

ORIGINAL ARTICLE

Influence of Applied Potential on The Structural and Optical Properties of Cu₂O Thin Films Grown by Electrochemical Deposition

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ABSTRACT – Cu₂O thin films have been deposited on indium-tin-oxide (ITO) coated glass substrate by electrochemical method or electrodeposition. The effect of potential deposition on the microstructure and optical properties of Cu₂O thin films is studied. Electrodeposition for two Cu₂O thin films was carried out at -0.5 V and -0.6 V, relative to the standard calomel electrode (SCE) as a reference electrode. Cu₂O thin films were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), and UV-Visible spectroscopy. The diffraction pattern indicated that the cubic structure of the Cu₂O crystal has been well grown. The lattice parameters of Cu₂O films slightly increased as the potential of electrodeposition increased. The crystallite size of Cu₂O films significantly enhanced as the enhancing applied potential. Cu₂O thin films showed a smooth and flat surface morphology based on SEM images. The optical transmittance of the Cu₂O thin films drastically decreased as the applied potential increased due to the increasing film thickness. The bandgap energy of Cu₂O films based on the Tauc plot increased as the applied potential increased, that is 2.0 eV for the film deposited at -0.5V and 2.13 eV deposited at -0.6V.

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INTRODUCTION

Semiconductor material is an important component in electronic device technology. Some of them are formed into thin films to improve their performance and expand their applications. One of semiconductor material that can be formed into thin films is copper oxide (Cu₂O). Cu₂O is a semiconductor material that has been known for a long time and has been extensively explored for its optical and electrical properties as well as its magnetic properties for related applications. Cu₂O is oxide with cubic lattice (cuprite structure) in its crystal structure. The electrical and optical properties of Cu₂O films are the focus of many studies due to their wide application potential. Cu₂O is a p-type semiconductor that has bandgap energy ranging from 1.9 to 2.2 eV [1], [2]. The bandgap energy of the Cu₂O semiconductor is a suitable range for solar energy conversion because it can absorb visible spectrum energy from the sun making it suitable for solar cell applications [3]-[5]. Besides being applied as solar cells, Cu₂O is also widely applied as gas sensors [6], [7], photocatalyst for organic dyes degradation [8], glucose sensors [9], photoelectrochemical water splitting [10], and thin-film transistor [11].

There are several techniques which can be applied to grow a Cu₂O thin film, including by anodizing Cu metal [2], electrochemical deposition (electrodeposition) technique [1], [5], [8], [12]-[14], plasma sputtering method [15]-[17], pulsed-laser deposition [18], successive ionic layer adsorption and reaction (SILAR) method [19], chemical vapor deposition [20], spray pyrolysis [21], sol-gel spin coating method [22], and chemical bath deposition (CBD) [23], [24]. The electrodeposition method is the most familiar and most widely used for growing Cu₂O thin films [12]-[14], [25]. Several studies related to the electrochemical deposition of Cu₂O thin films were carried out with different treatment parameters, including the deposition potential [13] as well as the ion type and concentration of the electrolyte solution [3], [14]. For example, research conducted by Jiang et al. (2019) varied the deposition potential and studied its effect on the microstructure and optical properties of Cu₂O thin films [1].

Electrochemical deposition or electrodeposition is an easy, inexpensive, and effective way to grow Cu₂O thin films on conductive substrates, such as transparent conducting oxide (TCO)-coated glass, for instance indium-tin-oxide (ITO)-coated glass substrates. The deposition parameters greatly affect the characteristics of the Cu₂O thin film, including microstructure, electrical properties, electrochemical and optical properties [25], [26]. In electrochemical deposition technique, several deposition parameters which can be varied are deposition potential [13], bath temperature [12], pH of the electrolyte solution, as well as type and concentration of electrolyte ions [3], [14]. Previous studies have shown that deposition potential is a parameter that greatly influences the characteristics and properties of Cu₂O films, including microstructure, electrical, and optical properties [1], [13], [25], [26].

The purpose of this research is to study the effect of applied potential on the characteristics of Cu₂O thin films grown by electrochemical deposition. Cu₂O thin films were deposited on an indium-tin-oxide (ITO)-coated glass substrate. The structure, morphology, and optical properties of Cu₂O thin films were studied under the influence of applied potential in electrochemical deposition.

EXPERIMENTAL METHOD

Materials and Instruments

Chemicals used in this study are $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Merck), NaOH (Merck), and lactic acid (Merck), which are analytical grade. The equipment used in this study includes Potentiostat/Galvanostat Model 2053 AMEL, X-ray Diffractometer (XRD) SHIMADZU XD-610, Scanning Electron Microscope (SEM) (JEOL-JSM 6360 LA), and UV-Visible Spectrophotometer (USB Ocean Optics 2000).

Preparation of electrolyte solution

The electrolyte solution was prepared by dissolving 3 M lactic acid in 50 mL distilled water. Then, 4.9922 g (0.4 M) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 9.99925 g (5 M) NaOH were added simultaneously into the previous prepared lactic acid solution. In this mixing process, an exothermic chemical reaction occurs indicated by temperature increment of the mixture to 80°C . After 10 minutes, the temperature of the mixture slowly decreased to room temperature. The mixture was stirred using a magnetic stirrer on a hot-plate surface at a constant temperature of 60°C for 17 hours to mix all the precursors into a homogeneous solution. The resulting electrolyte solution color is blue-blackish and has a pH level of 9.

Preparation of working electrode

The Cu_2O thin films were deposited on indium-tin-oxide (ITO)-coated glass substrate by electrochemical deposition technique. The ITO-coated glass substrate was pre-cleaned with soap to remove impurities then rinsed with distilled water, continued by boiling onto a hot plate with a temperature of 80°C . The ITO-coated glass substrate was put into the ultrasonic cleaner for 15 minutes, cleaned with acetone, and rinsed again with distilled water.

Deposition of Cu_2O thin films

Electrodeposition of Cu_2O thin films was carried out using Potentiostat/Galvanostat Model 2053 AMEL. The electrochemical cell using three types of electrodes: a working electrode (WE) using indium-tin-oxide (ITO)-coated glass substrate for Cu_2O thin film support, a counter electrode (CE) using a platinum rod, and a reference electrode (RE) using standard calomel electrode (SCE), as shown in Figure 1. The working electrode is connected to a negative terminal on the potentiostat/galvanostat, while the counter electrode is a positive terminal. An electrolyte solution bath is placed on a hot-plate magnetic stirrer. The three electrodes were simultaneously immersed in a bath containing a precursor solution of 4.992 g (0.4 M) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 9.99925 g (5 M) NaOH as electrolytes.

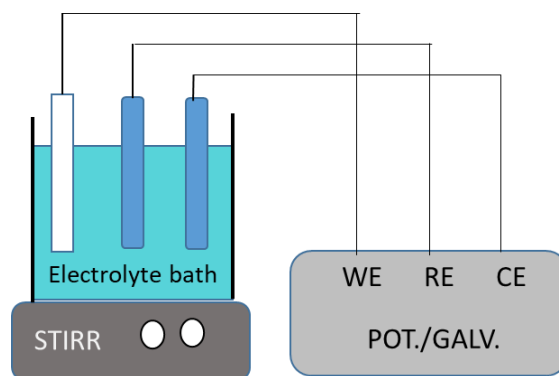


Figure 1. Scheme of equipment circuit for Cu_2O thin films electrodeposition

Two Cu_2O thin film samples were deposited on a glass substrate coated by ITO with different deposition potentials, at -0.5 V and -0.6 V , relative to the standard calomel electrode (SCE) as reference. The deposition of each Cu_2O thin film was carried out for 2.5 hours at an electrolyte solution temperature of 55°C while stirring at a rotational speed of 200 rpm using a magnetic stirrer.

Characterization of Cu_2O thin films

The crystalline properties of Cu_2O thin films were studied based on X-ray diffraction (XRD) analysis using a SHIMADZU XD-610 apparatus with a source wavelength of 1.5405 \AA . Cu_2O thin films were scanned at 2θ angle, from 20° – 80° . The data obtained is diffraction pattern consisting of characteristic diffraction peaks of Cu_2O phase. Lattice parameters were calculated using Cohen method [29] and crystallite size was determined using Scherrer method [13], [30].

To investigate the surface morphology of Cu_2O thin films, scanning electron microscope (SEM) was used (JEOL-JSM 6360 LA). This characterization was carried out to determine the surface morphological characteristics and thickness of Cu_2O thin films on a glass substrate coated with indium-tin-oxide (ITO). The film thickness will be used to determine the bandgap energy of Cu_2O thin films.

The optical properties of Cu_2O thin films were characterized using a UV-Visible Spectrophotometer (USB Ocean Optics 2000) in wavelength range 360–860 nm of electromagnetic spectrum. The optical transmittance of Cu_2O thin films is measured at each wavelength to determine the absorption coefficient and the bandgap energy value.

RESULTS AND DISCUSSION

Crystal properties

The X-ray diffraction pattern of Cu_2O thin films consisting of characteristic peaks of Cu_2O was used to determine the crystal phase, lattice parameters, and crystallite size. The X-ray diffraction patterns of two Cu_2O thin film samples in the 2θ ranging from 25° to 65° are shown in Figure 2, for the Cu_2O films deposited at -0.5 V and -0.6 V. The characteristic peaks of Cu_2O dominate the diffraction pattern in both Cu_2O thin films, as displayed by strong and sharp peaks. All of the diffraction peaks of Cu_2O films appeared in the diffraction pattern (diffractogram) for diffraction planes of (110), (111), (200), and (220). The diffraction peaks of Cu_2O film those deposited at -0.5 V were identified at 29.16° ; 36.02° ; 41.90° ; and 60.92° , with (110), (111), (200), and (220) planes, respectively. Meanwhile, Cu_2O films deposited at 0.6 V, the diffraction peaks were found at 29.24° ; 36.08° ; 41.96° , and 61.00° for (110), (111), (200), and (220) planes, respectively. Therefore, it can be seen that the Cu_2O film deposited at -0.5 V has larger diffraction angle than deposited at -0.6 V. The XRD results are in good agreement with the JCPDS card No. 05–0667 for cubic structure of Cu_2O phase [13], [14]. In addition, the Cu_2O films have been well growth prepared by electrochemical deposition, as indicated by Cu_2O peaks appeared in the diffraction pattern.

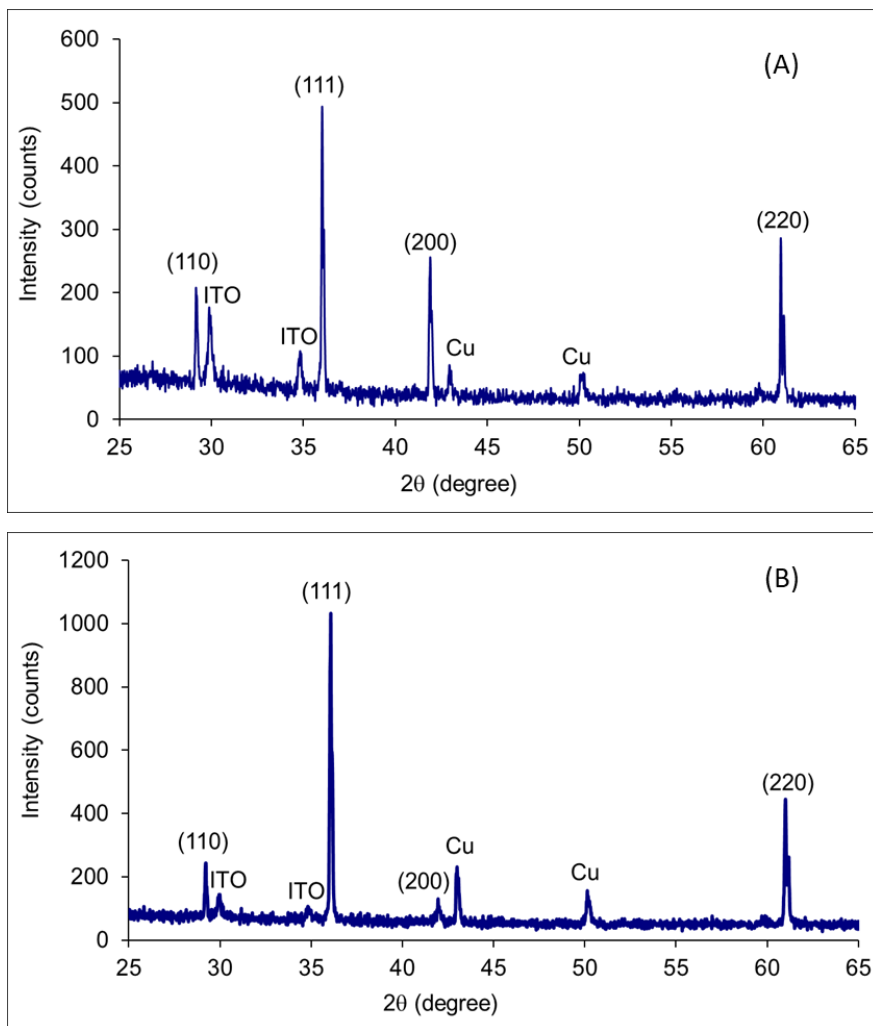


Figure 2. Diffraction pattern of Cu_2O thin films deposited at (a) -0.5 V and (b) -0.6 V

On the other hand, peaks of Cu (111) and Cu (200) metals also appeared, at 43.04° and 50.18° [26]. Those additional diffraction peaks belong to ITO, originating from the transparent conducting oxide (TCO) layer on the glass substrate [3]. The electrochemical growth of the Cu_2O thin film begins with Cu^{2+} ions from the acidic precursor solution, being driven onto the ITO glass substrate with a negative bias applied to the cathode then reduced to Cu^+ and reacts with OH^- to form a Cu_2O film on the ITO glass substrate as the working electrode at cathode [27]. Cu peaks also appeared in the diffraction pattern because not all Cu^+ ions successfully react with OH^- through reduction reaction thus Cu is formed on the substrate [13], [14], [28]. Cu^+ ions undergo a second reduction by electrons without involving OH^- and form Cu metal on the substrate [14].

The lattice parameters of the Cu_2O crystal were determined from each Cu_2O peak. It is known that Cu_2O crystal has a cubic structure, where $a = b = c$ and $\alpha = \beta = \gamma = 90^\circ$. The lattice parameters of Cu_2O were determined by Cohen method

[29] and the values are summarized in Table 1. There is no significant difference between the two Cu₂O films. The Cu₂O film deposited at -0.5 V has a lattice parameter of 4.31 Å, while the Cu₂O film deposited at -0.6 V is 4.30 Å. The lattice parameter values obtained are slightly larger than other work, 4.27 Å [3].

The crystallite size (D) of Cu₂O can be determined from the X-ray diffraction pattern based on the FWHM (full-width at half-maximum) values of each diffraction peak using the Scherrer formula [13], [30] as follows:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

where k is a constant of shape factor (0.9 for cubic structure) [32], λ is the wavelength of the X-ray source ($\lambda_{\text{CuK}\alpha} = 1.54$ Å), β is FWHM values (in rad) for each Cu₂O peaks, and θ is the angle of diffraction (in rad).

The calculation results of the average crystallite size (ACS) is summarized in Table 1. The Cu₂O film deposited at -0.5 V has an average crystallite size of 35.20 nm, while the Cu₂O film deposited at -0.6 V is 38.63 nm, which confirms the increase in the intensity of the diffraction peaks, the decrease of the FWHM [14]. Thus, the average crystallite size of Cu₂O increased significantly with increasing deposition potential, as found by Hssi *et al* [14], while the lattice parameters of Cu₂O films almost do not change with different deposition potential.

Table 1. Lattice parameters and crystallite size of Cu₂O films prepared by electrochemical deposition at different potential

Deposition potential (V)	Lattice parameter (a=b=c) (Å)	Crystallite size (nm)
-0.5	4.31	35.20
-0.6	4.30	38.63

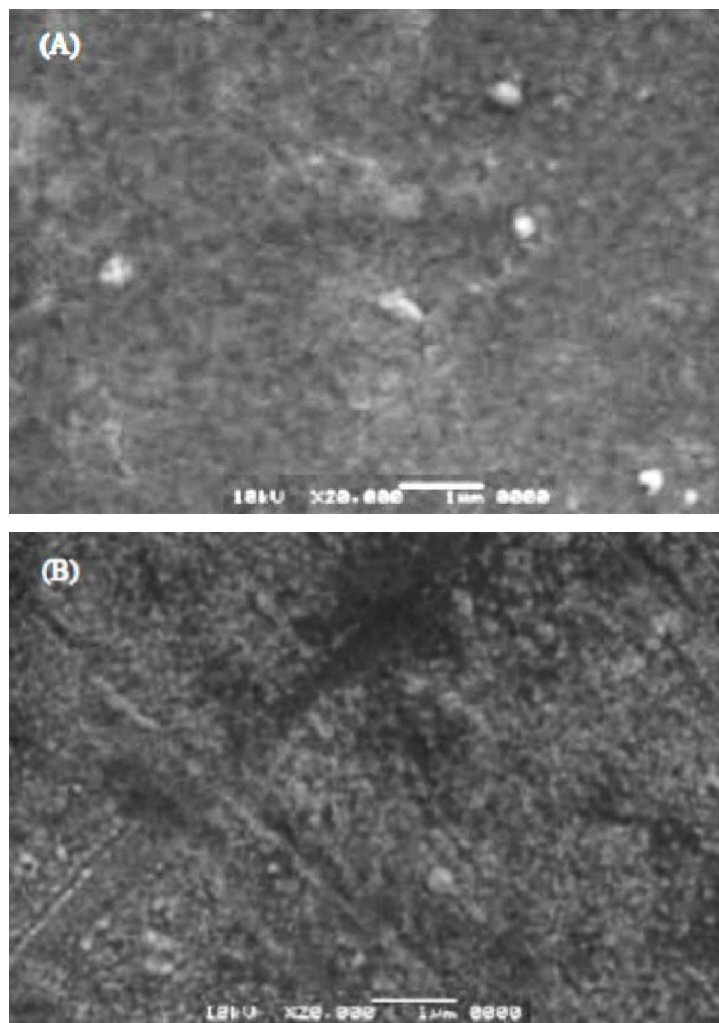


Figure 3. SEM images of surface morphology of Cu₂O thin films deposited at (a) -0.5 V and (b) -0.6 V

Surface morphology

The surface morphology of Cu₂O thin films on ITO-coated glass substrate was investigated using a scanning electron microscope (SEM) with a magnification of 20,000 times. Figure 3 shows SEM images of the two Cu₂O thin film samples. The SEM image shows that the surface morphology of the Cu₂O thin film samples is quite smooth and even. As the deposition potential increases, the Cu₂O film thickness increases, which is 0.567 μm for the film deposited at -0.5 V and 4 μm for the film deposited at -0.6 V. Laidoudi et al (2013) reported that the deposition voltage strongly influences the thickness of the Cu₂O film deposited by electrochemical methods [13]. They found that the Cu₂O film thickness increased with the increase in the deposition voltage, i.e, 80 nm for the film deposited at -0.4 V and 300 nm for the film positioned at -0.6 V [13]. The deposition conditions from Laidoudi et al are different from those carried out in this work, for example the deposition time is shorter (150 seconds) compared to 2.5 hours in this study and the concentration of the precursor solution is much lower, 0.05 M CuSO₄, while in this study 0.4 M CuSO₄ is used.

Optical properties and bandgap energy

Figure 4 shows the transmittance spectra of each Cu₂O film on an ITO-coated glass substrate. The transmittance spectra show that Cu₂O thin films transmit from visible light to infrared spectrum of the electromagnetic wave. The transmittance spectra of the two Cu₂O films are very different in the same range of spectra. The transmittance percentage of Cu₂O film deposited at -0.5 V was higher than -0.6V. The Cu₂O thin film deposited at -0.5 V is thinner than that deposited at -0.6V. The thickness of the film is very influential on the amount of light that is transmitted. The decreasing light transmittance with an increasing of thin film thickness can be attributed to an increasing of the effective optical path length with increasing film thickness. The film packing density increase when the film is thicker [32].

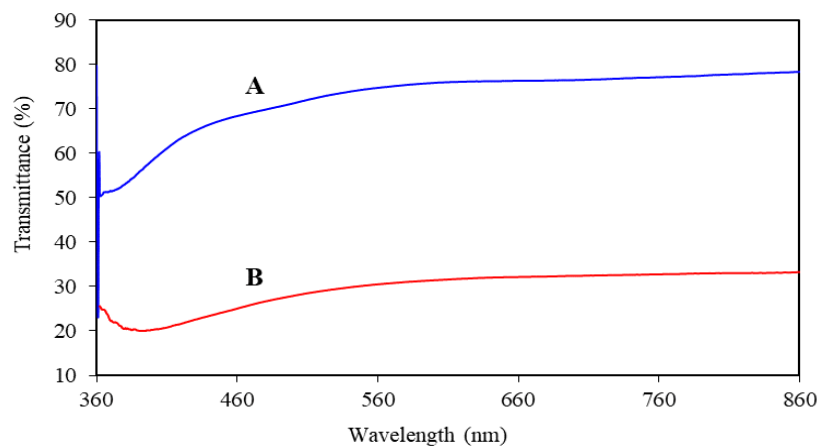


Figure 4. Transmittance of Cu₂O thin films deposited by electrochemical method at (a) -0.5 V and (b) -0.6 V

The transmittance data can be used to determine the value of the absorption coefficient (α) of Cu₂O films in each wavelength of incident light by using the logarithmic relationship between the absorption coefficient (α) and the transmittance of the films, which is written as follow [33]:

$$\alpha = \frac{1}{d} \ln(1/T) = \frac{1}{d} \ln(I_0/I) \quad (2)$$

where d is the thickness of the Cu₂O film, T is the transmittance of the Cu₂O film as a function of wavelength, I_0 is the intensity of the incident light, and I is the intensity of the light transmitted through the Cu₂O film. The absorption coefficient (α) is a function of wavelength of incident light.

To determine the bandgap energy of Cu₂O thin films, the Tauc plot was used by considering the type of electronic transition in Cu₂O material [13], [14], [34]. Tauc equation related to absorption coefficient of Cu₂O films that previously have calculated based on transmittance data. The electronic transition in semiconductor material Cu₂O is a direct transition ($n=1/2$), so the Tauc equation for Cu₂O can be written as follow [14], [33], [34]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (3)$$

where α is the absorption coefficient which is a function of wavelength, h is Planck's constant ($4,135 \times 10^{-15}$ eV.s), $h\nu$ is photon energy, E_g is bandgap energy, and A is a constant. A plot which is made of $e(\alpha h\nu)^2$ against the photon energy ($h\nu$) results a curve consisting of two parts, as shown in Figure 5, this curve is called a Tauc plot [13], [14], [33], [34]. The intersection of the linear line with the photon energy axis ($h\nu$) is the E_g value of the Cu₂O film.

The analysis results of the Tauc plot give different bandgap energies of the two Cu₂O films deposited at different potential. It is found that the bandgap energy of Cu₂O film increases as the deposition potential increases. The Cu₂O film deposited at -0.5V has a bandgap energy of 2.0 eV, while the Cu₂O film deposited at -0.6V is 2.13 eV. The band gap

energy increases due to the increase in atomic density in the film, resulting a decrease in the distance between atoms in the material. The film deposited with a higher potential has a higher atomic density thus the energy state density decreases.

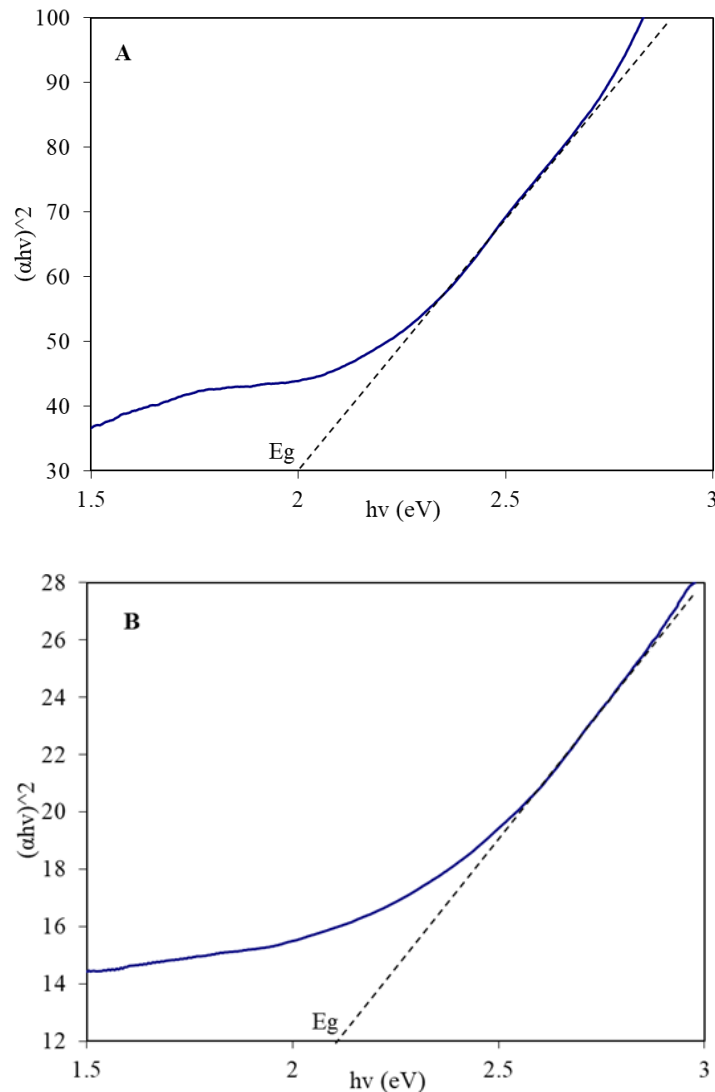


Figure 5. Tauc plot of Cu₂O thin films deposited at (a) -0.5 V and (b) -0.6 V

CONCLUSION

Electrochemical deposition or electrodeposition has is suitable for Cu₂O thin films fabrication. The results of the crystallographic study showed that a cubic structure of Cu₂O crystal phase had been formed, with sharp and strong characteristic of the X-ray diffraction peak of the Cu₂O phase. There are copper (Cu) diffraction peaks which fail to form into Cu₂O phase. Weak intensity peaks of ITO phase also appear in the diffraction pattern. The deposition potential significantly affects the crystal size and optical properties of Cu₂O films, meanwhile it does not significantly affect the lattice parameters. The increase in deposition potential significantly increased the average crystal size of Cu₂O, which is 35.20 nm for film deposited at -0.5 V potential and 38.63 nm for film deposited at -0.6V potential. The optical properties of Cu₂O films are significantly affected by the deposition potential as well, where the transmittance decreases with increasing deposition potential, which is caused by the thicker film thickness as the deposition potential increases. The band gap energy value of Cu₂O films is also significantly affected by the deposition potential. Cu₂O film deposited at a potential of -0.5V has a bandgap energy of 2.0 eV and film deposited at a potential of -0.6V is 2.13 eV. This bandgap energy values are within the range of Cu₂O bandgap energy values reported in other studies.

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