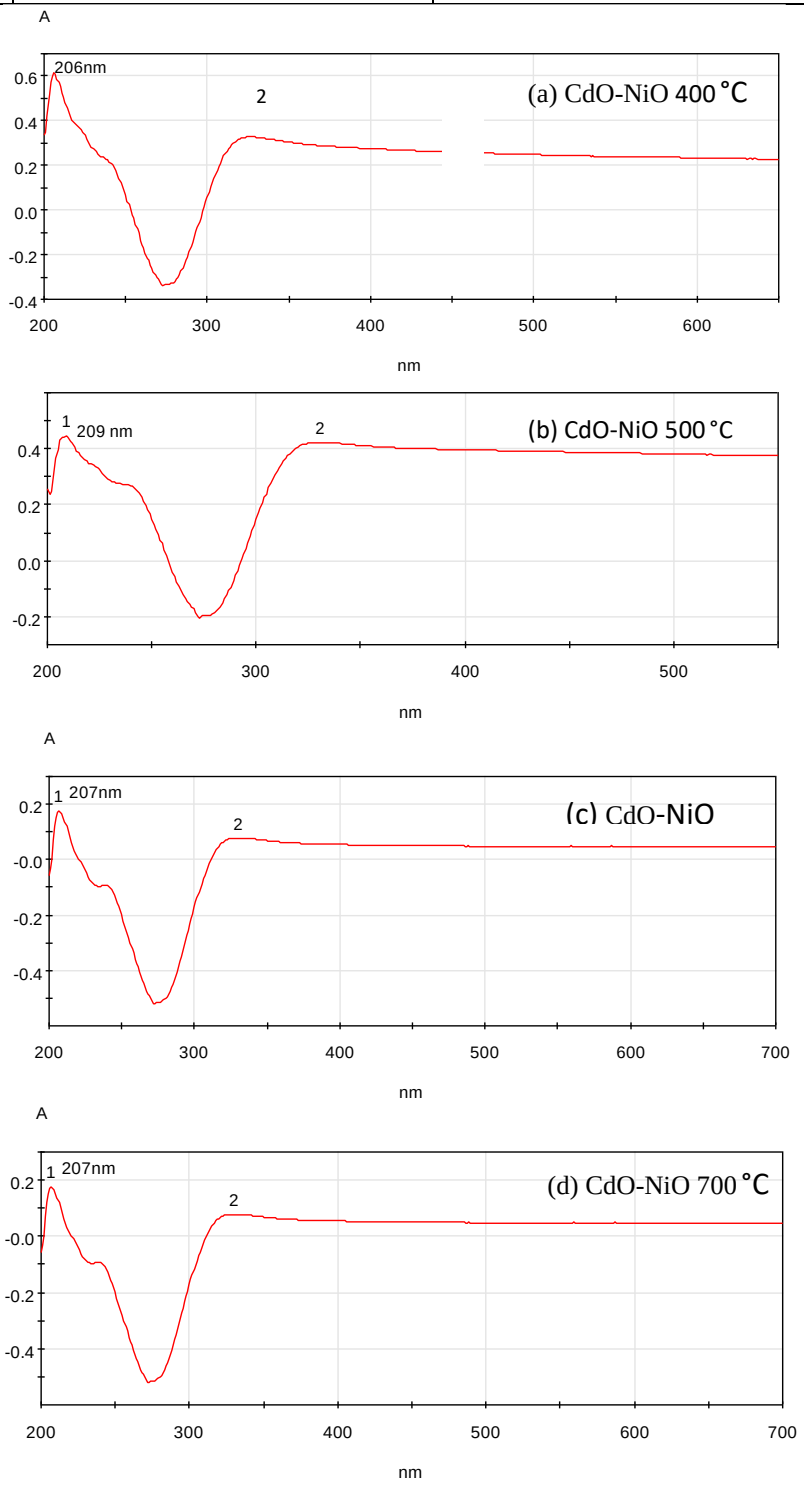

Figure 5(a~d) Absorption Spectrum of CdO-NiO Nanocomposite Film at Different Temperatures

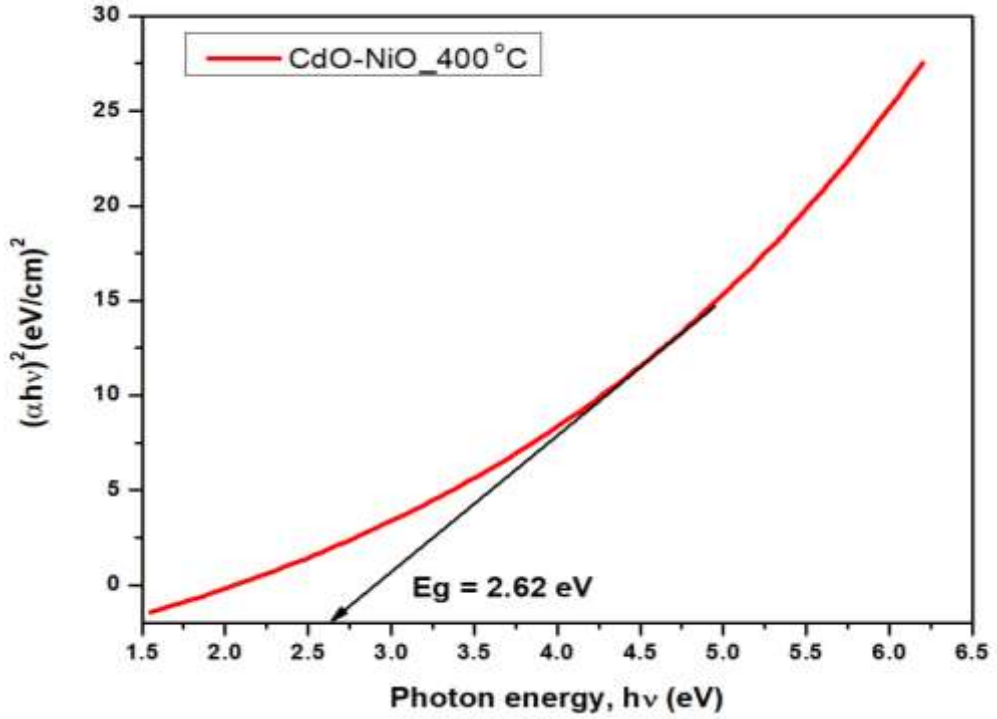

Figure 6 (a) Energy Band Gap Values of CdO-NiO Nanocomposite Thin Film at 400 °C

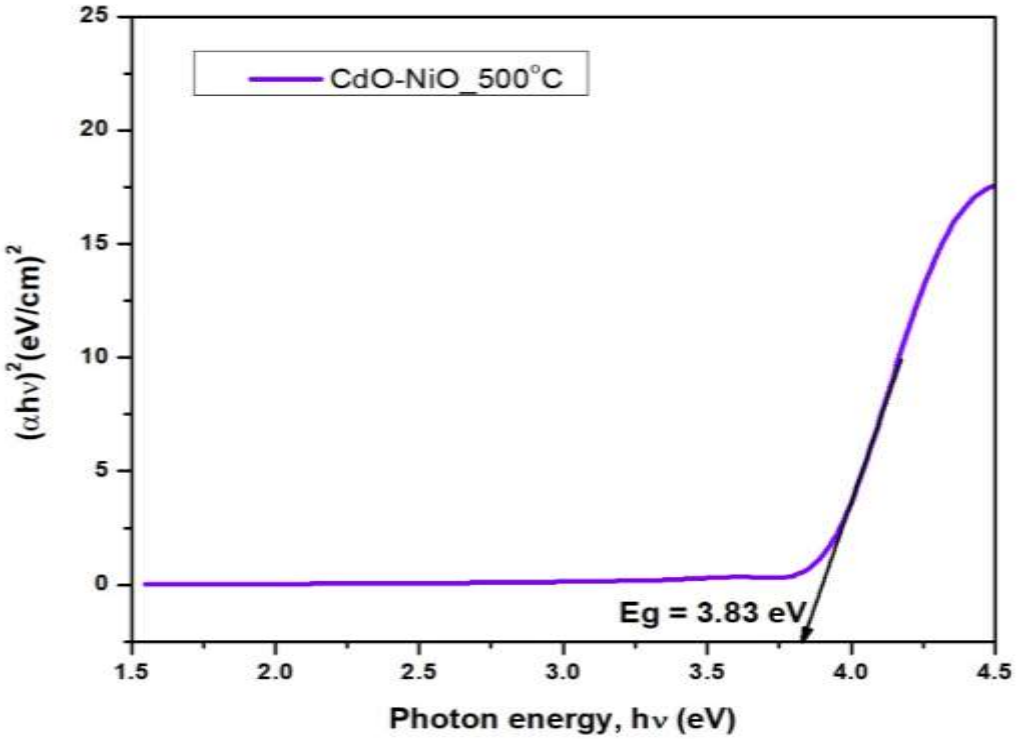

Figure 6 (b) Energy Band Gap Values of CdO-NiO Nanocomposite Thin Film at 500 °C

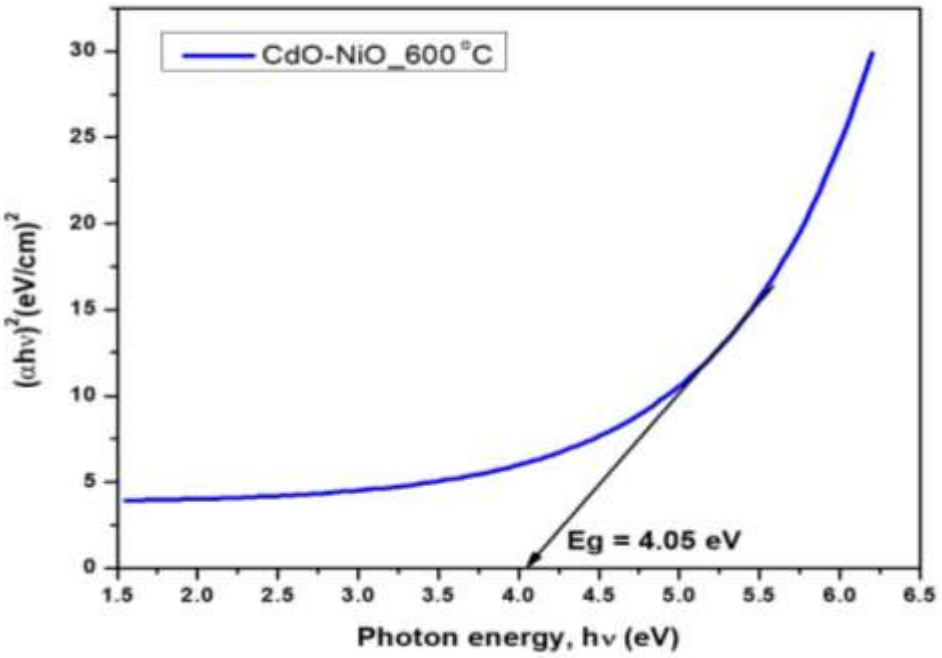

Figure 6 (c) Energy Band Gap Values of CdO-NiO Nanocomposite Thin Film at 600 °C

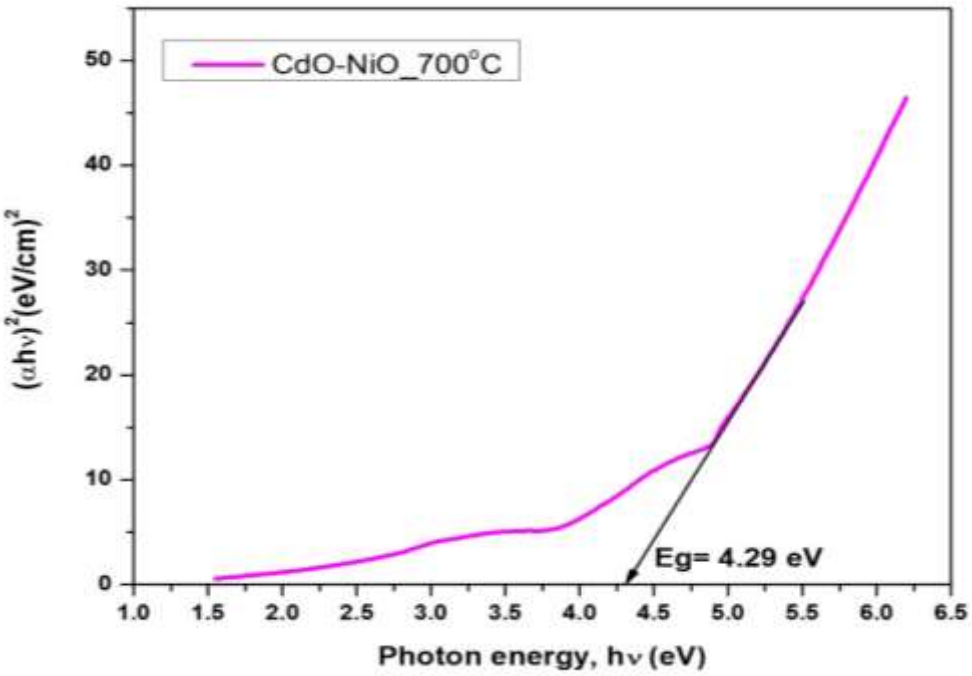

Figure 6 (d) Energy Band Gap Values of CdO-NiO Nanocomposite Thin Film at 700 °C

Conclusion

In conclusion, the temperature dependences of synthesis and characterization of CdO-NiO nanocomposites powders were firstly prepared by sol-gel reaction method at 400 ˚C, 500 ˚C, 600 ˚C and 700 ˚C respectively. Then, CdO-NiO nanocomposites thin films were be deposited onto FTO glass substrates by spin coating technique. According to XRD images, the XRD patterns of nanocomposites powders were well matched with corresponding standard library files. From the XRD results of CdO-NiO nanocomposites powder, the average crystalline sizes of CdO-NiO nanocomposites powders at 400 °C, 500 ºC, 600 ºC, and 700 ºC were 34.2118 nm, 36.3477 nm, 41.6114 nm and 48.5123 nm respectively. The crystal structures of nanocomposites powders were found to be cubic structure. When the annealing temperatures were increased, the average crystallite sizes of the CdO-NiO nanocomposites powders were also increased. From SEM analysis of CdO-NiO nanocomposite powders, some grains were large with agglomerated structures. The average grain size of the samples was 0.76 µm at 400 ºC, 0.69 µm at 500 ºC, 0.67 µm at 600 ºC and 0.55µm at 700 ºC respectively. As the results, it was concluded that the average grain sizes of the powder were decrease with increased annealing temperatures. For thin films, according to SEM results, the surface of the films became rough, but the sizes are smaller after annealing different temperatures. On the other side, the achieving relatively films cracking was increased with the decreasing temperature. According to the UV-Vis analysis results, the widest energy bandgap of CdO-NiO nanocomposites powders were 4.29 eV at 700 °C and the smallest energy bandgap was 2.69 eV at 400 °C. The intensity of absorption peak and energy band gaps increases with increasing temperatures. The experimental finding of CdO-NiO nanocomposites resulted from this research work indicated that the crystal structure, phase formation and energy band gaps were influenced by different annealing temperatures.

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References

Ashfari. B, et al, (2001), “single crystalline rocksalt CdO layers grown on organic molecular beam epitaxy”, Appl. Lett, vol.79, pp.470-472.

Abou-Helal MO & W.T. Seeber, (2002), Preparation of TiO_2 thin films by spray pyrolysis to be used as photocatalyst, Applied Surface Science 195,53.

Feinle, M. S. et al, (2016), ¨using, “Sol-gel synthesis of monolithic materials with hierarchical porosity,” Chemical Society Reviews, vol. 45, no. 12, pp. 3377–3399.

Liao.Y, Xu.Y, and Chan.Y., (2013),“Semiconductor nanocrystals in sol-gel derived matrices,” Physical Chemistry Chemical Physics, vol. 15, no. 33, Article ID 13704.

Merazga. A, Al-Juaid. F, Abdel-Wahab. F and Al-Amoudi. M.N, (2012), "ZnO Spin-Coating of TiO_2 photo-electrodes to enhance the efficiency of associated dye-sensitized solar cells", World J. Condensed Matter Physics. vol. 2, pp.192-196.

Niederberger.M and Pinna.N., (2009), Metal Oxide Nanoparticles in Organic Solvents: Synthesis, Formation, Assembly and Application (Engineering Materials and Processes), Springer, Berlin, Germany.

Owens. G.J, Singh. R.K, Foroutan. F et al., (2016), "Sol-gel based materials for biomedical applications," Progress in Materials Science, vol. 77, pp. 1–9.

Sule.E.E, (2013), "Photovoltaic Performance of ZnO Nanorod and ZnO: CdO Nanocomposite Layers in Dye-Sensitized Solar Cells (DSSCs)" International Journal of Photoenergy, Article ID 436831.