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Chemical Bath Deposited Copper Tin Sulphide Thin Films in the Presence of Complexing Agent: EDX and SEM Analysis

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ABSTRACT

Copper tin sulphide thin films have been prepared using chemical bath deposition technique on soda glass substrate. In this work, the chemical bath contains different solutions such as copper sulfate, tin chloride, triethanolamine and sodium thiosulphate which could serve as a Cu^{2+} , Sn^{2+} , complexing agent and S^{2-} ion, respectively for the deposition of thin films. The scanning electron microscopy and energy dispersive analysis X-ray technique have been used to study morphology and composition of films. The results indicate that smaller grains could be obtained using lower concentration of sodium thiosulphate solution as shown in scanning electron microscopy analysis. In conclusion, the morphologies of films indicate a clear dependence on the solution concentration.

Keywords: Ternary compound, Chalcogenide metal, Thin films, Solar cells

INTRODUCTION

Nowadays, there are many different types of binary, ternary and quaternary thin films have been deposited onto substrate. The applications of thin films include in optics field, mechanics and magnetics field. Extensive research has been carried out on the properties of thin films as pointed out by many researchers. For example, X-ray diffraction [1-5], atomic force microscope [6-10], transmission electron microscopy [11-15], X-ray photoelectron spectroscopy [16-20] and UV-Visible spectrophotometer [21-26] have been employed to investigate the structure, topography, morphology, surface chemistry and optical behavior of thin films, respectively as reported by many researchers.

Here, the influence of sodium thiosulfate solution on the ternary thin films in the presence of complexing agent was investigated. Then, the compositional of samples and the morphology of films were studied by using EDX and SEM technique.

MATERIALS AND METHODS

In this experiment, all the solutions were prepared in deionized water (Alpha-Q Millipore) and all the chemicals used (copper sulfate, tin chloride and sodium thiosulfate) for the depositions were analytical grade. Here, the influence of various concentrations (0.07 M and 0.03 M) of sodium thiosulphate solution was studied.

First of all, 15 mL of 0.1 M of triethanolamine was added into the beaker that contained 15 mL (0.06 M) of $SnCl_2$ and 15 mL of (0.06 M) CuSO₄, respectively. Then, 15 mL of sodium thiosulphate was placed into the 2 separate beakers. Lastly, these solutions were mixed together for few minutes. Hydrochloric acid was used to adjust the pH of solution to pH 1.1. The soda lime glass was used as the substrate during deposition process. The ultrasonically clean glass substrate was then placed vertically inside the beaker without disturbing it. The deposition process was carried out for 110 min at 60°C. After completion of film deposition, the glass substrate was removed from the beaker and cleaned with distilled water. Then the deposited films were dried in the desiccator and subjected to further analyses.

The morphological and compositional of obtained films were investigated by using scanning electron microscopy (JEOL JSM-6400) and energy dispersive analysis X-ray technique, respectively.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM) is a very powerful magnification instrument that produces detailed micrograph of surfaces of samples. Advantages of SEM include it can form much higher magnification images and can produce high resolution micrograph of a sample surface. Nowadays, there are many studies have been made by using this technique on the chalcogenide thin films. For instance, the influence of bath temperature on the obtained films has been studied using SEM by Kumarage et al. (40-80°C) [27] and Gopinath et al. (50-90°C) [28], respectively. Meanwhile, the effect of the deposition time on the Cu_2ZnSnS_4 films (10-40 mins) and SnS films (24-72 hrs) has been reported by Taoufik et al. [29] and Patel [30], respectively.

The morphology of thin films prepared by using 0.03 M and 0.07 M of sodium thiosulphate solution was studied as indicated in Figures 1 and 2, respectively. Obviously, we have seen that larger grains (50-200 μ m) for the films prepared in the presence of higher concentration. This is due to these films form after the aggregation of the smaller particle. On the other hand, smaller grain sizes of about 30-150 μ m could be detected for the films prepared using lower concentration. Also, these films show inhomogeneous distribution of particle over the surface and highly irregular shapes as indicated in SEM analysis.

In this work, the SEM is equipped with the Energy dispersive analysis X-ray (EDX) to investigate the chemical composition of films. Here, a few of the sample spots for the films prepared using various concentrations were focused and analyzed by using EDX technique as shown in Figure 3 (0.03 M sodium thiosulphate) and Figure 4 (0.07 M), respectively. The EDX analysis displays that average maximum and minimum ratio of atomic percentage of sulphur, copper and tin was 38.45%, 61.58%, 21.26% and 23.02%, 48.44%, 8.32%, respectively for the films prepared using 0.03 M of sodium thiosulphate (Table 1).



Figure 1: SEM micrograph of copper tin sulphide thin films prepared by using 0.03 M sodium thiosulphate solution



Figure 2: SEM micrograph of copper tin sulphide thin films prepared by using 0.07 M sodium thiosulphate solution



Figure 3: EDX spectra of copper tin sulphide thin films prepared using 0.03 M sodium thiosulphate solution

Table 1: Atomic composition obtained by EDX of copper tin sulphide thin films prepared by using 0.03 M sodium thiosulphate solution

Spectrum	S (%)	Cu (%)	Sn (%)
Spectrum 1	30.10	61.58	8.32
Spectrum 2	32.23	53.81	13.96
Spectrum 3	23.02	55.72	21.26
Spectrum 4	38.45	48.44	13.11
Spectrum 5	26.77	60.74	12.49
Maximum	38.45	61.58	21.26
Minimum	23.02	48.44	8.32



Figure 4: EDX spectra of copper tin sulphide thin films prepared using 0.07 M sodium thiosulphate solution

Table 2: Atomic composition obtained by EDX of copper tin sulphide thin films prepared by using 0.07 M sodium thiosulphate solution

Spectrum	S (%)	Cu (%)	Sn (%)
Spectrum 1	41.53	3.02	55.45
Spectrum 2	44.17	4.63	51.20
Spectrum 3	38.12	2.82	59.06
Spectrum 4	41.24	3.98	54.78
Spectrum 5	35.79	2.74	61.46
Maximum	44.17	4.63	61.46
Minimum	35.79	2.74	51.20

Meanwhile, the obtained EDX results show that raising the concentration of sodium thiosulfate solution to 0.07 M resulted in increasing of atomic percentage of sulphur (Table 2). The EDX data reflect that average maximum and minimum of atomic percentage of sulphur, copper, tin was 44.17, 4.63, 61.46 and 35.79, 2.74, 51.2, respectively for the films prepared using 0.07 M sodium thiosulphate.

CONCLUSION

Here, copper tin sulphide thin films have been successfully deposited onto soda lime glass substrate by using chemical bath deposition method in the presence of triethanolamine. The influence of different concentrations of sodium thiosulphate solution was investigated by using scanning electron microscopy and energy dispersive analysis x-ray technique. The obtained experiment results show that smaller grain size could be seen for the films prepared by using lower concentration of sodium thiosulphate. In other words, the compositional of films show significant dependence on the concentration of solution.

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