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Effect of thiourea on structure, morphology, and optical properties of spray deposited CZTS thin films for solar cell applications

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ABSTRACT

Cu_2ZnSnS_4 (CZTS) films of different Thiourea ($SC(NH_2)_2$) molarity were deposited by using a simple chemical spray technique at substrate temperature 275 °C. Analytical reagent Grade Copper chloride ($CuCl_2$), Zinc chloride ($ZnCl_2$), and Tin chloride ($SnCl_4.5H_2O$) were used as Cu^+, Zn^+, and Sn^+ ion sources respectively and thiourea ($SC(NH_2)_2$) (0.02, 0.04, 0.06, 0.08, and 0.1 M) was used as an S^- ion source. A set of five CZTS films was deposited using five different molarity of thiourea. The structure, morphology, elemental analysis, and optical properties of these films were studied using X-ray diffractometer (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX), and UV-visible spectroscopy techniques respectively. The XRD spectra showed that all films are polycrystalline tetragonal structures with preferential orientation along 112 planes. The calculated crystallite size was increased with an increase in thiourea concentration. Variations of optical band gap with thiourea molarity have been investigated using Tauc plots. SEM micrographs exhibit CZTS spherical granules regularly arranged with some void spaces. The purity of deposited films was investigated using EDX analysis. All the CZTS films exhibit a higher absorption coefficient ($\alpha>10^4$) cm^{-1} and a band gap in the reported range (1.2 to 1.53 eV) can be used as an absorber layer in solar cells.

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1. INTRODUCTION

Solar energy is one of the green and clean energy which is found to be an alternative and promising source of energy, to fulfill the present and future global energy demand. Solar cell devices or photovoltaic cells have been used for harvesting and converting the solar energy into usable form. Silicon-based solar cells are widely used solar cells, but it needs a very thick absorber layer. Thin film solar cells based on copper indium gallium sulfur (CIGS) [Cu_2 (InGa) S_4] and copper indium gallium selenide CIGSe [Cu_2 (InGa) Se_4] and cadmium telluride (CdTe) systems are well established systems of conversion efficiency up to 22% [1]-[3]. Latest report shows 11.1% and 12.6% conversion efficiency of CZTSe based solar cells [4]. However, Indium and gallium

elements are toxic, less abundance, and expensive which limits the large-scale production of device applications [5] and element Se is too toxic and expensive with CZTSe, whereas cadmium (Cd) is toxic to environment [6]. Therefore, researchers are much interested in copper Zinc tin sulfur Cu_2ZnSnS_4 (CZTS) eco-friendly thin film based solar cells. CZTS is quaternary p-type promising semiconductor has direct optical band gap about 1.2 to 1.5 eV, absorption coefficient $\geq 10^4$ cm^{-1} and of 9.6 % conversion efficiency is very similar to CIGS type thin film solar cells [7]-[10]. The elements used in fabrication of CZTS material are non-toxic, higher abundance and cheap in cost and ecofriendly. CZTS is therefore a suitable candidate to replace the CIGS as an absorber layer thin film solar cell device. Several techniques have been employed in the preparation of CZTS thin films, such as R F sputtering [11], Sol-Gel sulfurization method [12], pulsed laser deposition [13] chemical vapor deposition [14], elecrodeposition [15], and SILAR method [16], [17].

All of mentioned deposition techniques required sulfurization of deposited sample for stoichiometric CZTS phase formation. In this article we explore simple low-cost chemical spray pyrolysis (CSP) technique [18] using locally available perfume atomizer. Present CSP technique is simple, inexpensive, non vaccume and suitable for large area deposition with low chemical wastage. The novelty of this present technique is to achive the stoichiometry of CZTS phase formation without sulfurization process. In CSP method stoichiometry of CZTS is very sensitive to the concentrations of precursors in the spraying solutions. There are several reports on fabrication of CZTS thin films using CSP method [19]-[21]; however single report of Biplav Dahal et al. was reported on studies related to sulphur concentration on properties of CZTS and showed that the optical band gap was increased with increase in thiourea concentration [22]. Up to now, a record efficiency of 9.6% has been achieved but still improvements are essential to acchive high efficiency for industrial application. Therefore, in present paper we study influence of sulphur (S) concentration on structural, morphology, and optical properties of CZTS thin films prepared by present chemical spray pyrolysis (CSP) method using locally available perfume auomizer.

2. MATERIAL AND METHOD

Cu2ZnSnS4 (CZTS) thin films were deposited on simple soda lime glass substrates. Before deposition, the substrates were cleaned ultrasonically using acetone, ethanol and distilled water and dried by hair drier. To achive good quality CZTS thin film medium of precursor preparation plays vital role. Different aqueous and nonaqueous medium have been used for precursor preparation however Ethanol was found good in case of CZTS thin films using present CSP method. The precursor solution was obtained by mixing solutions of (0.04) M Copper chloride ($CuCl_2$), (0.02) M Zinc chloride ($ZnCl_2$), (0.02) M Stannic chloride ($SnCl_4$ 5 (H_2O)) and (0.02, 0.04, 0.06, 0.08, and 0.1 M) thiourea ($SC(NH_2)_2$). The pure ethanol medium is used for all three cationic solutions and thiourea solution is prepared by using 50% ethanol to avoide the formation of tin hydroxyl $Sn(OH)_2$ ions so that free Sn^+ ions are sufficiently available for CZTS phase formation. The experiment shows that turbidity of mixture was depend on molarity of thiourea (or cation to anion ratio). The cation to anion ratio was displayed in Table 1.

The above prepared solutions were stirrer for one and half hour. Three cationic solutions were mixed together one by one with stirring continue and then thiourea was added slowly such that turbid mixture becomes clear homogenous precursor. This precursor was sprayed on substrate maintained at desire temperature on hot plate by locally available perfume atomizer. The temperature of hot plate is controlled by digital temperature controller. The distance of spraying nozzle form substrate was adjusted such that the whole surface of substrate was exposed. The spray rate 5 ml per second and substrate temperature 275 ^oC were kept fixed to study the effect of thiourea on characterizations of CZTS thin films [23]. After deposition the samples were annealed in mufel furnace at 275 ^oC. The annealed samples were characterized using XRD, SEM, and UV-Visible spectrophotometer.

3. RESULTS AND DISCUSSION

3.1. Physical characterizatiopn

The thickness (t) of the deposited CZTS films is calculated by using weight and difference method [10], given by relation (1).

$$t = \frac{m}{\rho A} \quad (1)$$

Where m (*gm*) is the deposited mass, ρ (gm/cm^3) is density of deposited material and *A* (cm^2) is the area of deposited film. The film thickness was shown in Table 1. Film thickness was increased with increase in thiorea concentration. Thickness of the CZTS films was in the range of 266-289 µm. The maximum thickness 289 µm of CZTS film for 0.08 M thiourea was to be obtained however beyond 0.08 M concentration thickness of film sample (CZTS5) was to be decreased.

Table 1. Represents the film composition, cation to anion ratio and film thickness

Film Sample	Cationic Molarity	Sulfur ion Molarity	Cation/Anion Ratio	Film Thickness (µm)
CZTS1	0.08	0.02	0.04	266
CZTS2	0.08	0.04	0.02	268
CZTS3	0.08	0.06	1.33	271
CZTS4	0.08	0.08	1.0	289
CZTS5	0.08	0.1	0.8	269

3.2. Structure of CZTS thin films

XRD spectra were recorded in the range 20º to 80º and presented in Figure 1. XRD patterns for all films show Bragg's reflections at (2θ~28.35º, 32.06 º, 47.23°, and 56.98°) which are assigned to (112), (200), (220), and (312) lattice planes respectively. These values are in good agreement with the standard JCPDS card number (26-0575) which represents the polycrystalline tetragonal Kieserite crystal structure of CZTS material. The obtained XRD patterns are in good agreement with the pattern reported by Deokate *et al*. which confirm the purity of the CZTS films. They have been reported the pure and good quality CZTS thin films exhibited the crystal strcture with absence of the impurity peaks and secondary phases confirmed by Raman analysis [24], The Figure 1 showed that intensity of (112), (220), and (312) peaks gradually increased with increase in thiourea molarity, however for CZTS5 sample height of (112), (220), and (312) peaks was decreased as compared to CZTS4 sample. This concluded that crystallinity of CZTS films were depending on sulfur ion concentration.

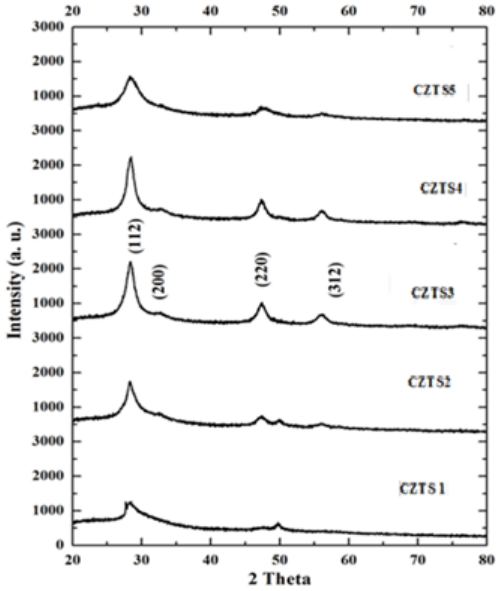


Figure 1. XRD spectra of CZTS thin flms

The full width at half maximum (FWHM) of (112) preferential orientation was decreased with increase in thiorea molarity. The crystallite size was calculated from following Suscherrer's relation (2) using FWHM (β) corresponding to (112) orientation [25].

$$D = \frac{0.94\,\lambda}{\beta \cos\theta} \quad (2)$$

Where, λ is wavelength of X-ray (0.1504 nm), β full width at half maximum and θ is Brag's angle (degree). The FWHM and crystallite size were displayed in Table 2. The crystallite size was increased with increase in thiourea molarity or sulfur ion concentration. However, the intensity of (112) peak, FWHM and crystallite size show odd behavior as thiourea molarity was increased above 0.08 M. Thus, the optimum value of thourea molarity or sulfur ion concentration plays important role to maintain the stoichiometry of CZTS films. It is reported that when the thiourea concentration (sulfur ion content) of the composition increases, the mobility of the atoms located on the surface increases, so these atoms can rearrange their positions to occupy more stable sites. This process controls the favorite growth orientation of the CZTS crystal structures imilar have been reported by Bakr *et al*. [26].

Table 2. Showing XRD data and grain size D (nm)

Film Sample	2θ (degree)	d (degree)	FWHM β (radians)	Millar Indices (hkl)	D (nm)
CZTS1	28.352	3.108	3.610	(112)	24
CZTS2	28.3757	3.139	2.569	(112)	33.5
CZTS3	28.4148	3.0723	2.302	(112)	37
CZTS4	28.5992	3.114	2.304	(112)	37.15
CZTS5	28.6314	3.139	4.592	(112)	20.47

3.3. Surface morphlogy ogf CZTS3 film

Scanning electron micrograph (SEM) of Sample CZTS3 was presented in Figure 2. Sample is scanned at 5000 magnifications. SEM image showed that whole surface is uniformly coated over which spherical granules of CZTS material schematically arranged with some void spaces. The average grain size 1.244 μm is obtained by using linear intercept method. The average grain size is larger than crystallite size obtained from XRD data. The increase of grain size is due to agglomeration of CZTS nano crystals.

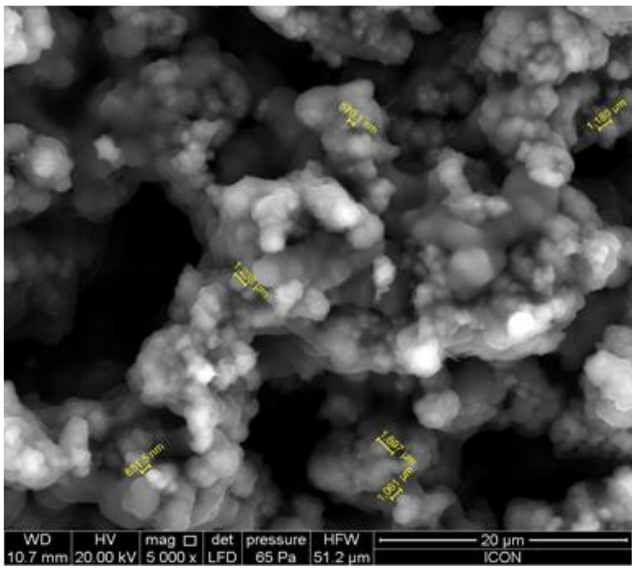


Figure 2. SEM micrograph of CZTS thin films

3.4. Elemental analysis of CTZS3 sample

Elemental analysis of CZTS sample was carried out using energy-dispersive X-ray analysis (EDX). The Figure 3 shows the EDAX spectra. The Figure 3 showed that all the peak corresponding to Cu, Zn, Sn, and S elements. Therefore, prepared sample composed of Copper, Zinc, Tin, and Sulfur elements. The initial and final atomic % was shown in Table 3. Initial and final and atomic % confirms the Cu_2ZnSnS_4 phase formation. Initial and final atomic precent shows that the prepared CZTS film was stiochiomertric.

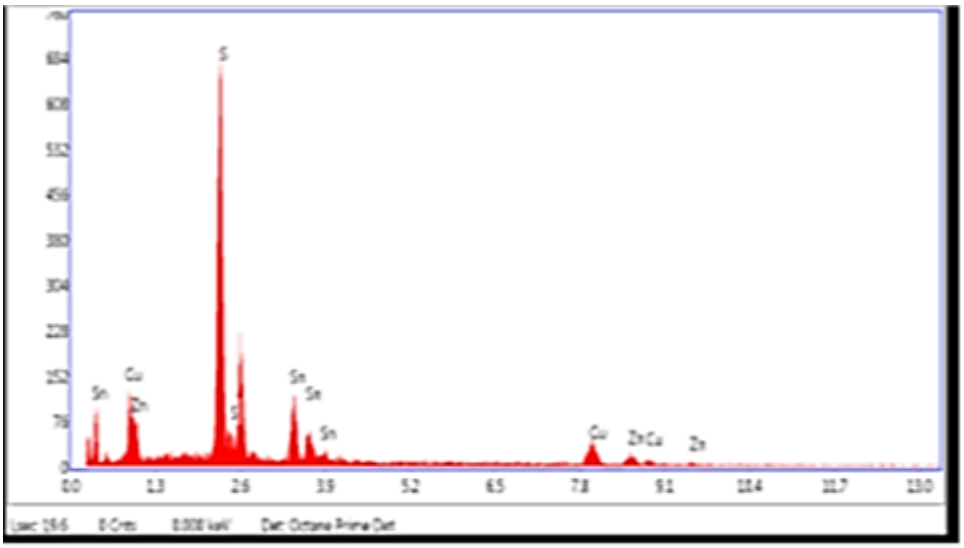


Figure 3. EDX spectra of CZTS thin films

Table 3. Initial and final weight and atomic percentage of elements

Element	Initial		Final		Grain size (μm)
	Weight %	Atomic %	Weight %	Atomic %	
S K	29.04	50.00	28.67	49.22	1.244
SnL	28.03	12.50	26.41	12.25	
CuK	26.86	25.00	28.67	24.84	
ZnK	16.07	12.50	16.25	13.69	
Total	100	100	100	100	

3.5. Optical absorption and band gap of CZTS thin films

Optical absorption spectra of CZTS films were recorded in spectral range of (350–999) nm by using Systronics (2201) double beam UV–visible spectrophotometer. The variation of optical absorption with wavelength (nm) was shown in Figure 4. It was noticed that the optical absorption for all CZTS films decreased exponentially as wavelength increased in the range 350-800 nm, and then increases slowly at higher wavelengths. The absorption was observed decreased with increase in thiourea concentration up to 0.08 M and above it shows abrupt increase. The absorption curves of sample CZTS1 and CZTS5 show similar nature. All the films show higher absorption coefficient ($\alpha > 10^4$) cm^{-1} in the visible and near infrared region of electromagnetic spectrum which favors increase of probability of direct transition. Similar results have been predicted in other reports [24], [27]. The deposited CZTS films exhibits higher absorption coefficient ($> 10^4$) cm^{-1} may be used in solar cell device applications. The optical band gap is obtained by using Tauc (3) [28].

$$\alpha h\nu = A(h\nu - E_g)^n \quad (3)$$

Where (E_g) the optical energy gap of the film, (A) is a constant, ($h\nu$) is the incident photon energy and n is a numeric value equal to (1/2) for allowed direct transition. The optical band gap was estimated by using Tauc's plots was as shown in Figure 5 for CZTS thin films, where a graph of $(\alpha h\nu)^2$ versus ($h\nu$) is plotted and the extrapolating linear portion of the curve gives the value of the direct optical band gap of the deposited CZTS films [20]. The band gaps of CZTS1, CZTS2, CZTS3, CZTS4, and CZTS5 obtained respectively were 1. 2, 1.52, 1.50, 1.275, and1.53 eV. It is clear that the optical band gap of all CZTS films lies in the range 1.2 to 1.53 eV similar findings have been reported in the earlier literature [29].

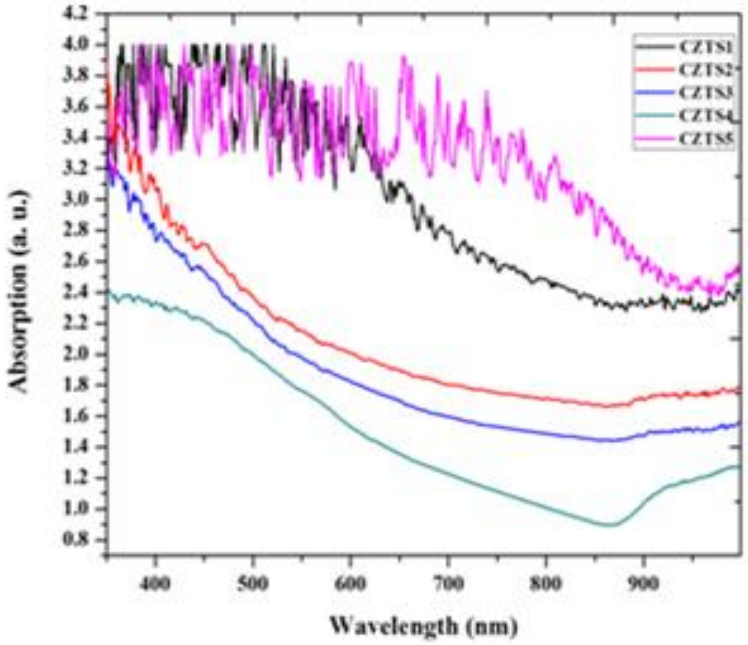


Figure 4. Absorption spectra of CZTS thin films

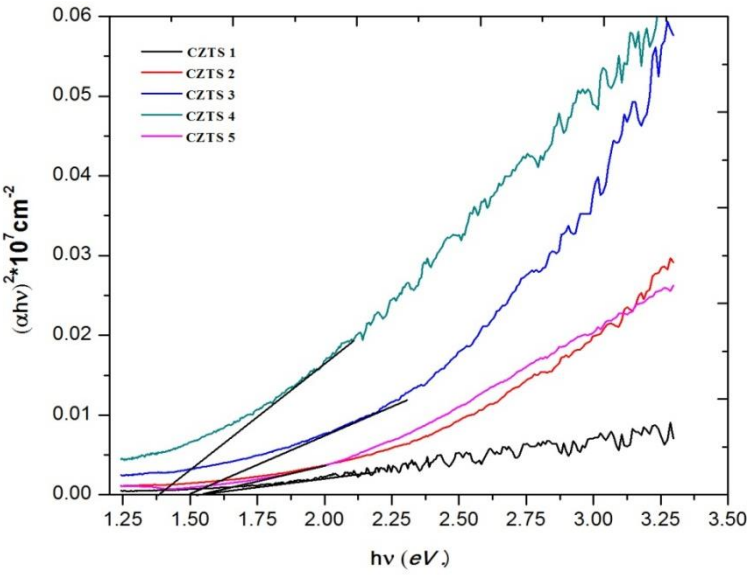


Figure 5. Tauc's plot of CZTS thin films

4. CONCLUSION

CZTS thin films were deposited simple soda lime glass substrates by chemical spray pyrolysis technique using locally available perfume atomizer. The XRD results showed that the prepared films are polycrystalline having tetragonal Kieserite crystal structure. The overall results and discussion concluded that optimum value of thiourea molarity plays important role in CZTS phase formation. However, CZTS4 sample of 0.8 M thiourea molarity exhibits good crystallity, maximum grain size 37.5 nm and 1.275 eV band gap. Hence it is concluded that CZTS4 is to be optimized sample with present reagent combination, represent a good candidate as an absorber layer in solar cells.

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