Fabrication of Homojunction Cuprous Oxide Solar Cell by Electrodeposition Method

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Abstract: Thin films of p-Cu_2O and n-Cu_2O were controllably prepared using electrochemical deposition method by using different conditions for each type with different thickness. Photocurrent measurement show that the Cu_2O thin films synthesized in acid and alkaline media present n-Cu_2O and p-Cu_2O semiconductor character, respectively. The morphology and orientation have been investigated. Thin film of p-Cu_2O show pyramid shape with orientation (111) and n-Cu_2O show dendritic shape with orientations (111) and (200). Optical characteristics of p-Cu_2O and n-Cu_2O were slightly affected. Homojunction Cu_2O solar cells were successfully prepared by consecutively depositing an n-Cu_2O layer on a p-Cu_2O layer. Then, evaporate gold on n- Cu_2O by thermal evaporator. The best performance of the cell fabricated in this study was short-circuit current density 12.7mA/cm^2, open-circuit voltage 31.5mV, fill factor 35%, and conversion efficiency 0.14% under standard test condition for thickness 2µm and 0.7µm for n-Cu_2O and p-Cu_2O, respectively. High short-circuit current density due to the pyramid surface structure of p- Cu_2O caused the increase of the light path in the solar cell that could facilitate the photons to absorb more effectively. Low open-circuit voltage was due to energy band bending influence by interface states. The main limiting factor for the Cu_2O cell efficiency was the high resistivity of both n-Cu_2O and p-Cu_2O. Doping of Cu_2O will be necessary to improve the efficiency of the homojunction Cu_2O solar cells further.

1. Introduction

Cuprous oxide (Cu_2O) is a very promising semiconductor for the fabrication of solar cells due to the theoretical efficiency approximately 20% [1], the direct band gap semiconductor $\cong$ 2 eV, abundant, non-toxic, prepared with simple methods at very low cost and relatively high absorption coefficient over the wavelength range of the solar spectrum [2,3]. Cu_2O can be prepared by many methods, including thermal oxidation, anodic oxidation, sputtering, and electrochemical deposition [4]. Different kinds of Cu_2O solar cells have been fabricated [5,6]. The efficiencies of the prepared solar cells were usually about 1%. Fabrication of heterojunction Cu_2O/ZnO solar cells made by deposition of transparent conducting oxide films on Cu_2O substrates reported by Mittiga et al.[7], and the best conversion efficiency of solar cell is 2%. This is the highest efficiency obtained with a p-n heterojunction, which is still much lower than the theoretical efficiency 20% of the Cu_2O solar cell. So, it may be the best way to improve the efficiency is fabricate a p-n homojunction of cuprous oxide due to the reduction of strain due lattice matching at the interface. But, due to the intrinsic p-type conductivity of Cu_2O, creating a p-n homojunction Cu_2O has been considered challenging [8]. There have only been a few reports of p- n homojunction Cu_2O to date due to the previous lack of synthesis methods to make n-Cu_2O.

Han et al. [9] fabricated Cu_2O homojunction Cu_2O solar cells by electrodeposition and optimized the resistivity of film with a result of a conversion efficiency of 0.1%. Recently, the effect of morphology of junction on the performance of Cu_2O solar cells was been studied [10]. More recently, Wei H.M. et al fabricate series of homojunction Cu_2O solar cells and found the best performance parameters are short-circuit current density 3.97mA/cm^2, open-circuit voltage 120mV, fill factor 22.6%, and conversion efficiency 0.104% under standard test condition [11].

In this study, a preparation and investigation of p-Cu_2O and n-Cu_2O by electrochemical deposition method have been done, in order to better understand p-n homojunction solar cell. Then fabricate homojunction Cu_2O solar cells with different thickness and characterize the performance of cells using solar simulator.

2. Experimental
(1) Cyclic voltammetry

Before any deposition, cyclic voltammetry is performed in the same cell as for deposition to figure out parameters needed for the deposition of cuprous oxide.

In Figure 1, a typical IV is shown with the corresponding conditions for p-type and n-type cuprous oxide thin film.
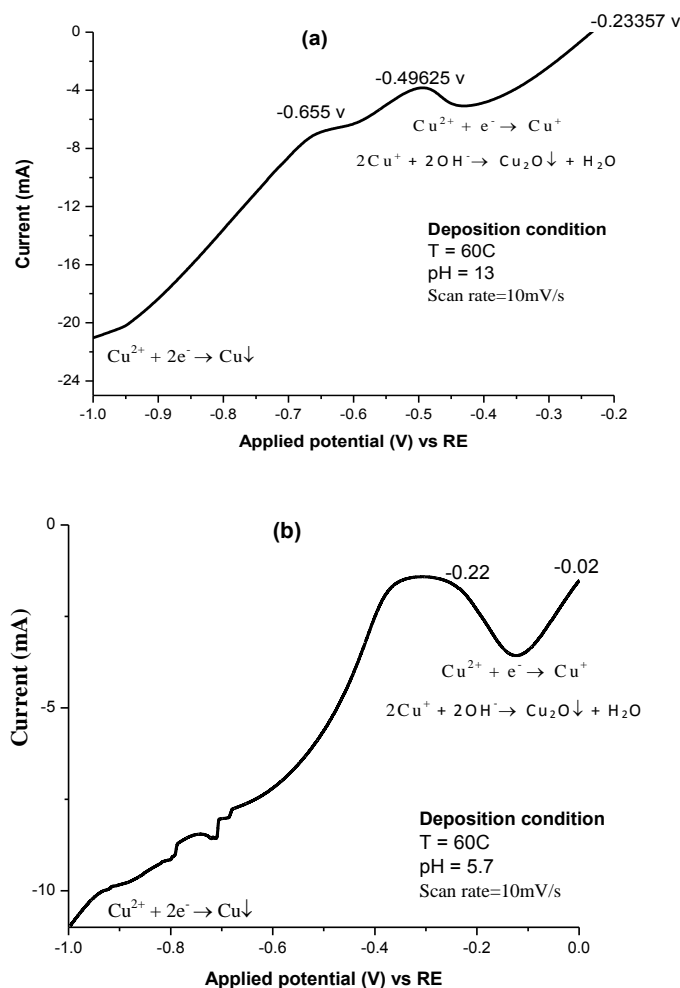
Figure 1: IV curves for solution containing
(a) 0.4M $CuSO_4$ and 3M lactic acid
(b) 0.02M copper acetate and 0.1M sodium acetate

According to the IV curve Figure (a), two reduction reactions were observed due to the presence of cupric ions in the electrolyte. The first reduction that took place between -0.23 V and -0.496 V (vs. Ag/AgCl/saturated NaCl) contributes to the formation of Cu_2O by the following reaction.

$$Cu^{2+} + e^- \rightarrow Cu^+$$
$$\text{(1)}$$
$$2Cu^+ + 2OH^- \rightarrow Cu_2O\downarrow + H_2O \quad \text{(2)}$$
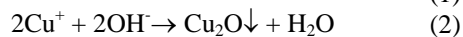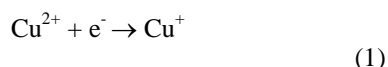
The second reduction that occurred after ~ -0.65 V contributes to the formation of Cu by the following reaction.

$$Cu^{2+} + 2e^- \rightarrow Cu\downarrow \quad \text{(3)}$$
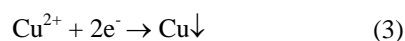
Therefore, the samples of p-Cu_2O were deposited at the different voltages between -0.23 V and -0.496 V. A constant potential of -0.4V was used for deposition of p-type Cu_2O for a reasonable deposition rate to avoid deposition of metallic copper.

According to the IV curve Figure (b), two reduction reactions were observed due to the presence of cupric ions in the electrolyte. The first reduction that took place between -0.02 V and -0.22 V (vs. Ag/AgCl/saturated NaCl) contributes to the formation of Cu_2O by Equations (1, 2).The second reduction that occurred after ~ -0.22 V contributes to the formation of Cu by Equation (3).

The corresponding current is much lower than that of p-type cuprous oxide deposition, which results in a much lower deposition rate of n-type cuprous oxide. The potential window for the Cu_2O deposition in acetate bath is very narrow, so a constant potential of -0.15 V was used for deposition of n-type Cu_2O for a reasonable deposition rate to avoid deposition of metallic copper.

(2) Photocurrent measurements
Photocurrent measurements were carried out using a home-built system, which includes a light source, an illumination switch, three-electrode cell and a controlling system. A 150W tungsten lamp is used to provide broad spectrum light. The illumination switch is actually a manually controlled light chopper with certain time intervals (5 sec), while the potentiostat is used to control the applied potential and scan rate.

Photocurrent measurements were performed on electrodeposited samples at 0V (vs. Ag/AgCl) to verify their conduction type. Figure 2 represents zero bias photocurrent measurements of Cu_2O samples. These results revealed anodic photocurrent for sample deposited at bath pH of 5.7, indicating n-type conductivity. For Cu_2O sample deposited at bath pH 13 we found that they produce cathodic current which mean p-type conductivity. Since, electrodeposition of Cu_2O involves two steps: The first step of the reaction would be the formation of Cu^+ ions at the electrode surface due to the cathodic reaction and the second step will be the formation of Cu_2O by reacting Cu^+ with OH^- according to Equations (1, 2).

It noted that the chemical reaction is strongly dependent on the concentration of [OH^-], in other word, bath pH. At low pH values the rate of reaction is low and presence of excess Cu^+ ions favors the formation of Cu rich or O deficient (i.e. having O vacancies) Cu_2O thin films. Thus high density of donors can be expected and therefore the film becomes n- Cu_2O. This good agreement with the result shown in Figure 2(b). Where at low pH values films grown are n- Cu_2O. However, when the electrolyte pH is higher, rate of reaction will be higher and insufficient Cu^+ ion concentration might yield Cu_2O

thin films with Cu deficiency. In other word, acceptor density will be higher and the thin film will be p-Cu_2O. This agrees with the result shown in Figure 2(a). From these results, it found that bath pH has massive role in controlling the type of conductivity for the prepared Cu_2O thin films.
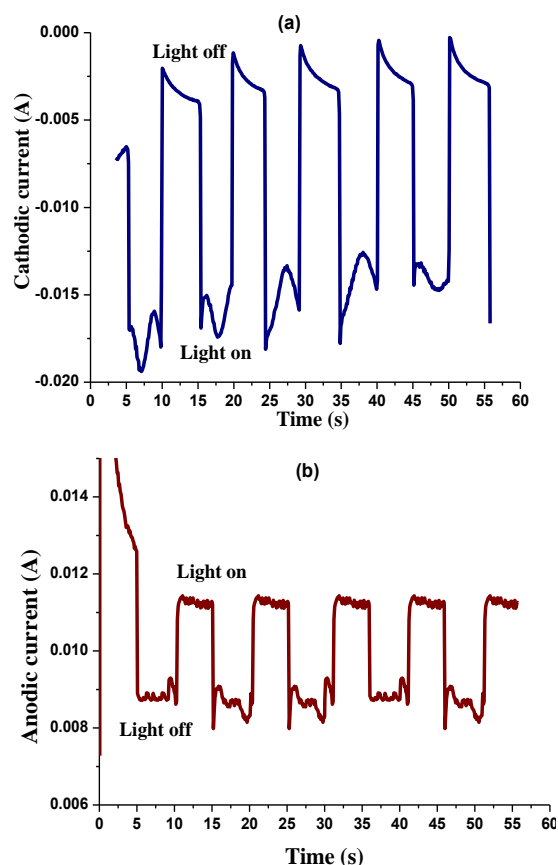

Figure 2: Photocurrent of (a) p-Cu_2O (b) n-Cu_2O.

(3) Solar cell fabrication

Cu_2O thin films were electrodeposited using a three-electrode setup consisting of platinum wire as a counter electrode, Ag/AgCl as a reference electrode, while FTO-coated glass substrate was employed as a working electrode. The size of the working electrode was 1.6x2.5cm^2. Prior to the film deposition, substrate was cleaned by successive sonication in acetone, isopropanol and finally DI water for 10 minutes, respectively and then dried in oven at 105°C for at least 30 min.

To make a p-n homojunction, p-type and n-type Cu_2O films were sequentially deposited in two different electrolyte solutions. A p-type Cu_2O Film was first electrochemically deposited on a FTO-coated glass substrate using electrolyte consists of 0.4M copper sulfate anhydrous ($CuSO_4$, 99.5%, BDH) and 3M lactic acid ($C_3H_6O_3$, 90%, Appli Chem). Bath pH

value of the electrolyte were adjusted carefully by addition of 4M Sodium hydroxide (NaOH, 99%, Oxford) to the deposition electrolyte to be 13 {varying pH from 9 to 13 and optimized pH 13 from SEM where sample at pH 13 have maximum grain size [12] which decrease recombination in the cell and have maximum cathodic current}.

The temperature of the electrolyte was maintained at 60 °C. Electrodeposition was carried out under a potentiostatic condition of -0.4V vs. Ag/AgCl using Bio-LogicSb-50 potentiostat. After deposition the film was good cleaned by distilled water and dried with air flow.

A n-type Cu_2O film was directly deposited on a p-type Cu_2O using electrolyte consist of 0.1M sodium acetate anhydrous ($C_4H_6NaO_4$, ADWIC) and 0.02M cupric acetate monohydrate ($C_4H_6CuO_4 \cdot H_2O$). Bath pH value of the electrolyte were adjusted carefully by addition of 0.08M acetic acid glacial ($C_2H_4O_2$, 99%, El Salam for Chemical Industries) to the deposition electrolyte to be 5.7{ varying pH from 5.2 to 6.4 and optimized 5.7 from photocurrent where samples prepared after pH 5.7 transfer to p-Cu_2O and before pH 5.2 formed copper metal [13]}.

The temperature of the electrolyte was maintained at 60 °C. Electrodeposition was carried out under a potentiostatic condition of -0.15V vs. Ag/AgCl using Bio-LogicSb-50 potentiostat. After deposition the film was good cleaned by distilled water and dried with air flow. The size of the deposited Cu_2O layer was 1.6x1.9 cm^2. After deposition, gold (Au) top contact was deposited on the n-Cu_2O using thermal evaporator operated at 10^{-5}Torr and 80A DC with an Au target (Purity 99.99%). The device fabrication was finished by evaporating of Au for top contact.

The finished device was 1×1 mm^2 in size. The performance of the solar cell was investigated with a solar simulator calibrated at standard test condition. The I-V curves were obtained with a Keithley 2400 SourceMeter.

3. Results and discussion
(1) p-Cu_2O and n-Cu_2O properties

The morphologies of the prepared Cu_2O samples by the electrodeposition processing under different optimum conditions are shown in Figure 3(a, b). The SEM images show that the p-Cu_2O samples formed in a three-faced pyramid shape as illustrated in Figure 3(a) which is similar to the textured surface structure of crystalline silicon solar cells [14, 15]. Whereas a dendritic shape was observed and shown in Figure 3 (b) for n-Cu_2O. A dendritic shape [16] of n-Cu_2O appear due to the lateral crystal growth is faster than vertical growth. It was shown that the branches grown on the substrate are significantly longer than the

branches grown out of the substrate, which indicates faster lateral growth than vertical growth. This was mainly because the FTO substrate is much more conductive than the nucleated semiconductor Cu_2O, resulting in growth on the conductive substrate faster than the growth out of the substrate. The shape formation of the Cu_2O deposition was attributed to the crystal growth with the presence of $[H^+]$ or $[OH^-]$ under different pH values, which affect the electrochemical reaction [17].
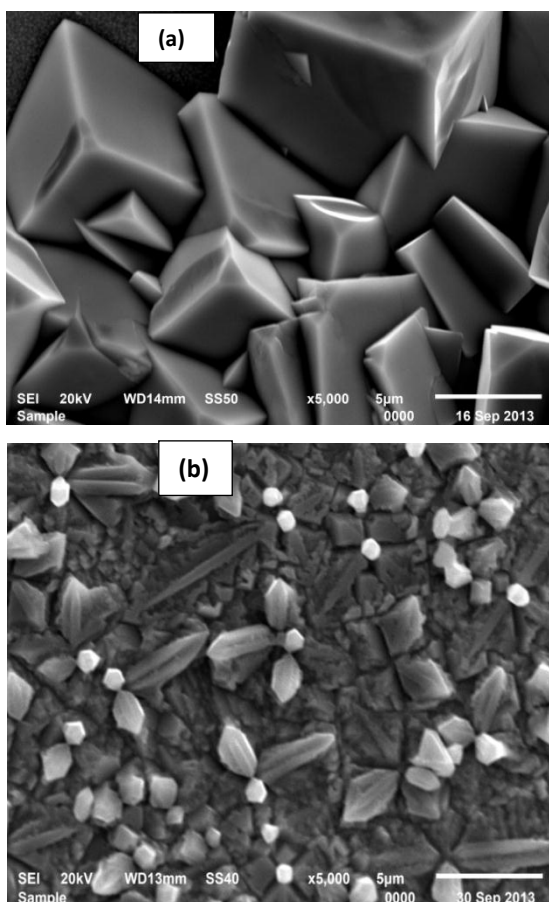

Figure 3: SEM of (a) p-Cu_2O (b) n-Cu_2O.

The Cu_2O crystal structure was characterized using XRD measurements for the prepared films as shown in Figure 4 (a, b), the diffraction peak with asterisks from the FTO conductive film on glass. These measurements indicated that electrodeposited films are polycrystalline and the crystallographic phase of the films is cubic as it is clear from the well-defined peaks. Also Cu_2O films deposited at the selected optimum conditions for p-type and n-type are pure cuprous oxide and there is no metallic copper. For n-Cu_2O, spectral peaks at 36.45° (111) and 42.33° (200) are detected from the XRD tests; whereas a prominent peak is demonstrated at 36.45° (111) for p-

Cu_2O. This indicates that the crystal growth of p-Cu_2O and n-Cu_2O deposition orientation was significantly influenced by pH value [18].
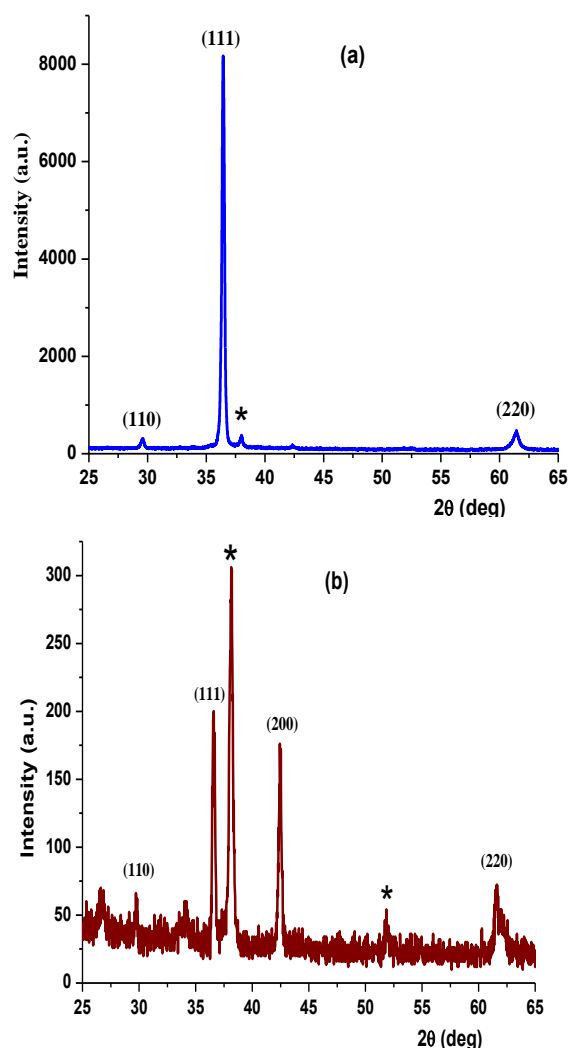

Figure 4: XRD patterns of (a) p-Cu_2O (b) n-Cu_2O (* refers to FTO substrate)

From these results, it possible to conclude that pH controls the orientation and grain size of Cu_2O films by affecting the growth rate of different crystallographic planes with different O density. For more understanding, in Cu_2O deposition, Cu^+ comes from the reduction of Cu^{2+} and O^{2-} comes from OH^- in the solution. Because $[Cu^{2+}]$ is kept constant throughout the deposition, $[OH^-]$, i.e., the pH will determine the growth rate of different crystallographic faces and thus control the orientation and grain size.

Optical characterization of Cu_2O thin film is performed with a Jasco V630 UV-Vis spectrophotometer. For p-Cu_2O, Significant increase of absorption for Cu_2O films is observed at

wavelengths shorter than 640 nm corresponding to a band gap of 2.0 eV [12] , which is close to the values in the previous report [19]. It was shown that incident light above 570 nm [13] can reach throughout the whole p-Cu_2O film, while the transmittance of incident light below 570 nm is close to zero. This demonstrates that the absorption coefficient of p-Cu_2O is extremely large at wavelength below 570 nm [12], which corresponds to a small penetration depth [20]. For n-Cu_2O, the transmittance of incident light below 475 nm is close to zero. This demonstrates that the absorption coefficient of n-Cu_2O is extremely large at wavelength below 475 nm [13].

To analyze the spectrum, the Tauc equation [21] is used:
$$(\alpha h v)^2 = A(hv - E_g) \qquad (4)$$
Where α is the absorption coefficient, hv represents the photon energy, E_g is the band-gap and A is a constant. n depends on the nature of the band-gap (n = 1/3 for indirect forbidden transition, n =1/2 for indirect allowed transition, n = 2/3 for direct forbidden transition, and n = 2 for direct allowed transition)., which indicates that electrodeposited Cu_2O has a direct band-gap. The intercept of the plot with the hv axis shows that Cu_2O has a band-gap of 2.08 eV [12] and 2.055eV [13] for p-Cu_2O and n-Cu_2O respectively, which is consistent with the reported band-gap values for Cu_2O. The band gap energy shows that no effect on the optical properties of p-Cu_2O and n-Cu_2O thin films.

(2) Device characterization
The prepared Cu_2O p-n homo junction solar cell is shown in Figure 5.
There are three junctions formed in this structure:
(1) hetero-junction between the FTO and p-Cu_2O
(2) p-n homojunction between the p-Cu_2O and the n-Cu_2O.
(3) Schottky junction between the n-Cu_2O and the Au.

Before J-V characterization of the solar cells, the behavior of junction 1, 3 were investigated in advance to confirm the formation of a Cu_2O p-n junction. In order to check the junction 1, a different structure with Au/ p-Cu_2O/ Au structure, shown in Figure 6(a), were prepared. Square Au electrode was thermally deposited on the top surface of p-Cu_2O. J-V measurements were performed between two Au electrodes. As shown in Figure 6(b), the interface between Au and p- Cu_2O shows nice ohmic behavior.
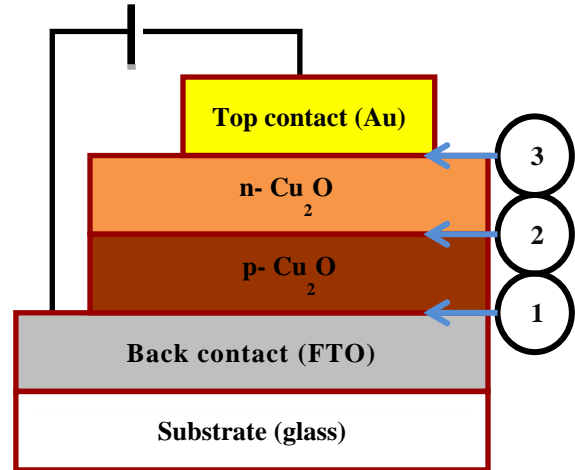

Figure 5: Structure of electro-deposited p-n homojunction Cu_2O solar cell

In order to examine the interface between n-Cu_2O and FTO, another structure with an Au/ n-Cu_2O/ Au structure, shown in Figure 7(a) were prepared. As shown in Figure 7(b), the interface between n-Cu_2O and FTO also shows ohmic contact. Therefore, any rectification in J-V characteristics from the structure in Figure 7 should come from the p-n homojunction Cu_2O.
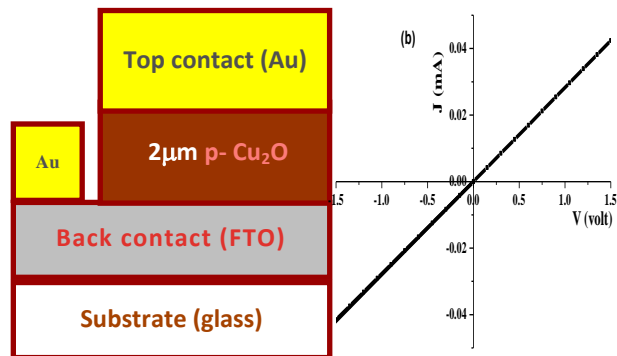

Figure 6: (a) Structure for the analysis of junction between the FTO and the p-Cu_2O (b) J-V characteristic between the FTO and the p-Cu_2O
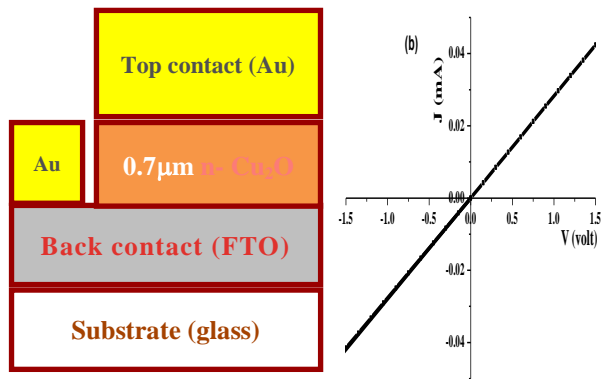
Figure 7: (a) Structure for the analysis of junction between the n-Cu$_2$O and the Au (b) J-V characteristic between the n-Cu$_2$O and the Au

Dark J-V characteristic of Cu$_2$O p-n homojunction solar cell is shown in Figure 8(a). This shows a typical rectification effect of a p-n junction. It represents the successful interaction of n-Cu$_2$O grown on a p-Cu$_2$O film during the elctrodeposition processing was revealed to interpret the formation of the p−n homojunction device. The photovoltaic performance parameters of a p-n homojunction Cu$_2$O solar cells with different thickness under standard test condition are shown in Table 1. When deposition time increase lead to thickness increase lead to efficiency decrease due to recombination increase. The best performance of the cell fabricated in this study was short-circuit current density 12.7mA/cm^2, open-circuit voltage 31.5mV, fill factor 35%, and conversion efficiency 0.14% is shown in Figure 8(b) under illumination intensity 1 Sun and temperature 25°C.
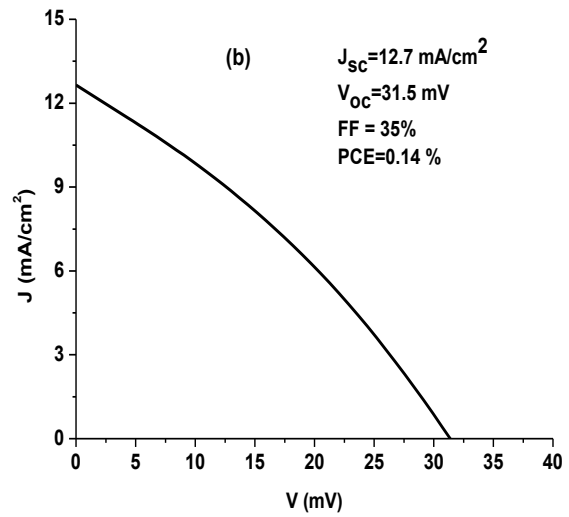
Fugure 8: (a) Dark J-V characteristic of Cu$_2$O p-n homojunction solar cell (b) Photo J-V characteristic of Cu$_2$O p-n homojunction solar cell

From solar cell parameters, it noted that there are high short circuit current density and low open circuit voltage. These may be explaining from influence of microstructure surface [22]. The influence of the microstructure surface of p-Cu$_2$O film on the solar cell properties is mainly due to pathway of light propagation in the Cu$_2$O solar cells. Both refraction and reflection can happen there as shown in Figure 9(a). This means that the pyramid shape structure of p-Cu$_2$O caused the increase the path of light in the cell that could allowed the photons to absorb more effectively. Therefore, solar cell has high short circuit current density.
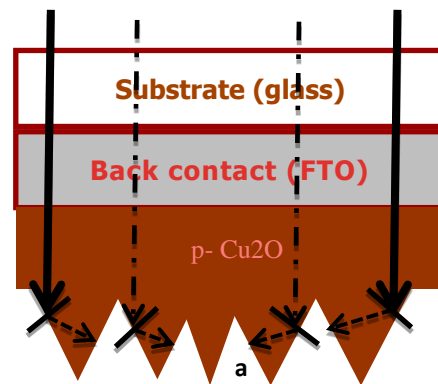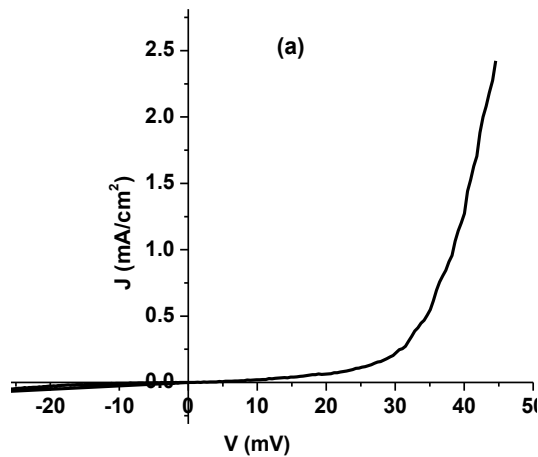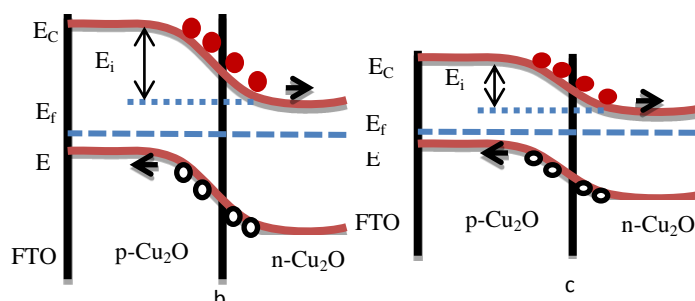
Figure 9: (a) Pathway of light propagation through homojunction Cu_2O solar cell
(b) Diagram of proposed energy band for the p-n homojunction Cu_2O solar cell.
(c) Energy band bending diagram influenced by interface states

Table.1: Effect of deposition time on solar cell efficiency

Deposition time				V_{oc} (mV)	J_{sc} (mA/cm²)	FF (%)	η(%)
p-Cu_2O	Thickness	n-Cu_2O	Thickness				
30 min	2μm	30 min	0.7μm	31.5	12.7	35	0.14
30 min	2μm	60 min	0.9μm	26.5	8.05	19	0.04
60 min	2.8μm	60 min	0.9μm	20.7	0.44	32	0.003

Figure 9(b) illustrates the diagram of proposed energy band for the homojunction Cu_2O solar cell. When the homojunction Cu_2O is fabricated, electrons diffuse from the n-Cu_2O film, which has a higher Fermi level, to the p-Cu_2O film, as it is majority in n-Cu_2O film. The same process happens for holes in the p-Cu_2O film. As a result, the flow of charges carriers established an internal electrical field. The internal potential depends on the difference of Fermi level between n-Cu_2O and p-Cu_2O thin film which can also be influenced by interface states of a homojunction. As a result, the interface states offset the effect of the internal potential through the energy

band bending [23], as shown in Figure 9(c), and lead to the open-circuit voltage reduction [24]. Also, reduction of voltage may be due to Cu_2O thin films prepared by electrodeposition are of a low doping level, especially for n-Cu_2O, and only a weak internal electrical field is established.

4. Conclusion
Cuprous oxide (Cu_2O) is a very promising semiconductor for the fabrication of solar cells due to the theoretical efficiency is approximately 20%, the direct band gap semiconductor $\cong$ 2 eV, abundant, non-toxic, prepared with simple methods at very low cost and relatively high absorption coefficient over the wavelength range of the solar spectrum. Thin films of p-Cu_2O and n-Cu_2O were controllably prepared using electrochemical deposition method by using optimum condition for each type. Photocurrent experiments show that the Cu_2O thin films synthesized in acid and alkaline media present n- Cu_2O and p- Cu_2O semiconductor character, respectively. The morphology and orientation were changed where p-Cu_2O show pyramid shape with orientation (111) and n-Cu_2O show dendritic shape with orientation (111) and (200). Optical characteristics of p-Cu_2O and n-Cu_2O were slightly affected. Homojunction Cu_2O solar cells were electrochemically fabricated by consecutively depositing an n-Cu_2O layer on a p-Cu_2O layer. Then, evaporate gold on n- Cu_2O. The best performance of the cell fabricated in this study was short-circuit current density 12.7mA/cm², open-circuit voltage 31.5mV, fill factor 35%, and conversion efficiency 0.14% under standard test condition. Thickness increase lead to efficiency decrease due to recombination of charge carriers' increase. High short-circuit current density due to the pyramid surface structure of p-Cu_2O caused the increase of the light path in the solar cell that could facilitate the photons to be absorbed more effectively. Low open-circuit voltage was due to energy band bending influence by interface states. The main limiting factor for the cell efficiency was the high resistivity of both p-Cu_2O and n-Cu_2O. Doping studies will be necessary to improve the performance of the homojunction Cu_2O solar cells further.

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