

## ORIGINAL ARTICLE

# Influence of Applied Potential on The Structural and Optical Properties of Cu<sub>2</sub>O Thin Films Grown by Electrochemical Deposition

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**ABSTRACT** – Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin films is studied. Electrodeposition for two Cu<sub>2</sub>O thin films was carried out at -0.5 V and -0.6 V, relative to the standard calomel electrode (SCE) as a reference electrode. Cu<sub>2</sub>O 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 Cu<sub>2</sub>O crystal has been well grown. The lattice parameters of Cu<sub>2</sub>O films slightly increased as the potential of electrodeposition increased. The crystallite size of Cu<sub>2</sub>O films significantly enhanced as the enhancing applied potential. Cu<sub>2</sub>O thin films showed a smooth and flat surface morphology based on SEM images. The optical transmittance of the Cu<sub>2</sub>O thin films drastically decreased as the applied potential increased due to the increasing film thickness. The bandgap energy of Cu<sub>2</sub>O 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 (Cu<sub>2</sub>O). Cu<sub>2</sub>O 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. Cu<sub>2</sub>O is oxide with cubic lattice (cuprite structure) in its crystal structure. The electrical and optical properties of Cu<sub>2</sub>O films are the focus of many studies due to their wide application potential. Cu<sub>2</sub>O is a p-type semiconductor that has bandgap energy ranging from 1.9 to 2.2 eV [1], [2]. The bandgap energy of the Cu<sub>2</sub>O 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, Cu<sub>2</sub>O 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 Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin films [12]-[14], [25]. Several studies related to the electrochemical deposition of Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin films [1].

Electrochemical deposition or electrodeposition is an easy, inexpensive, and effective way to grow Cu<sub>2</sub>O 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 Cu<sub>2</sub>O 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 Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin films grown by electrochemical deposition. Cu<sub>2</sub>O thin films were deposited on an indium-tin-oxide (ITO)-coated glass substrate. The structure, morphology, and optical properties of Cu<sub>2</sub>O 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),  $\text{NaOH}$  (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

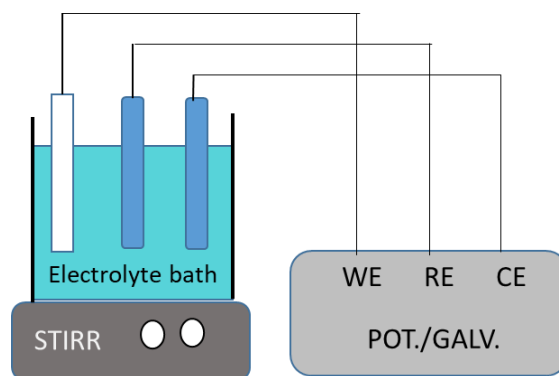
The electrolyte solution was prepared by dissolving 3 M lactic acid in 50 mL distilled water. Then, 4.9922 g (0.4 M)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and 9.99925 g (5 M)  $\text{NaOH}$  were added simultaneously into the previous prepared lactic acid solution. In this mixing process, an exothermic chemical reaction occurs indicated by temperature increment of the mixture to  $80^\circ\text{C}$ . 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  $60^\circ\text{C}$  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  $\text{Cu}_2\text{O}$  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  $80^\circ\text{C}$ . 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 $\text{Cu}_2\text{O}$ thin films

Electrodeposition of  $\text{Cu}_2\text{O}$  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  $\text{Cu}_2\text{O}$  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)  $\text{NaOH}$  as electrolytes.



**Figure 1.** Scheme of equipment circuit for  $\text{Cu}_2\text{O}$  thin films electrodeposition

Two  $\text{Cu}_2\text{O}$  thin film samples were deposited on a glass substrate coated by ITO with different deposition potentials, at  $-0.5\text{ V}$  and  $-0.6\text{ V}$ , relative to the standard calomel electrode (SCE) as reference. The deposition of each  $\text{Cu}_2\text{O}$  thin film was carried out for 2.5 hours at an electrolyte solution temperature of  $55^\circ\text{C}$  while stirring at a rotational speed of 200 rpm using a magnetic stirrer.

### Characterization of $\text{Cu}_2\text{O}$ thin films

The crystalline properties of  $\text{Cu}_2\text{O}$  thin films were studied based on X-ray diffraction (XRD) analysis using a SHIMADZU XD-610 apparatus with a source wavelength of  $1.5405\text{ \AA}$ .  $\text{Cu}_2\text{O}$  thin films were scanned at  $2\theta$  angle, from  $20^\circ$ – $80^\circ$ . The data obtained is diffraction pattern consisting of characteristic diffraction peaks of  $\text{Cu}_2\text{O}$  phase. Lattice parameters were calculated using Cohen method [29] and crystallite size was determined using Scherrer method [13], [30].

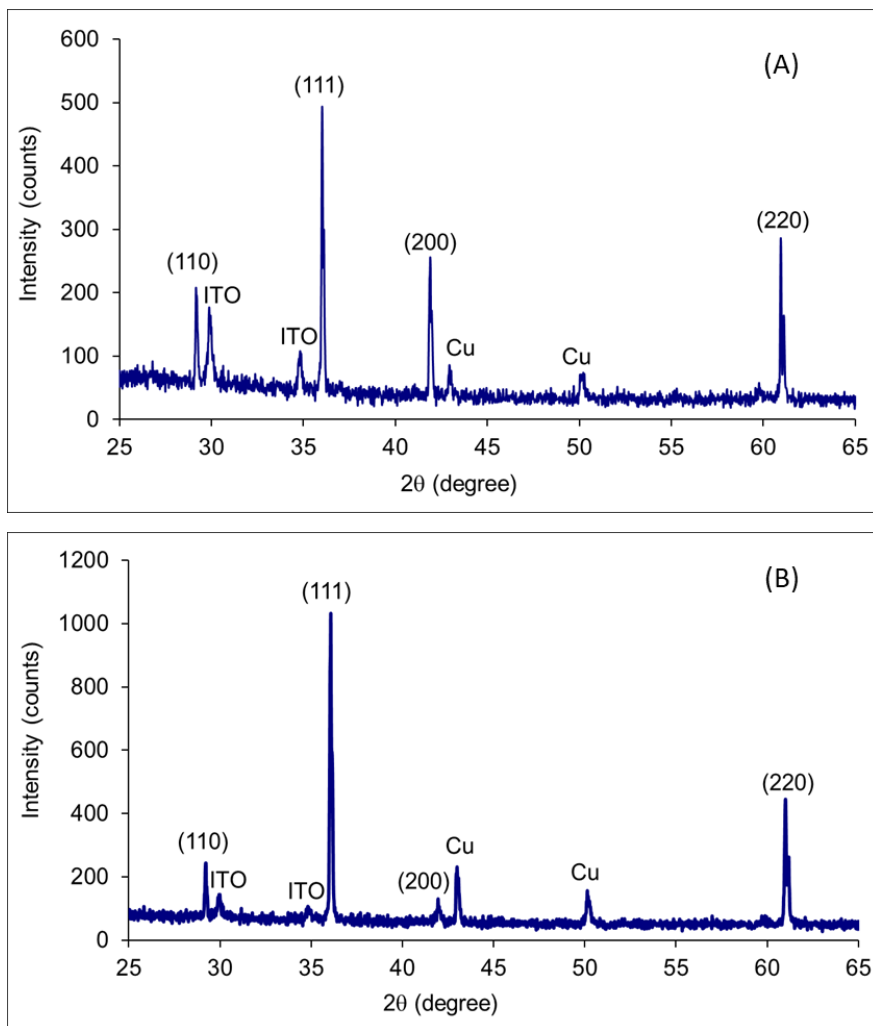
To investigate the surface morphology of  $\text{Cu}_2\text{O}$  thin films, scanning electron microscope (SEM) was used (JEOL-JSM 6360 LA). This characterization was carried out to determine the surface morphological characteristics and thickness of  $\text{Cu}_2\text{O}$  thin films on a glass substrate coated with indium-tin-oxide (ITO). The film thickness will be used to determine the bandgap energy of  $\text{Cu}_2\text{O}$  thin films.

The optical properties of  $\text{Cu}_2\text{O}$  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  $\text{Cu}_2\text{O}$  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  $\text{Cu}_2\text{O}$  thin films consisting of characteristic peaks of  $\text{Cu}_2\text{O}$  was used to determine the crystal phase, lattice parameters, and crystallite size. The X-ray diffraction patterns of two  $\text{Cu}_2\text{O}$  thin film samples in the  $2\theta$  ranging from  $25^\circ$  to  $65^\circ$  are shown in Figure 2, for the  $\text{Cu}_2\text{O}$  films deposited at  $-0.5$  V and  $-0.6$  V. The characteristic peaks of  $\text{Cu}_2\text{O}$  dominate the diffraction pattern in both  $\text{Cu}_2\text{O}$  thin films, as displayed by strong and sharp peaks. All of the diffraction peaks of  $\text{Cu}_2\text{O}$  films appeared in the diffraction pattern (diffractogram) for diffraction planes of (110), (111), (200), and (220). The diffraction peaks of  $\text{Cu}_2\text{O}$  film those deposited at  $-0.5$  V were identified at  $29.16^\circ$ ;  $36.02^\circ$ ;  $41.90^\circ$ ; and  $60.92^\circ$ , with (110), (111), (200), and (220) planes, respectively. Meanwhile,  $\text{Cu}_2\text{O}$  films deposited at  $0.6$  V, the diffraction peaks were found at  $29.24^\circ$ ;  $36.08^\circ$ ;  $41.96^\circ$ , and  $61.00^\circ$  for (110), (111), (200), and (220) planes, respectively. Therefore, it can be seen that the  $\text{Cu}_2\text{O}$  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  $\text{Cu}_2\text{O}$  phase [13], [14]. In addition, the  $\text{Cu}_2\text{O}$  films have been well growth prepared by electrochemical deposition, as indicated by  $\text{Cu}_2\text{O}$  peaks appeared in the diffraction pattern.



**Figure 2.** Diffraction pattern of  $\text{Cu}_2\text{O}$  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  $43.04^\circ$  and  $50.18^\circ$  [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  $\text{Cu}_2\text{O}$  thin film begins with  $\text{Cu}^{2+}$  ions from the acidic precursor solution, being driven onto the ITO glass substrate with a negative bias applied to the cathode then reduced to  $\text{Cu}^+$  and reacts with  $\text{OH}^-$  to form a  $\text{Cu}_2\text{O}$  film on the ITO glass substrate as the working electrode at cathode [27]. Cu peaks also appeared in the diffraction pattern because not all  $\text{Cu}^+$  ions successfully react with  $\text{OH}^-$  through reduction reaction thus Cu is formed on the substrate [13], [14], [28].  $\text{Cu}^+$  ions undergo a second reduction by electrons without involving  $\text{OH}^-$  and form Cu metal on the substrate [14].

The lattice parameters of the  $\text{Cu}_2\text{O}$  crystal were determined from each  $\text{Cu}_2\text{O}$  peak. It is known that  $\text{Cu}_2\text{O}$  crystal has a cubic structure, where  $a = b = c$  and  $\alpha = \beta = \gamma = 90^\circ$ . The lattice parameters of  $\text{Cu}_2\text{O}$  were determined by Cohen method

[29] and the values are summarized in Table 1. There is no significant difference between the two Cu<sub>2</sub>O films. The Cu<sub>2</sub>O film deposited at -0.5 V has a lattice parameter of 4.31 Å, while the Cu<sub>2</sub>O 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 Cu<sub>2</sub>O 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:

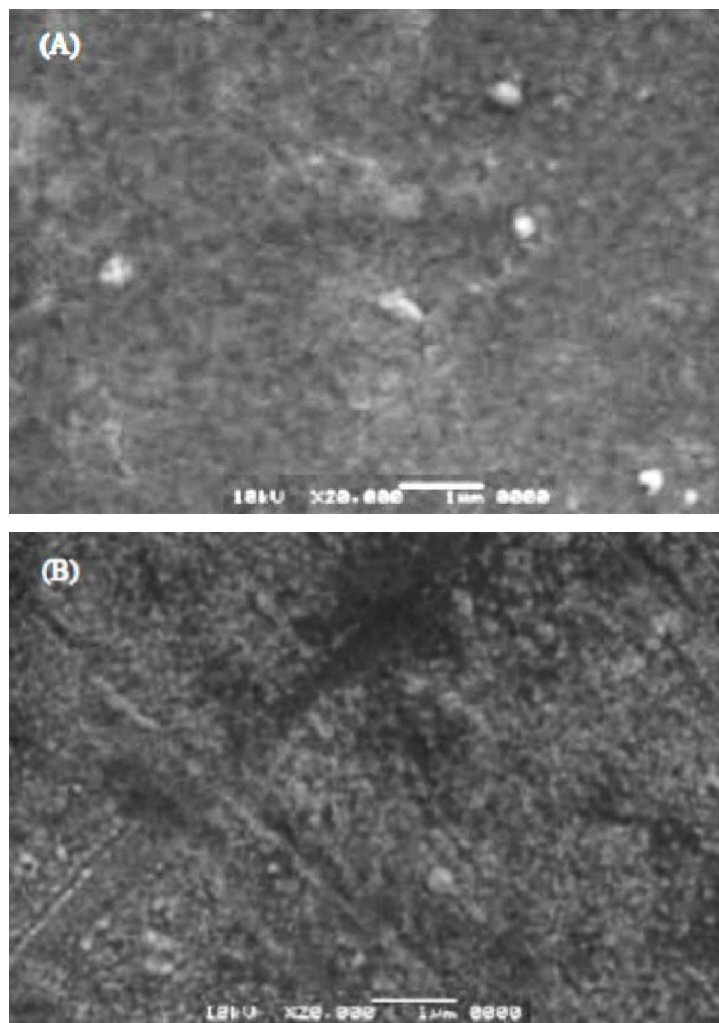
$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

where k is a constant of shape factor (0.9 for cubic structure) [32],  $\lambda$  is the wavelength of the X-ray source ( $\lambda_{\text{CuK}\alpha} = 1.54$  Å),  $\beta$  is FWHM values (in rad) for each Cu<sub>2</sub>O peaks, and  $\theta$  is the angle of diffraction (in rad).

The calculation results of the average crystallite size (ACS) is summarized in Table 1. The Cu<sub>2</sub>O film deposited at -0.5 V has an average crystallite size of 35.20 nm, while the Cu<sub>2</sub>O 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 Cu<sub>2</sub>O increased significantly with increasing deposition potential, as found by Hssi *et al* [14], while the lattice parameters of Cu<sub>2</sub>O films almost do not change with different deposition potential.

**Table 1.** Lattice parameters and crystallite size of Cu<sub>2</sub>O 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



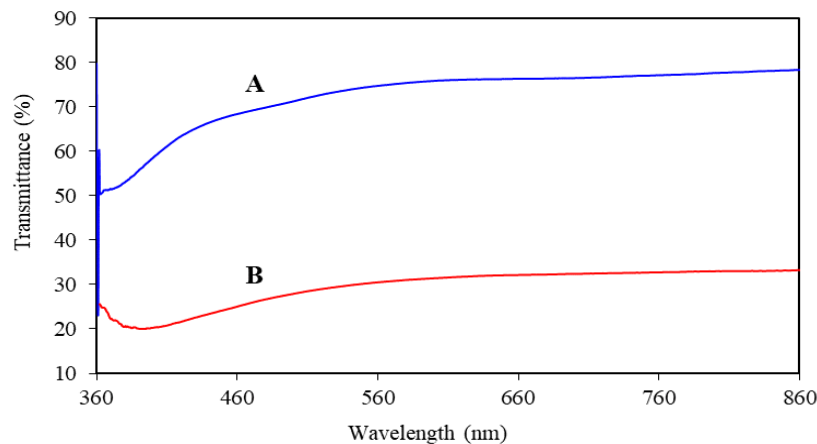
**Figure 3.** SEM images of surface morphology of Cu<sub>2</sub>O thin films deposited at (a) -0.5 V and (b) -0.6 V

### Surface morphology

The surface morphology of Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin film samples. The SEM image shows that the surface morphology of the Cu<sub>2</sub>O thin film samples is quite smooth and even. As the deposition potential increases, the Cu<sub>2</sub>O 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 Cu<sub>2</sub>O film deposited by electrochemical methods [13]. They found that the Cu<sub>2</sub>O 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 CuSO<sub>4</sub>, while in this study 0.4 M CuSO<sub>4</sub> is used.

### Optical properties and bandgap energy

Figure 4 shows the transmittance spectra of each Cu<sub>2</sub>O film on an ITO-coated glass substrate. The transmittance spectra show that Cu<sub>2</sub>O thin films transmit from visible light to infrared spectrum of the electromagnetic wave. The transmittance spectra of the two Cu<sub>2</sub>O films are very different in the same range of spectra. The transmittance percentage of Cu<sub>2</sub>O film deposited at -0.5 V was higher than -0.6V. The Cu<sub>2</sub>O 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 Cu<sub>2</sub>O 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 ( $\alpha$ ) of Cu<sub>2</sub>O films in each wavelength of incident light by using the logarithmic relationship between the absorption coefficient ( $\alpha$ ) 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 Cu<sub>2</sub>O film,  $T$  is the transmittance of the Cu<sub>2</sub>O 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 Cu<sub>2</sub>O film. The absorption coefficient ( $\alpha$ ) is a function of wavelength of incident light.

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

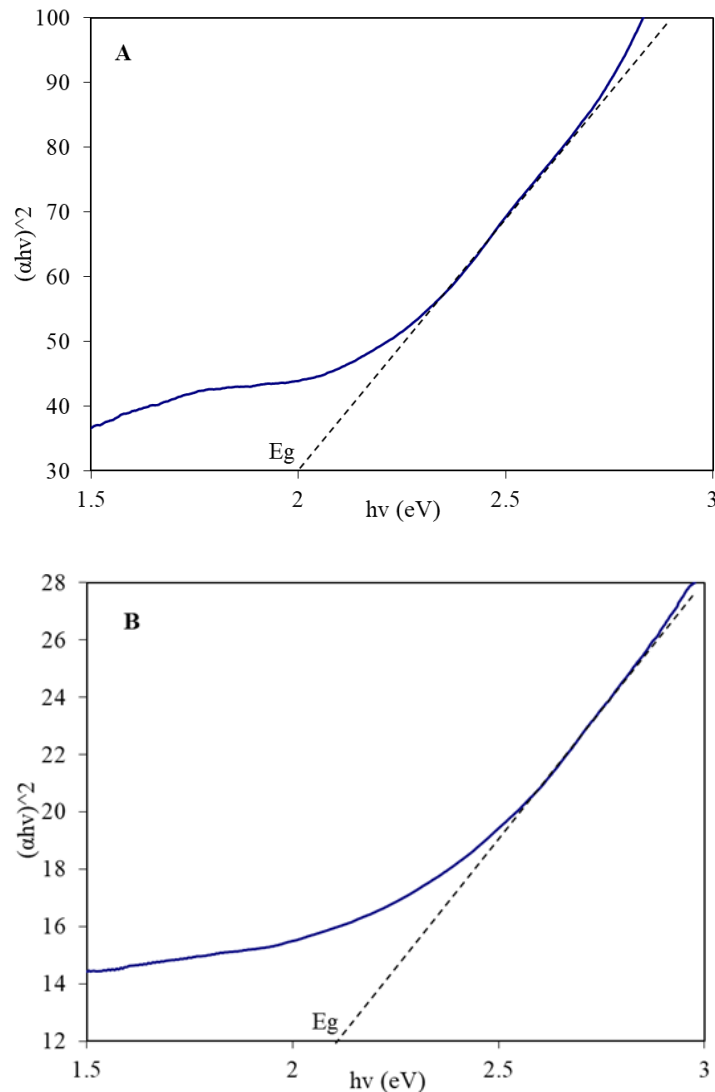
$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (3)$$

where  $\alpha$  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  $(\alpha h\nu)^2$  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 Cu<sub>2</sub>O film.

The analysis results of the Tauc plot give different bandgap energies of the two Cu<sub>2</sub>O films deposited at different potential. It is found that the bandgap energy of Cu<sub>2</sub>O film increases as the deposition potential increases. The Cu<sub>2</sub>O film deposited at -0.5V has a bandgap energy of 2.0 eV, while the Cu<sub>2</sub>O 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 Cu<sub>2</sub>O thin films deposited at (a) -0.5 V and (b) -0.6 V

## CONCLUSION

Electrochemical deposition or electrodeposition has is suitable for Cu<sub>2</sub>O thin films fabrication. The results of the crystallographic study showed that a cubic structure of Cu<sub>2</sub>O crystal phase had been formed, with sharp and strong characteristic of the X-ray diffraction peak of the Cu<sub>2</sub>O phase. There are copper (Cu) diffraction peaks which fail to form into Cu<sub>2</sub>O 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 Cu<sub>2</sub>O films, meanwhile it does not significantly affect the lattice parameters. The increase in deposition potential significantly increased the average crystal size of Cu<sub>2</sub>O, 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 Cu<sub>2</sub>O 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 Cu<sub>2</sub>O films is also significantly affected by the deposition potential. Cu<sub>2</sub>O 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 Cu<sub>2</sub>O bandgap energy values reported in other studies.

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