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SEM, EDAX AND UV-VISIBLE STUDIES ON THE PROPERTIES OF Cu_2S THIN FILMS

ANUAR KASSIM[a*], HO SOON MIN[a], LIM KIAN SIANG[a], SARAVANAN NAGALINGAM[b]
[a]*Department of Chemistry, Faculty of Science, Universiti Putra Malaysia, 43400 Serdang,Selangor, Malaysia.*
[b]*Faculty of Science, Universiti Tunku Abdul Rahman, 31900 Kampar, Perak, Malaysia*

Cu_2S thin films were produced by simple chemical bath deposition technique at various bath temperatures ranging from 55 °C to 75 °C. For chemical bath deposited thin films, copper sulphate solution was employed as Cu^{2+} source while thiourea solution provided the S^{2-} ions. The morphological, compositional and optical properties were investigated using scanning electron microscopy, energy dispersive analysis of X-ray and UV-Visible spectrophotometer, respectively. The grain size and average atomic ratio of Cu/S increased when the bath temperature was increased from 55 °C to 75 °C. The films deposited at 75 °C indicated high absorbance as compared with other bath temperatures.

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Keywords: copper sulfide, chemical bath deposition, thin films, bath temperature

1. Introduction

Copper sulfide thin films receiving ever-increasing attention due to its wide variety of applications such as solar cells, optoelectronic device and tubular solar collectors. Several techniques have been used to produce copper sulfide thin films such as spray pyrolysis [1], successive ionic layer adsorption and reaction technique [2], photochemical deposition [3], electrodeposition [4] and chemical bath deposition [5]. Among them, the chemical bath deposition method is hugely attractive since the technique possesses a number of advantages over conventional thin films deposition methods. The main advantages of this method are low cost, low deposition temperature and easy coating of large surfaces. The chemical bath deposition method has been actively investigated by other researchers for the growth of binary compound (CdS [6], In_2S_3 [7], CdSe [8], SnS [9], BaSe [10], SnSe [11], FeS [12] and NiSe [13]) and ternary thin films ($Cd_{0.5}Zn_{0.5}Se$ [14], $CuBiS_2$ [15] and CdZns [16]).

In this paper, we present for the first time the effects towards the chemical bath deposited Cu_2S thin films properties by varying the bath temperatures in the presence of tartaric acid as complexing agent. The morphological, compositional and optical properties of thin films were investigated using scanning electron microscopy, energy dispersive analysis of X-ray and UV-Visible spectrophotometer.

2. Experimental

2.1 Preparation of thin flms

Microscope glass slides were used as the substrate during the deposition process. The substrates were first cleaned in ethanol solution, subsequently ultrasonically washed with distilled

*Corresponding author: anuar@science.upm.edu.my

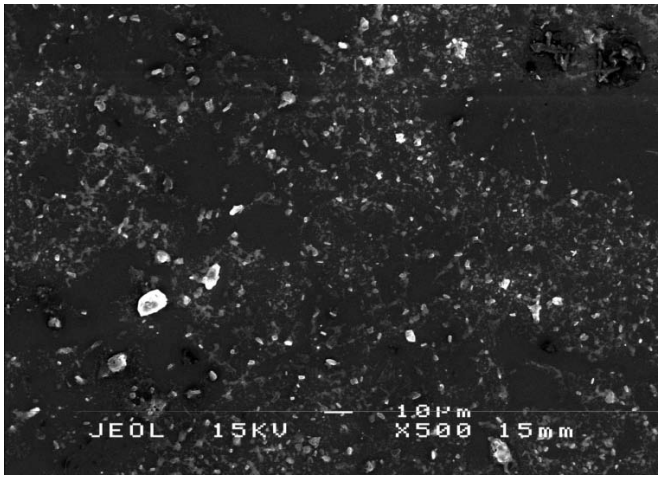
water. Substrates were then dried in an oven at 90 °C. Copper sulfate, thiourea, tartaric acid and hydrochloric acid of analytical reagent grade were used as received. Deionized water (Alpha-Q Millipore) with a resistivity of 18.4 MΩcm was used as solvent. Aqueous solutions of copper sulfate, thiourea and tartaric acid were separately prepared before experiment. 25 mL of copper sulfate (0.15 M) and 25 mL of tartaric acid (0.15 M) were mixed in a beaker. The tartaric acid was served as complexing agent to chelate with Cu^{2+} to obtain complex solution. Then, 25 mL of thiourea (0.15 M) was added and the pH of the solution was adjusted to 2.5 by addition of hydrochloric acid. Substrates were immersed vertically in the beaker. The beaker was not stirred during the thin films deposition. In order to determine the best conditions for the deposition process, the films were deposited at different bath temperatures (55, 65 and 75 °C). After completion of films deposition (120 min), the deposited films were then washed with distilled water and dried in air at room temperature for further characterization.

2.2. Characterization of thin films

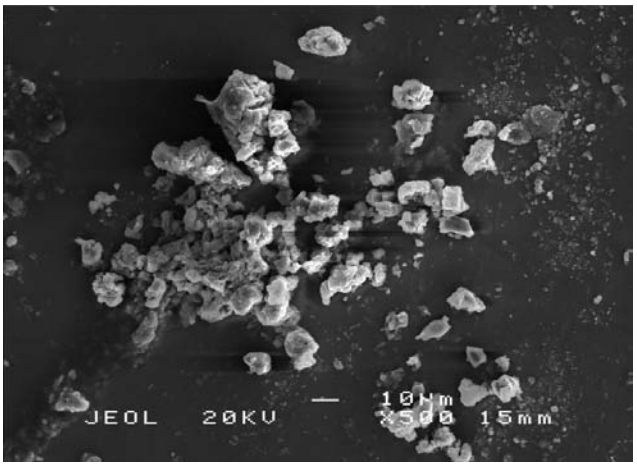
The surface morphology was observed by a scanning electron microscopy (JEOL, JSM-6400). All the samples taken at 20 kV with a 500 X magnification. The elemental composition of the films was studied by scanning electron microscope attached with energy dispersive analysis of X-ray (EDAX) analyzer. Optical absorption study was carried out using the Perkin Elmer UV/Vis Lambda 20 Spectrophotometer. The film-coated microscope glass slide was placed across the sample radiation pathway while the uncoated microscope glass slide was put across the reference path.

3. Results and discussion

Fig. 1 shows the scanning electron microscopy (SEM) micrographs for the films deposited at various bath temperatures from 55 °C to 75 °C. The morphologies are very different in these thin films as shown in Figure 1. Considering first the films deposited at lower bath temperature (55 °C), the films obtained present less compact morphology due to less materials deposited on the surface of the substrate. Ii is also observed that fine grains (2-3 μm) are distributed randomly throughout the substrate. In contrast, the grain sizes are found to increase with bath temperature and found to be 8-10 μm for films deposited at 65 °C. Based on the SEM micrographs, it is clearly observed that the grains of sample deposited at the highest bath temperature (75 °C) is relatively larger in size than that of the films prepared at lower bath temperature (55 °C and 65 °C). The grain size ranges from 25 μm to 30 μm and they are agglomerated in some places. These grains are distributed to cover the surface of the substrate completely. It was evident that with the increase in bath temperature, the grains become larger in diameter.



a



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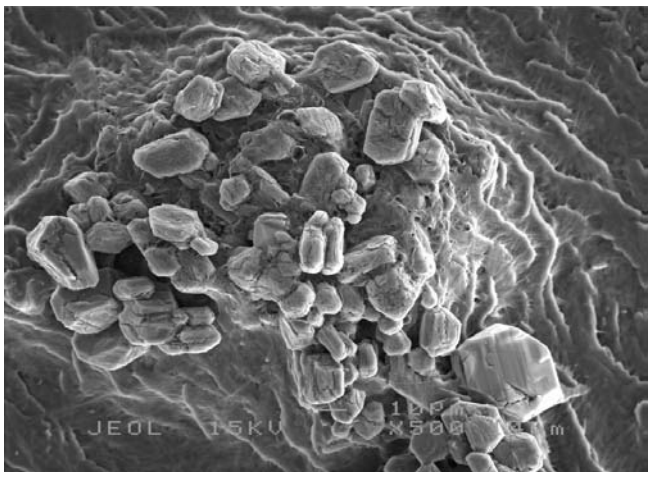

c

Fig. 1. Scanning electron microscopy (SEM) micrographs of Cu_2S thin films deposited at different bath temperatures (a) 55 °C (b) 65 °C (c) 75 °C

The optical properties of the chemical bath deposited Cu_2S thin films were investigated from the absorption measurement in the range of 350-800 nm as shown in Figure 2. All the films show the same patterns with higher absorption on the shorter wavelength side. The films deposited at 75 °C have high absorbance as compared with other bath temperatures, indicating more surface area could be observed for these thin films.

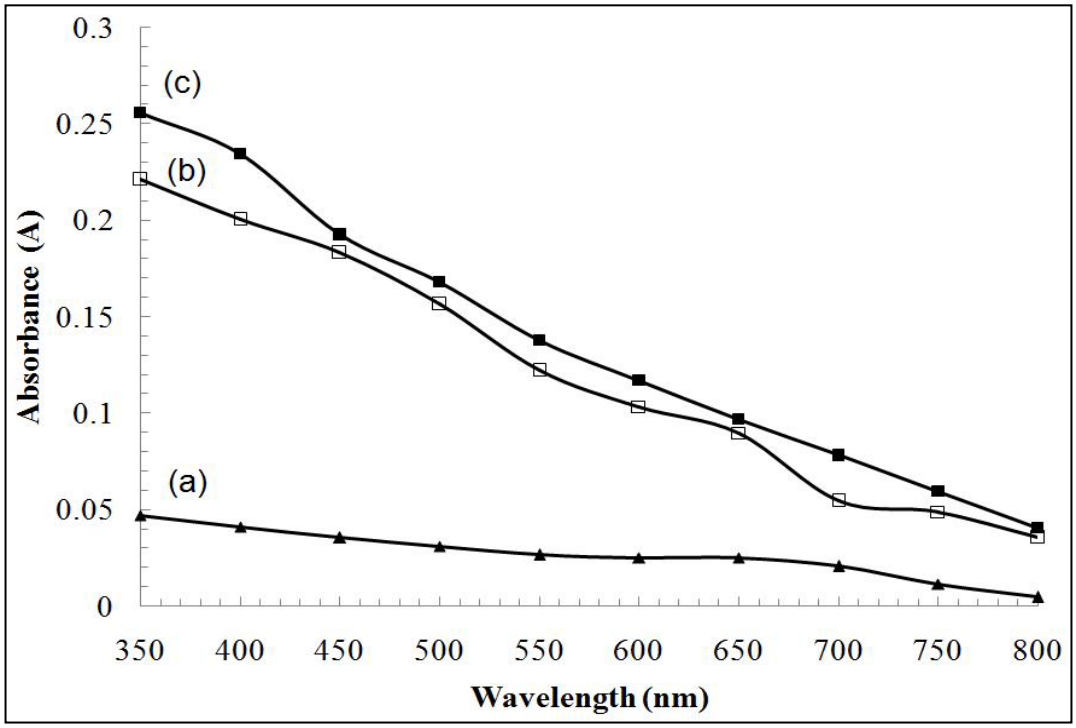

Fig. 2. Optical absorbance versus wavelength of Cu_2S thin films deposited at different bath temperatures (a) 55 °C (b) 65 °C (c) 75 °C

The compositional analysis of the thin films was investigated by energy dispersive analysis of X-ray (EDAX) method (Figure 3). Table 1 lists the atomic ratio of the copper sulfide thin films prepared at various bath temperatures. EDAX analysis indicates the presence of copper and sulphur for all the deposited films. The average atomic ratio of Cu/S calculated from the quantification of the peaks, gives the value of 1.89, 1.95 and 2.25 for different bath temperatures of 55 °C, 65 °C and 75 °C, respectively. These results indicate that the average atomic ratio of Cu/S increases with increasing bath temperature.

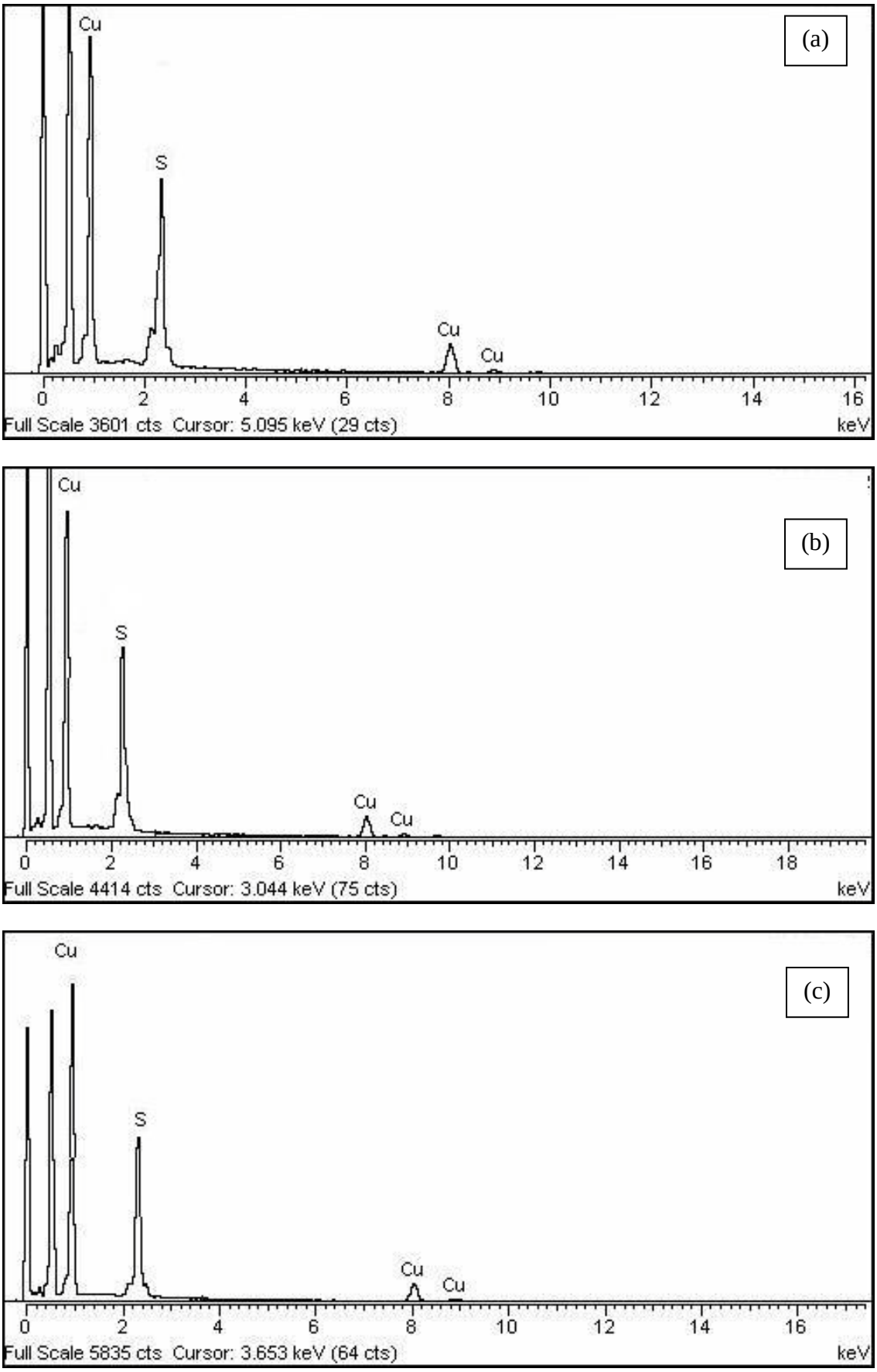

Fig. 3. Energy dispersive analysis of X-ray (EDAX) spectra of Cu_2S thin films deposited at different bath temperatures (a) 55 °C (b) 65 °C (c) 75 °C.

Table 1: Atomic percentage composition of Cu_2S thin films deposited at different bath temperatures

Bath temperature (°C)	Atomic percentage of copper (%)	Atomic percentage of sulphur (%)	Cu:S
55	65.44	34.56	1.89
65	66.05	33.95	1.95
75	69.20	30.80	2.25

4. Conclusion

Synthesis of Cu_2S thin films was carried out by chemical bath depsoition method using copper sulfate, thiourea and tartaric acid. Based on the SEM analsyis, the grain size decreased as the thin films prepared at lower bath temperature. The average atomic ratio of Cu/S increased to 2:1 as the bath temperature was increased from 50 °C to 70 °C. The films deposited at 70 °C have high absorbance as compared with other bath temperatures.

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