International Research Journal of Advanced Engineering and Science
ISSN (Online): 2455-9024

Characterization of SILAR Deposited Co_9Se_8 Films (trisodium citrate=complexing agent)

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Abstract**—** *The Co_9Se_8 films have been synthesized onto substrate (microscope glass slide) by adding complexing agent (trisodium citrate) via SILAR deposition method. Optical properties were studied and the band gap was observed to be 1 eV. XRD investigations indicated the polycrystalline structure with cubic phase. The grain size and film thickness were 0.25 µm and 1.8 µm, respectively based on the AFM images.*

Keywords**—** *Cobalt selenide; thin films; band gap; semiconductor*.

I. INTRODUCTION

Generally, thin films could be deposited onto various types of substrates including fluorine doped tin oxide [1], alumina substrate [2], silicon [3], mica [4], titanium [5], indium tin oxide coated glass [6] and microscope glass slide [7]. The film thickness ranges from a few nanometers to micrometers, showed very unique properties such as high absorption coefficient [8], excellent dielectric constant, excellent refractive index, and appropriate band gap value [9]. So far, thin films have been used in opto-electronic [10], electroluminescence devices [11], solar cells, sensor devices [12] and medical devices [13]. Up-to-date, there are several deposition techniques (physical method and chemical deposition technique) have been used to produce thin films as indicated in Table 1. It is noted that each deposition method has advantage and limitation as well. Therefore, selection of deposition method is very important to prepare thin films at significantly lower cost.

On the other hand, characterization of the obtained thin films was considered as very important part to study the properties of samples. Many researchers have characterized these films by using different tools including Field emission scanning electron microscopy (morphology studies), atomic force microscopy (topography investigation), X-ray diffractometry (structural properties), Fourier transform infrared spectroscopy [28] (identify unknown compound and chemical bonds), Raman spectroscopy (crystallinity, phase), reflection high-energy electron diffraction (surface of materials), X-ray photoelectron spectroscopy (composition), transmission electron microscopy [29] (morphology of the films), scanning probe microscopy (morphology of the samples), spectroscopic ellipsometry [30] (polarization of light) and energy Dispersive X-Ray Analysis [31] (compositional of the products).

As reported by many scientists, successive ionic layer adsorption and reaction (SILAR) technique has several advantages such as simple [32], large area deposition at low temperature [33] and inexpensive technique [34]. During the formation of films, the cleaned glass substrate was immersed into two beakers, containing cationic and anionic precursor, respectively [35]. Then, the substrate was rinsed with distilled water to eliminate undesired contaminants onto the substrate surface [36].

TABLE 1: The advantages and disadvantages of different deposition techniques

Deposition technique	Advantages	Disadvantages
spray pyrolysis	Low-cost, open atmosphere process and high rate of production [14]	Very complicated process and the percentage of yield is very low
electron beam evaporation	Multiple thin film could be deposited due to ability to rotate some materials into the path of electrons [15]	The filament degradation caused non-uniform evaporating rate
Electrodeposition method	High deposition rate, low cost [16]	Unsuitable for the very large scale production
chemical bath deposition method	Very simple deposition technique. It needs substrate and solution container	Wastage of solution after completion of each deposition process [17]
Atomic layer deposition	High quality films could be produced at low temperature	Very high energy waste rate [18]
Chemical vapour deposition	Very high deposition rate and can produce thick coatings [19]	Very high temperature is requited
molecular beam epitaxy	Can produce high purity and epitaxial materials [20]	Very expensive, and complex process
radio frequency sputtering	It could be operated with insulating targets [21]	Deposition rate is very low and expensive technique
Electrophoretic deposition	Rapid deposition process	High cost of precursors [22]
Sol gel method	The samples could be prepared at low temperatures showed high adhesive strength [23]	High cost of the raw materials
Ion beam deposition	Uniform coating thickness could be observed [24]	Expensive method and deposition rate was very low
Pulsed laser deposition	Dense and porous coating [25]	Expensive technique
Hydrothermal method	Can control size, crystallinity of the product, and the shape distribution	Very expensive autoclaves and faced safety issues [26]
Magnetron sputtering	High adhesion and uniform coating thickness could be obtained	Low deposition rate and very expensive method [27]

Ho Soonmin, "Characterization of SILAR Deposited Co_9Se_8 Films (trisodium citrate=complexing agent)," *International Research Journal of Advanced Engineering and Science*, Volume 7, Issue 2, pp. 335-339, 2022.

International Research Journal of Advanced Engineering and Science
ISSN (Online): 2455-9024

The main objective of this work is to prepare cobalt selenide thin films via SILAR method in the presence of trisodium citrate. The morphological, optical and structural properties of these films were reported for the first time. The trisodium citrate showed mildly tart flavor with the chemical formula ($Na_3C_6H5O_7$) as indicated in figure 1. General properties of trisodium citrate such as non-toxic substance, not irritant to the skin, and slightly irritant to the eyes.

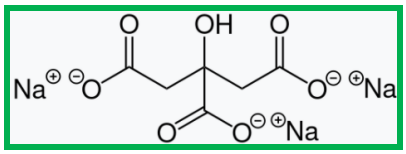


Fig. 1: The structural of trisodium citrate

II. Experimental

In this work, formation of cobalt selenide could be carried out by using cobalt (II) chloride hexahydrate ($CoCl_2.6H_2O$), trisodium citrate and sodium selenite (Na_2O_3Se). The microscope glass slide was washed with acetone and deionized water in order to remove undesired contaminants on the substrate surface. Deposition process was done by using SILAR method at room temperature with 18 deposition cycles. Firstly, cleaned microscope glass slide was immersed into beaker containing 0.2M of Co^{2+} ion and 0.15M of trisodium citrate (35 seconds). Following that, rinsing with the de-ionized water (10 seconds). Next, the substrate was placed in beaker containing 0.2M of Se^{2-} ions and 0.15M of trisodium citrate (35 seconds). Lastly, rinsing with de-ionized water again (10 seconds). Characterization of the obtained films was studied by using XRD, AFM and UV-visible spectrophotometer as indicated in Table 2.

TABLE 2: Characterization techniques employed in this work

Tool	Brand name /manufacturer	Purposes
XRD	LabX-6000 (Shimadzu)	Study the structure of the samples
AFM	QUESANT-USA (Bruker)	Investigate the morphology
UV-visible spectrophotometer	UV/Vis Lambda 35 (Perkin Elmer)	optical properties and band gap value of the sample

III. Results and Discussion

Preparation of thin films by using complexing agent has been reported by many scientists. These complexing agents including hexamethylene tetramine [37], hydrazine hydrate [38], tri-sodium citrate, ethylenediamine tetra acetate acid, triethanolamine [39], ammonia [40], ammonium sulphate [41], Sodium citrate, sodium tartrate, ammonium nitrate [42], tartaric acid [43], nitrilotriacetic acid, hexamine, citric acid [44], diethanolamine, oxalic acid [45] and tetramethyl ammonium hydroxide [46]. Research findings showed that the properties of thin films strongly depended on the use of the complexing agent during the formation of films.

The X-ray diffractomer was equipped with CuKα (λ=1.5406 Å) radiation sources. The scan area and step size values were 10° to 90° and 0.02° (2θ), respectively. The figure 2 showed the X-ray diffraction pattern of SILAR deposited thin films. Several diffraction peaks corresponding to 13.3°, 18.9°, 23.4°, 28.3°, 55.1° and 71.6° were observed. The XRD analysis confirmed polycrystalline in nature with cubic Co_9Se_8 phase. As indicated in Table 3, the observed d-spacing values (from experimental data) were matched with standard d-spacing data (JCPDS: 98-004-4857).

TABLE 3: Compare the observed and standard d-spacing values (SILAR deposited cobalt selenide thin films)

Observed position, 2 theta (°)	Observed d-spacing data (Å)	Standard d-spacing data (Å)	*Hkl* plane
13.3	6.1	6.0	111
18.9	5.1	5.2	002
23.4	3.8	3.7	022
28.3	3.1	3.1	113
55.1	1.6	1.6	026
71.6	1.3	1.3	008

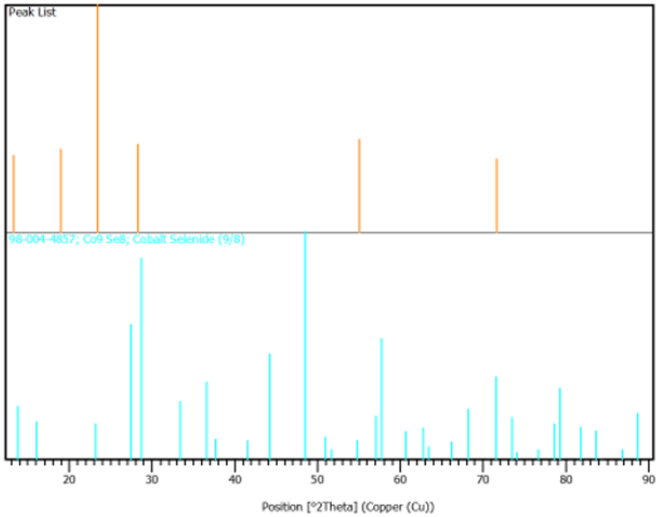
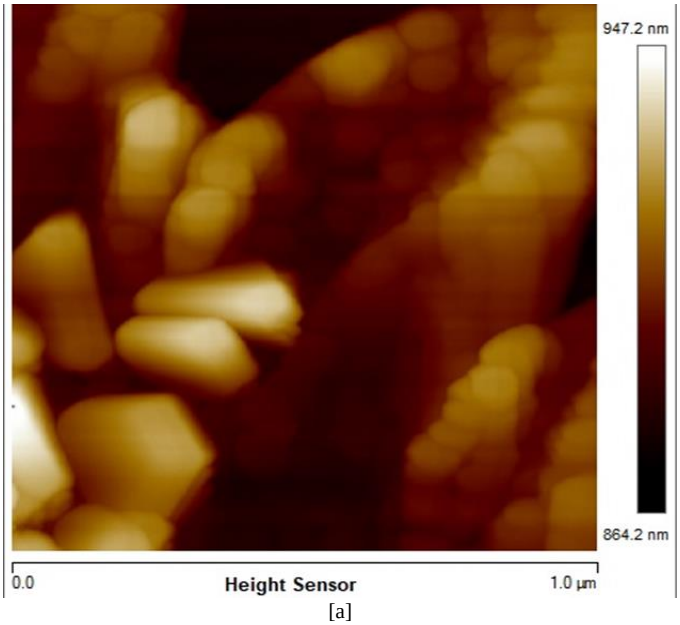


Fig. 2: XRD of the SILAR deposited thin films.



[a]

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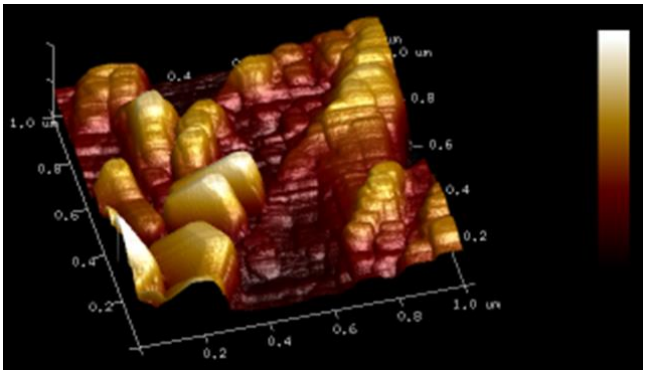

[b]

Fig. 3: Atomic force microscopy images in 2-dimensional [a] and 3-dimensional [b] of cobalt selenide thin films

The AFM technique was used to study morphology of the obtained films such as surface roughness, grain size and film thickness. The mode and cantilever (spring constants=0.4 N/m; resonance frequencies=70 kHz) were employed in this work so called Scanasyst peak force tapping and scanasyst-air, respectively. The atomic force microscopy images indicated the 2-dimensional (figure 3a) and 3-dimensional surfaces (figure 3b) of SILAR deposited thin films. The AFM analysis was conducted over 1µm X 1µm scanning range. Based on the AFM image, grain size (0.25µm), film thickness (1.8 µm) and surface roughness (0.01 µm) were highlighted.

The UV-visible spectroscopy (range of 300 nm to 900 nm) is very useful technique to investigate optical properties. Higher absorption could be observed in shorter wavelength if compared to longer wavelength based on the figure 4. The obtained spectrum data could be used to calculate the band gap value by using Stern equation as reported by many researchers [47-49].

$$A = \frac{[k(hv - E_g)^{n/2}}{hv} \quad [1]$$

In this equation, several symbols such as *v,h, k represented* frequency value, Planck's constant value, and constant value, respectively. Meanwhile, the *n* value could be contributed to the value of either 1 (direct gap) or 4 (indirect gap). The graph was plotted ($(Ahv)^2$ against *hv)* based on the data collected from UV-visible spectrum. The extrapolation of this straight line will generate the band gap about 1 eV (figure 5).

There are many thin films have been produced successfully by adding trisodium citrate as complexing agent. Jun and co-workers [50] have reported that stronger complexation ability could be seen in chemical bath deposited ZnS films. Agawane and co-workers [51] highlighted that grain size reduced, however improved the uniformity of chemical bath deposited ZnS films with an increase in complexing agent concentration. Offor and co-workers [52] described the sprayed ZnS films were cubic phase, crystallite size was 20.45 nm. Gustavo and co-workers [53] proposed the best electrodeposited Cu_2ZnSnS_4 films were produced by using 0.2M complexing agent. These films showed excellent uniformity and the band gap was 1.52 eV, suitable for solar cell applications. Jeon and co-workers [54] pointed out that good stoichiometric electrodeposited Cu_2ZnSnS_4 films could be produced by using this complexing agent, which served as absorber layer for solar cell devices.

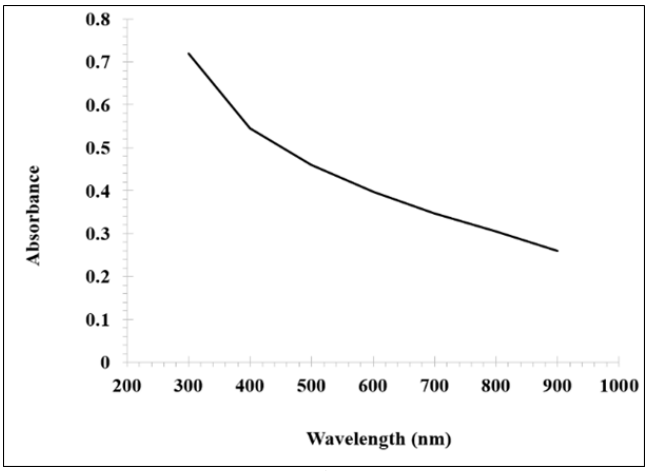

Fig. 4: Optical absorbance spectrum of SILAR deposited cobalt selenide thin films

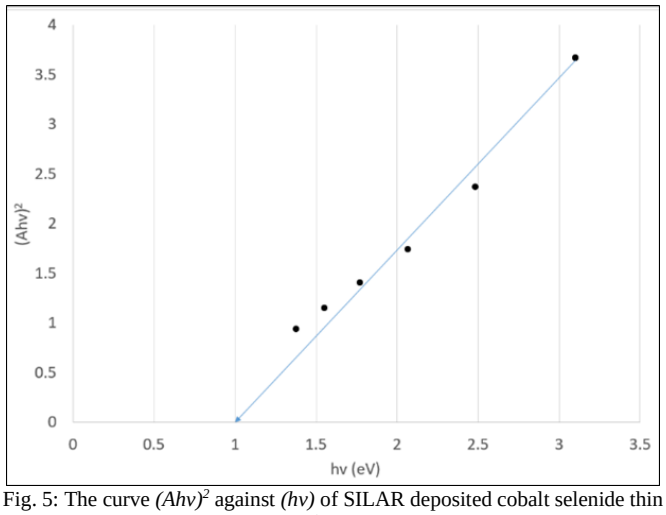

Fig. 5: The curve $(Ahv)^2$ against *(hv)* of SILAR deposited cobalt selenide thin films

IV. Conclusions

The Co_9Se_8 films growth onto microscope glass slide through SILAR technique (complexing agent=trisodium citrate). XRD studies confirmed polycrystalline in nature with cubic phase. The obtained grain size, film thickness and band gap were 0.25µm, 1.8 µm and 1 eV, respectively.

Acknowledgment

This research work was supported by INTI International University under INTI Internal Research Grant INTI –CAE-01-01-2018.

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