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Synthesis of Iron Copper Sulphide (FeCuS) Thin Film and its Characterization

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ABSTRACT
The Influence of SILAR cycle on the optical, morphological as well as compositional properties of Iron Copper Sulphide (FeCuS) thin film was reported. Utilizing the straightforward and reasonably priced SILAR technique, the deposition was carried out by the use of soda lime glass (SLG) substrates. With a concentration of 1.0M for Iron (Fe) ions, the deposition process was repeated 50, 60, and 70 times, with an 80-seconds dip time per cycle. Morphological characterization of the thin film was analyzed with Scanning Electron Microscopy (SEM) instrument. The compositional properties were analyzed with Energy Dispersive X-Ray machine. A UV-VIS Spectrophotometer was utilized to examine the thin film's optical characteristics. The presence of deposited thin film samples is confirmed by the SEM results, which display higher peaks for the elements Fe, Cu, and S. The results of the EDX analysis demonstrated that an increase in concentration enhances the homogeneity and adhesion of the deposited films in addition to their crystallinity. It was discovered that the thin film's energy band gap for each SILAR cycle was 0.9 eV, 1.3 eV, and 2.99 eV respectively.

Keywords:
Morphological,
Compositional,
Deposition,
Cycle,
SILAR.

INTRODUCTION

Thin film is known as a material layer applied to a metal, ceramic, semiconductor, or plastic substrate that is incredibly thin. The process of covering a surface with a thin film is called thin film deposition. It refers to any method of applying a thin layer of material to layers that have already been applied or to a substrate (Chopra, 1969). Diverse deposition techniques including chemical bath deposition, electrodeposition, vacuum evaporation, electron beam evaporation, spray pyrolysis, and other techniques can be employed in the deposition of thin film. The majority of these methods are costly and demand highly regulated formation conditions. A highly controlled formation condition is necessary for the majority of these costly techniques. The Successive Ionic Layer Adsorption and Reaction (SILAR) technique is a more appealing technique for producing thin films because of its ease of use, cost next to nothing and prospect for bulky deposition (Guzeldir *et al.*, 2012). The deposition process is affected by variables including solution temperature, substrate type, pH and concentration, substrate area as well as dipping and rinsing times (Pathan and Lokhande, 2004). The production of these thin films is done in this modification by submerging the substrate in cationic and anionic precursors that are placed independently, and then cleaning the substrate with ion-exchanged water in between each immersion in order to prevent homogenous precipitation in the solution. Several researchers have successfully deposited FeCuS thin films by the use of chemical bath deposition (CBD) (Obasi *et al.*, 2016), solution growth technique (SGT) (Uhuegbu, 2010) and SILAR technique (Adeniji *et al.*, 2020).

In this work, we use the SILAR technique to deposit FeCuS thin films by the use of Triethanolamine acid (TEA) and Ethylenediaminetetraacetic acid (EDTA) as complexing agents. To obtain high quality film, a parameter such as deposition cycle was considered.

MATERIALS AND METHODS

The slides were washed with detergent and cotton wool to get rid of any dirt on the glass. They were then degreased in acetone for duration of 24 hours to eliminate impurities as well as provide a nucleation center for the thin film's growth.

In the current study, 1.0 M of ferrous nitrate and copper chloride, each complexed with triethanolamine (TEA) and ethylenediaminetetraacetate (EDTA), served as the cationic precursor. The mixture's pH was stabilized using ammonia solution.

The anionic precursor is 1.0 M of Thiourea solution. In both situations, de-ionized water is used as the rinsing solvent. The cationic precursor was magnetically stirred for two minutes so as to create a homogenous mixture. The intervals between dipping and rinsing are maintained at 20 s. Each cycle of the deposition took 80 seconds of dip time. The procedure is repeated with 1.0M concentration of iron (Fe) ions for 50, 60, and 70 deposition cycles. The glass slides were taken out and left to dry naturally after the deposition. FeCuS thin films were obtained correspondingly and their characteristics were examined.

RESULTS AND DISCUSSION

Optical studies

A spectrophotometer was used to measure the UV-visible absorbance spectra of the deposited sample at room temperature in order to examine its optical characteristics.

UV-visible studies

The wavelength range for the optical absorbance measurements is 200 nm to 1000 nm. The optical absorption spectra were utilized for the calculation of the energy band of FeCuS thin films. By Plotting a graph of $(\alpha h\upsilon)^2$ against $(h\upsilon)$, the band gap energy was found as depicted in Figure 1. Equation 1 can be used to analyze the transmission data using the classical relation so as to obtain the optical band gap (Chauhan and Chaudhary, 2011).

$$\alpha h\nu = A(h\nu - E_g)^n \quad (1)$$

Where A is a constant, $h\nu$ is the photon energy and E_g is the optical band gap.

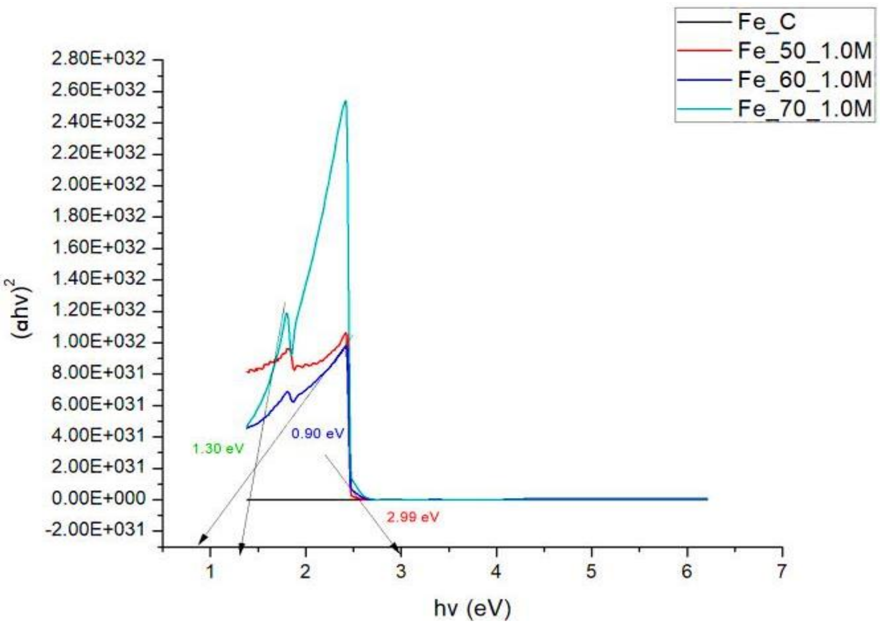

Figure 1: Graph of $(\alpha h\nu)^2$ against photon energy of 1.0 M deposited on SLG substrates

The high energy area in the plot is where the energy axis intercept is located following the extrapolation of the straight-line path. The samples' band gap values range from 0.90 eV to 2.99 eV, as can be seen. For samples deposited at 60 and 70 SILAR cycles, band gap values increased with the cycle, whereas the sample deposited at 50 SILAR cycles had a higher band gap value of 2.99 eV. There is an obvious contrast between the range of these direct energy band gap values and the result of Adeniji *et al.*, 2020. The values obtained were found to be lower which could be attributed to the variation of SILAR cycle used. This implies that 1.0M Fe ion concentration at lower SILAR Cycle gives rise to higher energy band gap values and vice-versa.

Morphological and Elemental analysis studies

The prepared film is analyzed by SEM to investigate the surface morphology; the resulting images are displayed in Figures 2, 4, 6, and 8. The pictures demonstrate that there are no visible pores and that the grains are evenly spaced throughout the surface.

Using an EDX machine, the elemental analysis study is conducted. The EDX result, which verifies the presence of elements Fe, Cu, and S in the deposited thin films, is displayed in Figures 3, 5, 7, and 9.

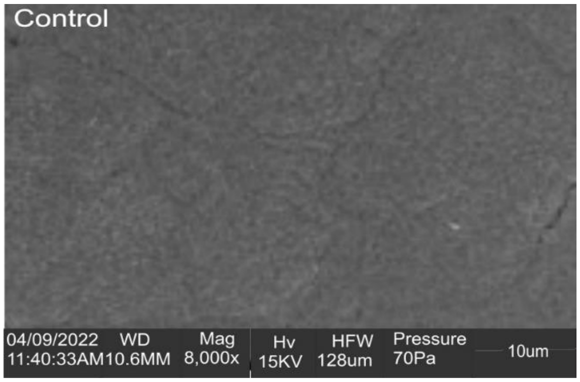

Figure 2: SEM micrograph for control

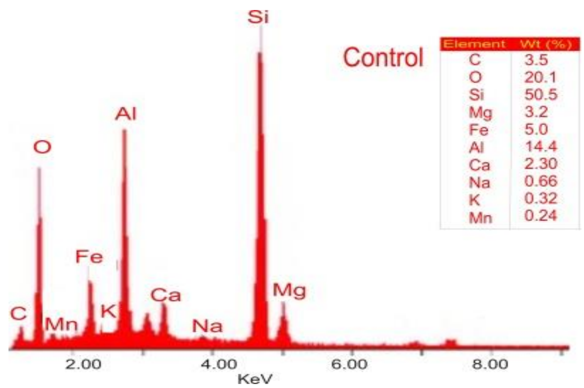

Figure 3: EDX spectra for control

The control, depicted in Figure 2, has a smooth surface with uniformly spaced tiny, rounded grains. The glass substrate is the source of a higher peak of Si in the control, as depicted in Figure 3. Other elements like C, O, Fe, K, Al, Na, and Mg appeared as a result of the adhesive element and glass substrate used during sample preparation (Okorieimoh *et al.*, 2019).

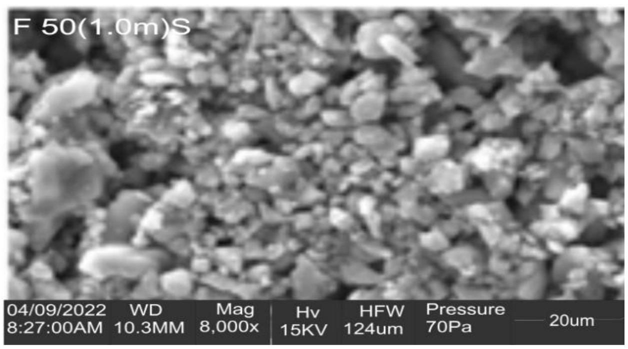

Figure 4: SEM micrograph at 50 cycles

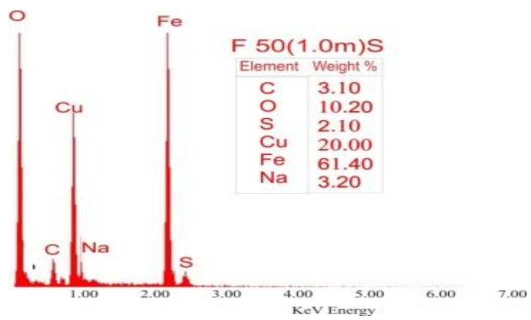

Figure 5: EDX spectra at 50 cycles

The spherically shaped, uneven, void-free pattern shown in Figure 4 is consistent with the findings of Osanyinlusi & Aregbesola (2021). Higher peaks for the elements Fe, Cu, and S were shown in the EDAX results displayed in Figure 5, which verified the existence of deposited FeCuS thin film samples.

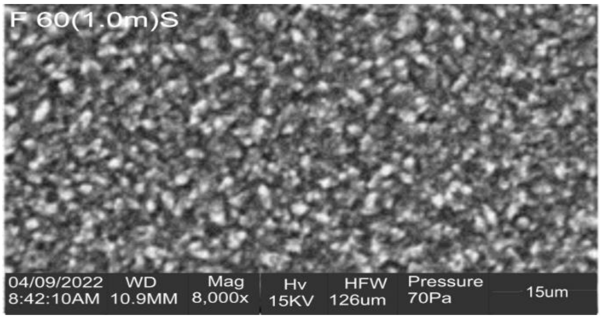

Figure 6: SEM micrograph at 60 cycles

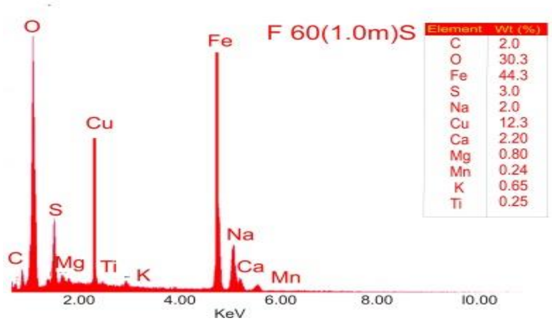

Figure 7: EDX spectra at 60 cycles

1.0 Mol. FeCuS film on an SLG substrate is depicted in Figure 6. The micrograph reveals a compact structure made up of one kind of small, closely spaced objects. The grains are nearly identical in size, spherical, and clearly defined. This is in agreement with the submission of Kolandavel and Suresh, (2015). The smooth grain background and smaller, more compact grains are signs of one-step growth via multiple nucleation (Kolandavel and Suresh, 2015). The presence of deposited FeCuS thin film samples was confirmed by the EDAX results displayed in Figure 7, which also showed higher peaks for the elements Fe, Cu, and S.

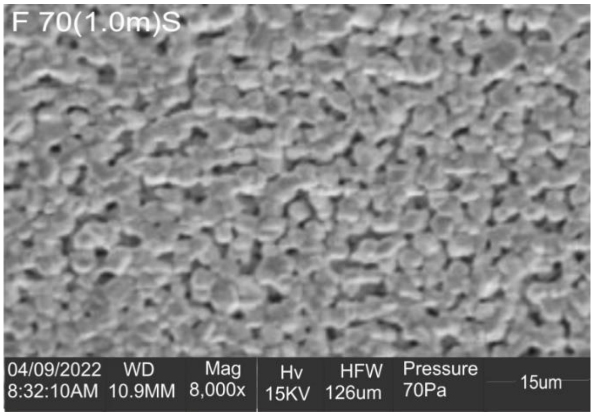

Figure 8: SEM micrograph at 70 cycles

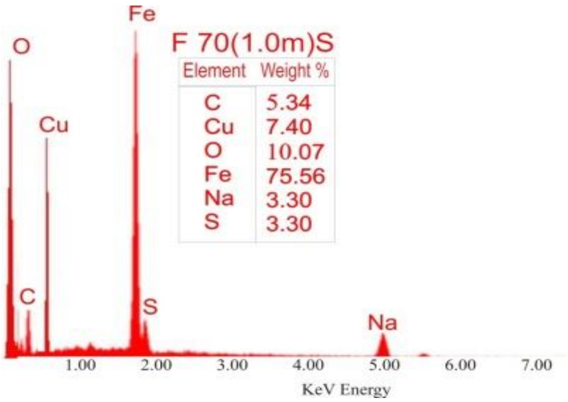

Figure 9: EDX spectra at 70 cycles

A high magnification SEM examination of 1.0 Mol. of FeCuS film on SLG revealed that it had a nanoporous morphology (Figure 8). In the FeCuS films, atoms gathered into interconnected nanoparticles. The nanoparticles are partially separated by gaps that are relatively average in width. Liyi *et al.* (2015) obtained similar results. The presence of deposited FeCuS thin film samples was confirmed by the EDAX results displayed in Figure 9, which also showed higher peaks for the elements Fe, Cu, and S. The Fe ion has the highest peak.

CONCLUSION

FeCuS thin film deposition on Soda lime glass (SLG) substrate and characterization was accomplished successfully through SILAR technique from the aqueous solution of Ferrous nitrate (as a source of iron), cuprous chloride (as the copper source), Thiourea (as Sulphide source) was used as precursors. The optical, morphological and compositional properties of the samples were investigated. After computing the energy gap values from UV-Vis absorption measurements, it was discovered that they range from 0.90 to 2.99 eV. Elevated peaks for elements Fe, Cu, and S in the EDX results verify the presence of deposited thin film samples; the SEM images demonstrated a uniform film morphology covering the entire substrate surface, with grain size developing as immersion cycles increase. The results of the deposited FeCuS thin film demonstrate that it can be used to produce solar cells and has numerous optoelectronic applications where low energy band gaps are preferred.

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