ISSN Online: 2756-3898, ISSN Print: 2714-500X https://doi.org/10.47514/phyaccess.2022.2.2.003

Synthesis and Characterization of Semiconducting Iron Copper Sulphide Thin Films: A Review

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Received 17-07-2022
Accepted for publication 20-08-2022
Published 22-08-2022

Abstract

Iron copper sulphide (FeCuS) thin films have attracted much importance due to their exceptional properties for device applications. The chemical deposition techniques of chemical bath deposition (CBD) and Successive ionic layer absorption and reaction (SILAR) have been employed to synthesize FeCuS thin films with good substrates adhesion. The various advantages and shortcomings of these methods were discussed with their capability to control the morphology and opto-electrical properties of the films. The experimental measurements indicated that the films exhibit great potential for optoelectronic applications such as window materials in solar cell fabrications and coatings of different types. Due to the current interest in FeCuS thin films, this review is intended to provide current information on the synthesis, characterization, and applications of iron copper sulphide thin films.

Keywords: Band gap; Chemical bath deposition; Iron copper sulphide; SILAR; Thin films.

I. Introduction

Iron sulphide (FeS) as an important semiconducting material has gained much interest due to its high absorption coefficient (-$10^5\ cm^{-1}$) and low optical band-gap (0.95 eV) [1], which are potentials in optoelectronics and spintronics device applications [1-4]. The iron sulphide system has complex chemistry because of its various phases and stoichiometric forms, i.e. pyrite (cubic FeS_2), mascasite (FeS_2), pyrrhotite-4M (Fe_7S_8), pyrrhotite-IT ($Fe_{1-x}S$), greigite (cubic spinel Fe_3S_4), troilite -2H (FeS) as well as Marcasite ($Fe_{1+x}S$) [2, 4]. Due to these various phases, iron sulphide has received applications in high capacity anode materials in lithium batteries production, photo-electrochemical applications, sensors, photovoltaic, among others [1, 2]. In view of the fact that surphur ions in Fe-S system is relatively unstable, the oxidation of sulphur during the recrystallization process has led to the inconsistency in conduction types, as p-type and n-type conductivity have been reported for iron sulphide in literature [1].

Copper sulphide (CuS) on the other hand, is an important p-type material that belongs to group I-VI compound semiconductor [5, 6]. As a chalcogenide thin film, CuS has received attention in its applications in various field of science and innovation. The attraction of this material is mainly due to its semiconducting properties and application in various optical and electrical devices such as solar control coatings, microwave shielding coatings, potential nanometer scale switch, solar cells, LEDs, photoconductor, antireflection coating, lasers, IR detection, low temperature gas sensor applications, optical filters, cathode material in lithium rechargeable batteries among others [7-9]. Also, copper and sulphur which constitute copper sulphide are abundant in nature and less toxic. At room temperature, copper sulphide forms five stable solid phases of covellite (CuS), anilite ($Cu_{1.7}S$), digenite ($Cu_{1.8}S$), Djurleite ($Cu_{1.95}S$)

and Chalcocite (Cu_2S) [8]. Different crystal structures of copper sulphide are possible depending on the various value of "x" in Cu_xS, such as hexagonal, Pseudo-cubic, orthorhombic and tetragonal [6]. However, of all the possible phases of copper sulphide, chalcocite Cu_2S, has the largest electron diffusion length, the longest wavelength cut-off, the highest optical absorption and stability against oxidation [10]. It is also the most used form of copper sulphide due to its large optical band gap [8].

Iron copper sulphide (Fe-Cu-S) as a special alloyed of iron sulphide (FeS) and copper sulphide (CuS), has gained much interest in science and technology due to its versatility and potential applications in opto-electronics and microelectronics devices. The material has properties between the binary constituents of FeS and CuS with a structure that could be extremely non-stiochiometric [11]. Fe-Cu-S exhibits interesting properties such as anomalous optical band-gap values, high optical constants as well as high mobility that make the material suitable for application in various opto-electronics devices. The Fe-Cu-S system presents a crystalline structure in which the iron and copper ions are tetrahedrally coordinated with sulphur [12].The material elements of iron (Fe), copper (Cu) and sulphur (S) are naturally available and are more eco-friendly when compared to ternary materials like copper zinc selenide ($CuZnSe_2$), copper zinc telluride (CuZnTe2) and copper aluminium selenide ($CuAlSe_2$) [13]. This paper is aimed at reviewing the methods and characterization techniques that have been used over the years to synthesize iron copper sulphide (Fe-Cu-S) alloyed thin films, which hitherto has not been made. The paper focuses mainly on the chemical deposition processes, bearing in mind the advantages they have over the physical processes like sputtering and evaporation. The chemical deposition processes are simple, clean with high deposition rate and are mostly used in microelectronics applications, such as gate insulating layers, oxidation barriers and polycrystalline silicon [14]. The study will help researchers to invest their time in the advancement and application of the material instead of wasting time to working out an ideal method. The paper also aids future researchers by highlighting the research gaps related to Fe-Cu-S thin films, thus promoting great study on the material.

II. SYNTHETIC METHODS OF THE FILMS

Synthesizing iron copper sulphide thin films have been achieved using well-established chemical deposition processes such as chemical bath deposition (CBD) or solution growth technique (SGT) and Successive ionic layer absorption and reaction (SILAR) on glass (soda-lime) substrates at various pH of 7.5 – 10.10 at room temperature (300 to 353k) [15-20]. These various approaches have the ability to control the structural, optical and resistive properties of the deposited films for device applications [21]. The various preparative conditions as well as their characterization techniques reported in literature are shown in Table I. The most used synthetic methods of CBD or SGT and SILAR are discussed in the following sections.

A. Chemical bath deposition

Chemical bath deposition (CBD) technique sometimes referred to as the controlled precipitation (CP) or solution growth technique (SGT) [22], is an important thin film deposition technique due to its versatility in producing high quality films for opto-electronic applications [22, 23]. The method has largely been used to deposit wide range of materials such as binary, ternary and quaternary thin films [24]. In CBD, deposition occurs at low temperature from an aqueous solution of the reacting mixture with the help of a ligand or a complexing agent [22-24]. Usually, it involves dipping the desired substrate in the bath solution containing the metal ions along with the source of sulphide [22, 24]. The method is based on the complexing agents controlled release of complex ions (cations or anions) in an aqueous solution on which the substrate is dipped. The process starts with the nucleation phase then, followed by the growth phase which is characterized by increased thickness and terminates with the separation of the ions over certain duration of time [22, 24]. The CBD has several advantages which include; (i) simplicity, (ii) high deposition rate (iii) easy control of stoichiometry of deposited materials, and (iv) easy doping via the introduction of impurities [22-25].

FeCuS thin films synthesized by CBD or SGT method have been characterized for a variety of properties. The SGT synthetic method was exploited by [18] to deposit FeCuS thin films, where 5 ml of (1 M) ferrous nitrate, cuprous chloride and thiourea were used to grow the material. The complexing agents (EDTA and TEA) were used to reduce the precipitation process. Experimental characterizations revealed a high reflecting and absorbing material with a refractive index that varies from 1.2 to 2.3. Essentially, the high transmittance, as well as the direct and indirect band-gap range of (2.4 – 2.8 eV), and (0.6 – 1.0 eV) respectively, confirmed that the grown FeCuS thin films would be appropriate for eye glasses coating, anti-reflection coating, solar cells production as well as solar control and thermal conversion processes [18]. Crystalline thin films of FeCuS were prepared at 353 K via the CBD on glass substrates by Reference [15]. Distilled water was used as the solvent along with other preparative parameters indicated in Table I. The structural (XRD and photomicrograph) results indicated polycrystalline films with grain sizes that vary with deposition duration. Optical measurements also indicated a low reflectance and direct-band-gap values that vary from 2.50 to 2.80 eV. These results indicated that the deposited material is a wide band gap semiconductor, and could be applied in solar cell production as an absorber layer [15]. Reference [16] synthesized tetragonal structured ternary FeCuS thin films using iron (III) trioxonitratenanohydrate, copper chloride dehydrate and thiourea at 303 K as outlined in Table I. The addition of thiourea into the reacting mixture yielded a 'sky blue jelly-like solution'.

Table I. Iron copper sulphide synthesis methods and characterizations.

S/N	Method	Precursors Parameters	Varying Parameters	Characterization employed	References
1	CBD	3ml iron (III) trioxonitrate and copper chloride dehydrate, 2 ml thiourea, 1 ml EDTA, 1ml TEA, 5 ml (14.0M) ammonium solution and water.	Deposition time (30-150 min)	* UV-VIS Spectrophotometer * X-ray diffraction * Microscopic techniques	[15]
2.	CBD	3ml iron (III) trioxonitrate and copper chloride dehydrate, 2 ml thiourea, 1ml EDTA, 1 ml 3 ml ammonium solution, 35ml distilled water	Deposition time (12-60 hr)	* UV-VIS Spectrophotometer * X-ray diffraction * Microscopic techniques	[16]
3.	CBD	3 ml iron (III) trioxonitrate and copper chloride dehydrate, 2 ml thiourea, EDTA, 1ml TEA, 35ml distilled water, and (2-6) ml ammonium solution	pH (7.9 to 9.9)	* UV-VIS Spectrophotometer * Microscopic techniques	[17]
4.	SGT	5 ml ferrous nitrate, cuprous chloride, 3 ml (0.1m) EDTA and TEA, 3 ml ammonia, 10 ml thiourea and 20 ml water	--------	* UV-VIS Spectrophotometer * Microscopic techniques	[18]
5.	SILAR	5 ml ferrous nitrate and cuprous chloride, 3ml (14M) ammonia solution, 3ml TEA, 3ml (0.1M) EDTA, 20ml (1.0M) thiourea, and 20ml distilled water.	Deposition cycles (20, 30 and 40)/ Concentrations (0.7M and 1.0M)	* UV-VIS Spectrophotometer * Four point probe	[19]
6.	SILAR	Same as in [19]	Deposition cycles (20, 30 and 40)	* UV-VIS Spectrophotometer *Four point probe	[20]

For the preparation, the deposition duration was varied between 12 to 60 hours to engineer the optical and structural characterization of the material. Commonly, the authors indicated that the thickness, transmittance, refractive index, composition as well as the structural properties of the material were influenced by the deposition time, and achieved a band gap of 2.40 eV, which could effectively be considered as absorber layers in solar cells fabrication [16].In another study, [17] investigated the effect of pH values on the optical properties of iron copper sulphide thin films. The outline of the experimental parameters is presented in Table I and follows the chemical mechanism;

$$(NO_3)_3.9H_2O + EDTA \longrightarrow [(EDTA)]+ + NO_3^-$$

$$[Fe(EDTA)]+ \longrightarrow Fe^{3+} + EDTA^{2-} \quad (1)$$

$$CuCl_2.2H_2O + TEA \longrightarrow [(TEA)]^+ + Cl^-$$

$$[Cu(TEA)]^+ \longrightarrow Cu^{2+} + TEA \quad (2)$$

$$(NH_2)_2CS + OH- \longrightarrow (NH_2)_2CO + HS^-$$

$$HS^- + OH^- \longrightarrow H_2O + S^{2-} \quad (3)$$

$$Fe^{3+} + Cu^{2+} + S^2 - \longrightarrow FeCuS_3 \quad (4)$$

The ions Fe^{3+} and Cu^{2+} were complexes formed from the various metal salts and the complexing agents (EDTA and TEA) as indicated in (1) and (2). While the hydrolysis of the thiourea yielded the sulphide (S^{2-}) ions as in (3). Thus, the deposition of FeCuS was achieved by the slow release of these ions on the glass substrates as in (4) [17].The micrographs showed polycrystalline films with smaller grains sizes along the pH range (Fig. 1). A band gap range of 2.5 to 2.8 eV was estimated for the films with absorbance that decreased with an increase in pH values. Conclusively, the study confirmed that the synthesized films are potential material for several optoelectronic devices [17].

Although the CBD or SGT is an effective chemical method to grow quality films, it has various drawbacks such as; (i) poor understanding of the complex thermodynamic and reaction kinetics involved in the deposition process (ii) the toxic and corrosive nature of the reacting baths, (iii) the difficulty in the control of the uniformity of the films [26]. These downsides of CBD motivated some authors to study about SILAR technique for the deposition of iron copper sulphide thin films.

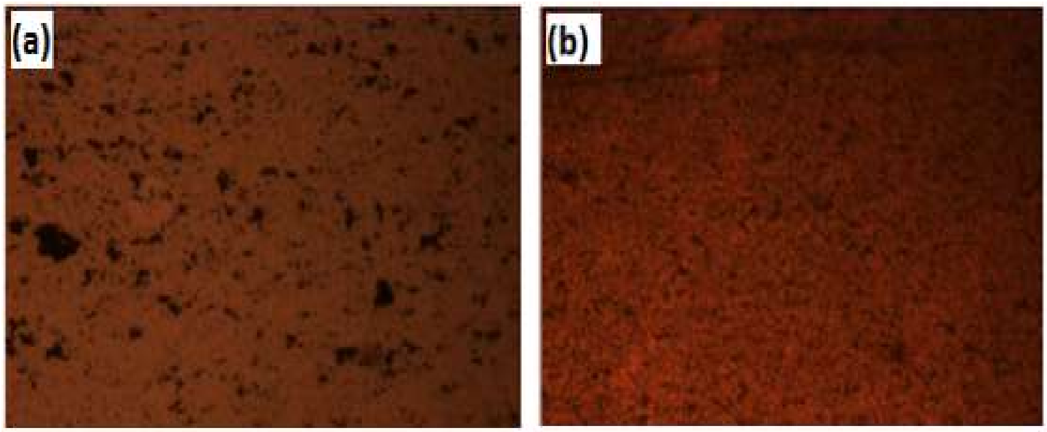

Fig. 1 Micrographs of FeCuS thin Films at pH of (a) 7.9 and (b) 9.0 [17]

B. Successive ionic layer absorption and reaction

The SILAR is a comparatively simple and less examined chemical thin film deposition technique. Ions are the main building blocks and employ complex ions-by- ions reactions on an appropriate substrate [27]. The SILAR system necessitates immersing a required substrate into different beakers containing well-prepared cationic and anionic precursors at a particular temperature. Connecting every immersion, the substrate is washed in water to prevent precipitation within the process [28, 29]. The immersion process could be computer-generated for any number of phases. Various deposition parameters such as nature of the chemical solution, concentration of precursors, the substrate used, pH values as well as the deposition temperature influence the growth system [30]. The technique comes with various benefits which consist of; (i) reduction of local heating that are harmful to films using physical methods (ii) the utilization of vacuum is not necessitated at any phase of the process, (iii) the process is viable at normal room temperature, (iv) substrates of complex surfaces are well-suited with the process, making SILAR suitable for large area depositions [31, 32].

Interestingly, unique optical and electrical properties of ternary Fe-Cu-S thin films can be achieved using the SILAR technique. In [19], Fe^{2+} induced opto-electrical properties of iron copper sulphide thin films were grown on soda-lime glass (SLG) substrates by controlling the molar concentrations of iron (0.7 M and 1.0 M) at deposition cycles of 20, 30 and 40. In this experiment, a typical ‘four-beaker-system’ was employed as indicated in Fig. 2. Ferrous nitrate, cuprous chloride, ammonia solution (to control the power of hydrogen), TEA and EDTA (complexing agents) were utilized as the cationic solution in beaker I. Thiourea was used as the anionic precursor in beaker III, and the beakers (II and IV) contained the distilled water. These preparative parameters as well as their molar concentrations are outlined in Table 1. The optical characterization via the Avantes UV-VIS Spectrophotometer in the wavelength range of 200 to 1000 nm indicated a variation in the transmittance and reflectance due to the Fe^{2+}concentration, along with a decrease in the band-gap energy from 3.84 to 3.61 eV with deposition cycles. These, they, reasoned may be owing to the introduction of impurities from the experimental environment, nature of the reagents used, in addition to the deposition duration [19].

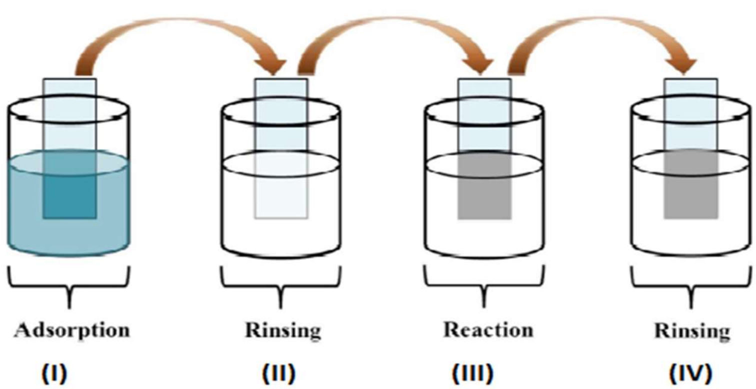

Fig. 2 Experimental set-up of SILAR technique [19].

From the electrical results determined using the four point probes (KEITHLEY 4ZA4 2400), the conductivity was found to increase with deposition cycles for Fe^{2+}molar concentrations. Thus, results obtained showed that the high absorbing and conducting Fe-Cu-S thin films are potential material for solar cell fabrications and coatings of various kinds [19].

Similarly, [20] investigated the influence of SILAR deposition cycles on the band-gaps and electrical properties of Fe-Cu-S thin films. The films were deposited onto (soda-lime glass) substrates at 300 K (Table I). The optical absorbance was observed to be high in the UV regions and ranged between 2.5 and 4.7, which decrease to below 1.0 in the near-infrared-regions of the electromagnetic spectra. The films also exhibited a band-gap (3.76, 3.51 and 3.42 eV) that decreases with deposition cycles. The average transmittance (above 80 %) and the enhanced electrical conductivity make the deposited films suitable for various optoelectronic applications [20].

The SILAR processes described in this section are often used by researchers in recent times to synthesize ternary thin films of Fe-Cu-S but come with its shortcomings, which make it difficult to fabricate consistent properties of the material. This includes wastages of precursors (chemicals) as well as the natural limitations connected with the deposition parameters and the boundary between the thin films and substrates.

III. Conclusion

This paper is aimed at examining the various methods exploited by researchers in fabricating FeCuS thin films and recommends the most suitable technique for its fabrication. The study largely focused on the chemical synthetic methods of chemical bath deposition or solution growth technique as well as the SILAR method. The effects of the various methods on the optical, electrical and structural properties of synthesized thin films were reviewed. In general, CBD was seen to be the simplest, cost-effective and most efficient method to prepare the Fe-Cu-S thin films which is the justification why it has been preferred by various authors in recent times. Extensively, this study x-rayed the various precursor materials which are used in synthesizing FeCuS thin films with variety of properties. Thus, CBD is the common suitable technique inferred and is recommended for future research study.

Furthermore, the effects of deposition parameters such as deposition time, pH, concentration and deposition cycles on the properties of prepared FeCuS thin films were also studied. Conclusively, if a decrease in the transition band gap energy is the priority; an increase in SILAR cycles for iron concentrations is preferred. Variation in SILAR cycles (20, 30 and 40 cycles) at room temperature have resulted in FeCuS thin films of high transmittance, excellent reflectance and low resistivity, which are ideal parameters for diverse technological applications, mainly coatings of various types and solar cells applications. From this review, research gaps were recognized, and it is recommended that future works should be performed to further improve the growth of FeCuS thin films using the physical deposition methods of sputtering and evaporation and compare results.

As most of the researchers only worked on the synthesis as well as the optical, electrical and structural characterizations, detailed characterizations including the Hall effect measurements, luminescence and photoconductivity measurements should be carried out to determine the films Hall coefficient, carriers types as well as mobility and concentrations, so as to identify its industrial applications. To improve the quality of the material for device applications, researchers should also investigate the effect of annealing temperatures on the films at various conditions. Since the authors suggested that the material is promising for various opto-electronic devices, future studies should focus on extending this research by fabricating any of the opto-electronic devices for industrial uses. The review has offered a promising pathway for future researchers to direct their efforts with respect to FeCuS thin films.

Acknowledgment

The authors express their appreciation to Novena University, Ogume, Delta State, Nigeria, and authors whose publications were used for writing this review study.

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