

Extensive Study of Spin Coated CZTS Thin Film Absorbers for Photovoltaic Utilization

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Abstract: In this study, we used the sol - gel spin coating method to synthesize $\text{Cu}_2\text{ZnSnS}_4$ (CZTS) absorber layers on common glass substrates. We investigated the morphological, optical, and structural characteristics of thin films. Various characterization techniques, including x - ray diffraction (XRD), Raman spectra, scanning electron microscopy (SEM), atomic force microscopy (AFM), and UV - visible spectroscopy, have been used to investigate these films. The XRD analysis revealed a kesterite phase with an intense peak at 28.35° representing the (112) plane. These samples have crystallite size around 5 nm, and lattice parameters $a = b = 5.2 \text{ \AA}$ and $c = 8.5 \text{ \AA}$. A single strong peak (at 329 cm^{-1}) in the Raman spectrum verifies the development of a single - phase CZTS thin film. The SEM image depicts the homogeneous, compact, and dense surface morphological thin films. The AFM 3D micrographs show spike - like structures with average and root mean square (RMS) roughness of 0.146 nm and 0.203 nm. UV - Visible spectroscopy revealed the direct band gap energy of around 1.52 eV for CZTS thin films. With these characteristics, the fabricated CZTS films are suitable for absorber layers in photovoltaic utilization.

Keywords: Sol - gel, spin coating, kesterite, single phase, band gap

1. Introduction

Energy utilization is accelerated by the growing need for energy. The utilization of photovoltaic cells, which produce power from an endless source, is the most environmentally friendly and efficient option to address this requirement. Thin film technology, particularly in the field of photovoltaic has garnered a lot of interest as a solution to the problems because of their peculiar size dependant features and wide range of possible applications [1, 2]. Non - traditional energy, based on photovoltaic cells is advantageous for the large duration energy - providing chain. In recent years, maximum conversion efficiencies of 20.8 and 19.6% for lab - scale devices were achieved by thin film photovoltaic cells based on polycrystalline $\text{CuIn}_{1-x}\text{Ga}_x\text{Se}_2$ and CdTe absorbing materials, respectively. These technologies have also already entered the commercial manufacturing stage [3]. The eventual development of CIGS and CdTe - based photovoltaic cells is constrained by the lack of In and Te in the earth's crust [4]. Thus, the critical components for meeting future demands for clean energy are effective photovoltaic devices made of materials that are both non - hazardous and plentiful on the Earth [5]. In this context, CZTS is a promising candidate for absorber material having a kesterite phase [6]. CZTS belongs to the p - type semiconducting material with a tunable direct energy band gap that varies between 1.4 and 1.6 eV [7, 8] with a significant absorption coefficient ($>10^4 \text{ cm}^{-1}$) [9]. By using zinc (Zn) and tin (Sn) components rather than indium (In) and gallium (Ga), it offers a less expensive and ecologically friendly alternative to $\text{CuIn}_x\text{Ga}_{1-x}\text{Se}_2$ (CIGS) and CuInS_2 (CIS) [10]. In this regard, the primary studies concentrate on the $\text{Cu}_2\text{ZnSnS}_4$ (CZTS) material, which has a pure kesterite phase. After the year 2000, the previously slow - moving investigations on CZTS - based solar cell systems accelerated [11]. Whereas initial samples were mainly

produced using vacuum technology, the next generation of effective CZTS absorbing layers was primarily accomplished using sol - gel techniques [12, 13]. The spin coating technique is one of the sol - gel technologies that are typically less expensive, requires minimal previous preparation, and is extremely viable. Furthermore, the spin coating approach is used to create the most effective photovoltaic devices with CZTS absorbing layers [14]. Although there was no deep parameter investigation for the spin - coated CZTS absorber layer development during these studies, many particular fabrication parameters, including annealing temperature [15], crystal arrangement [16], solution characteristics, layer count [17], and spinning rate [18], were altered and their impact was studied. Many researchers already looked into the synthesis of CZTS films using various vacuum as well as non - vacuum - based methods. [19, 20]. The last few years have seen extensive use of a variety of techniques, including chemical bath deposit [21], atomic beam sputtering [22], thermal evaporation [23], RF magnetron sputtering [24], co - evaporation, pulsed laser deposition [25], ion beam sputtering [26], photo - chemical deposition [27], electrochemical deposition [28], spray pyrolysis [29], spin coating [30, 31] and plasma polymerization [32] for the fabrication of thin films for PV applications. Nevertheless, spin coating is now the most widely used method for creating homogeneous thin films of semiconducting materials with thicknesses in the micrometer and nanometer range.

2. Literature Survey

Ozidal et al. [33] reported that the chemical composition, particle size, surface morphology, crystal structure, and thickness of thin films were examined concerning the impacts of spin velocity, solution molarity, stabilizer

quantity, drying, and additional annealing conditions. Heat treatment has a significant impact on the synthesized CZTS thin film's quality. To achieve the best heat treatment results, a variety of drying, and annealing environments were applied. It has been discovered that stoichiometric CZTS absorbing thin layers may be synthesized in under-regulated and advantageous annealing environments. Islam et al. [34] reported that CZTS thin films have been created on glass slides using spin coating techniques at a variety of speeds. Using a variety of characterisation techniques the produced samples' optical, structural, and morphological characteristics have been investigated. For CZTS - 1 and CZTS - 2 samples, the energy band gap has been identified to be 1.50 eV and 1.45 eV, respectively. Urbach energy and steepness parameters for the CZTS - 1 sample are 0.99 eV and 0.026, while for the CZTS - 2 sample 0.95 eV and 0.027, respectively. According to the XRD analysis, the dominant peak for both films was located at (112), which corresponds to a 2θ value of 28.437° . The texture architecture for CZTS thin films was identified using SEM investigation. Ziti et al. [35] investigated how the annealing temperature affects the structural, optical, and electrical characteristics of $\text{Cu}_2\text{ZnSnS}_4$ thin film formed by the sol-gel process and fabricated through the spin coating method. These specimens were annealed in the air for 10 minutes at 300°C , 325°C , and 350°C temperatures. The development of the kesterite phase of CZTS is confirmed by Raman and X-ray diffraction spectra. Based on the transmission and absorption data provided by the UV-Visible spectrometer (in the 350 to 1000 nm range), the optical characteristics have been determined. The energy band gap value varies between 1.5 and 1.63 eV, and the four-probe technique revealed improved electric resistivity from 0.1 to 0.15 ($\Omega \cdot \text{cm}$). These synthesized films are a useful material for an absorbing layer in photovoltaic devices due to their superior resistivity and ideal energy band gap value.

3. Problem Definition

In this study, we have synthesized the $\text{Cu}_2\text{ZnSnS}_4$ (CZTS) thin films using sol-gel spin coating technique, on the glass substrates. The optical, structural, surface morphological, and surface topographical characteristics of these films have identified.

4. Methodology

4.1 Preparation of a sol-gel precursor

In the beginning, 0.02 M of ZnCl_2 and 0.04 M of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ have been dissolved in 50 mL of 2-Methoxy ethanol, and the mixture was heated to 50°C on a hot plate with a magnetic stirrer for 30 minutes. Following the addition of 0.02 M of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, 0.16 M of thiourea ($\text{SC}(\text{NH}_2)_2$), and 30 drops of monoethanolamine (MEA) to the abovementioned solution. A clear yellow solution was

obtained after an hour of magnetic stirring at 50°C on a hot plate.

4.2 Fabrication of CZTS thin film

Initially, the glass slides were cleaned with distilled water, isopropyl alcohol, and acetone respectively. The thin film deposited glass slides were then placed on a heated plate for 4 minutes at 105°C after being spin-coated with the precursor solution at 2000 revolutions per minute. This technique was carried out three to four times to produce thin films of the desired thickness. Finally, these synthesized films were annealed in the air for 10 minutes at 350°C .

4.3 Characterization Techniques

The CZTS thin films were analysed using various techniques. An X-ray diffraction spectrometer (XRD; Panalytical X Pert Pro using $\text{CuK}\alpha$ radiation) was applied to determine the crystallinity of thin films. A UV-Vis spectrometer (LAMBDA 750 Perkin Elmer) was used to study the optical characteristics of $\text{Cu}_2\text{ZnSnS}_4$ thin films in the 200 - 800 nm wavelength range. Raman spectroscopy (STR 500 CONFOCAL MICRO) was used to get the Raman spectra at ambient temperature. Surface morphology was analyzed using a ZEISS scanning electron microscopy (SEM). The surface topography was determined using Bruker's atomic force microscopy (AFM).

5. Results and Discussion

5.1 Structural characteristics

Fig.1 (a) depicts the XRD spectrum of synthesized CZTS thin films. The diffraction peaks occur in the vicinity of angles 2θ equal to 28.35° , and 49.0° which are in agreement with diffraction at the planes (112), and (220) respectively. The presence of these peaks confirms the formation of a kesterite phase having a tetragonal structure. This is supported by the JCPDS # 26 - 0575 card [36]. It demonstrates that no further secondary sulphide compounds were identified in the prepared thin film.

The Debye-Scherrer formula -

$$D = \frac{0.9\lambda}{\beta \cos\theta} \dots \dots \dots (1)$$

where $\lambda = 1.54 \text{ \AA}$ and β is the full width at half maximum (FWHM); may be applied to calculate the crystallite size (D) of the samples. The relation between d -spacing, miller indices ($h k l$) and lattice constants ($a=b, c$) for tetragonal geometry is given by -

$$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \dots \dots \dots (2)$$

which can be used to calculate the values of lattice parameters.

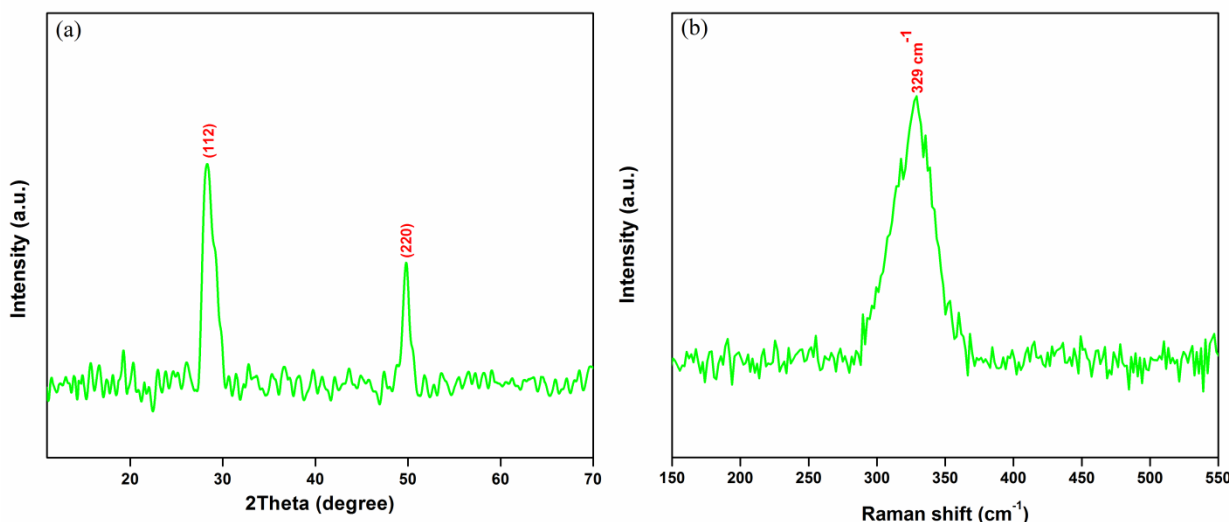


Figure 1: (a) XRD spectra of Cu₂ZnSnS₄ film, and (b) Raman spectra of Cu₂ZnSnS₄ film

The tetragonal distortion (Δ) and dislocation density (δ) of synthesized CZTS thin films can be calculated using the formulas-

$$\Delta = 2 - \frac{c}{a} \dots \dots \dots (3)$$

$$\text{and } \delta = \frac{1}{D^2} \dots \dots \dots (4)$$

The calculated values of FWHM (β), crystallite size (D), inter - planar spacing (d), lattice constants ($a=b, c$), tetragonal distortion (Δ) and dislocation density (δ) of synthesized thin films are shown in Table 1.

Table 1: The values of $\beta, D, d, a=b, c, \Delta$, and δ for CZTS thin film sample.

Sample	$\beta_{(112)}$ (deg.)	D (nm)	d (Å)	$a=b$ (Å)	c (Å)	Δ	δ (nm ⁻²)
CZTS	1.49	5.5	1.8	5.2	8.5	0.36	0.04

For identifying the phase of CZTS, a Raman spectrometer is an effective technique. Fig.1 (b) shows the Raman spectrum of the synthesized CZTS thin film sample. It contains a single intense peak at around 329 cm⁻¹ which belongs to the A vibrational mode of CZTS [37]. The Raman spectrum does not contain any extra peak corresponding to additional sulphide compounds. Thus, the existence of a single intense peak verifies the formation of a single - phase CZTS thin film [38].

5.2 Optical characteristics

Optical parameters including absorbance (A), transmittance (T), and band gap (E_g) are crucial for choosing a good absorber material for thin film photovoltaic. The optical properties of CZTS films were investigated using an ultraviolet - visible spectrophotometer. Fig 2 (a) and 2 (b) represents the graphs between absorbance vs wavelength and % transmittance vs wavelength for CZTS samples, respectively.

One may use the following relation, to determine the thin film's absorption coefficient (α) [39] –

$$\alpha = 2.303 \frac{A}{t}$$

where A and t indicate the absorbance and thickness of the thin film, respectively. The coefficient of absorption of the thin films, which was determined from the transmittance spectrum, turned out to be 10⁴ cm⁻¹ in the visible range. This larger value shows that these thin films may absorb the majority of the incident light. The optical band gap value of thin film samples can be determined using the Tauc formula [40] –

$$(\alpha h\nu)^2 = \beta (h\nu - E_g)$$

where β and E_g denote energy independent constant and energy band gap, respectively.

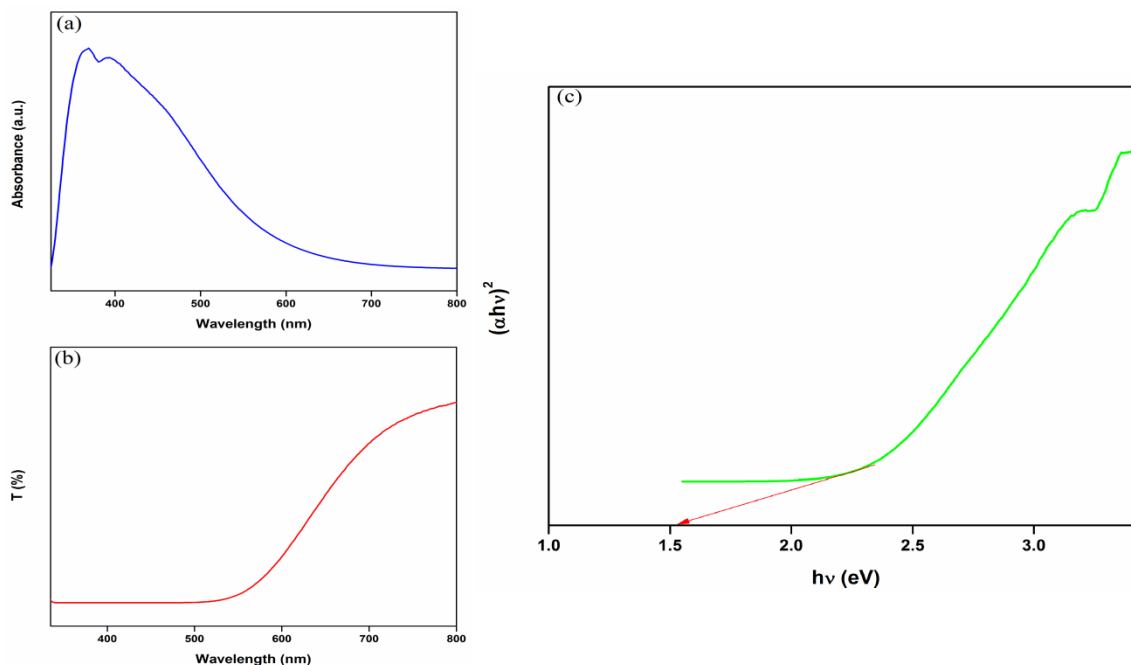


Figure 2: (a) Absorbance vs. wavelength, (b) Transmittance vs. wavelength, and (c) $(\alpha hv)^2$ vs. (hv) graph of CZTS thin film.

Fig 3 (c) displays the graph between hv and $(\alpha hv)^2$ for CZTS thin film sample. The band gap value (E_g) of the sample is given by the intersection of the tangent drawn to the graph between hv and $(\alpha hv)^2$, and the hv axis. The estimated value of the energy band gap from the aforementioned graph is around 1.52 eV . This band gap value is considered ideal for PV cells [41]. These optical characteristics suggest that the synthesized CZTS films are perfect for photovoltaic applications.

5.3 Surface analysis

The surface morphological characteristics of thin films have been identified by scanning electron microscope (SEM). Fig.3 (a) displays the surface SEM images of the CZTS thin films. The entire region of the CZTS thin film has identical surface features. The observed value of the average grain size of these synthesized thin films is about 32 nm . Also, these samples have a compact structure with the absence of fractures. Consequently, the thin films' reported average crystalline size of 5.5 nm (from XRD analysis) points to their compact polycrystalline nature. Thus, the synthesized thin films may be a viable option for photovoltaic studies.

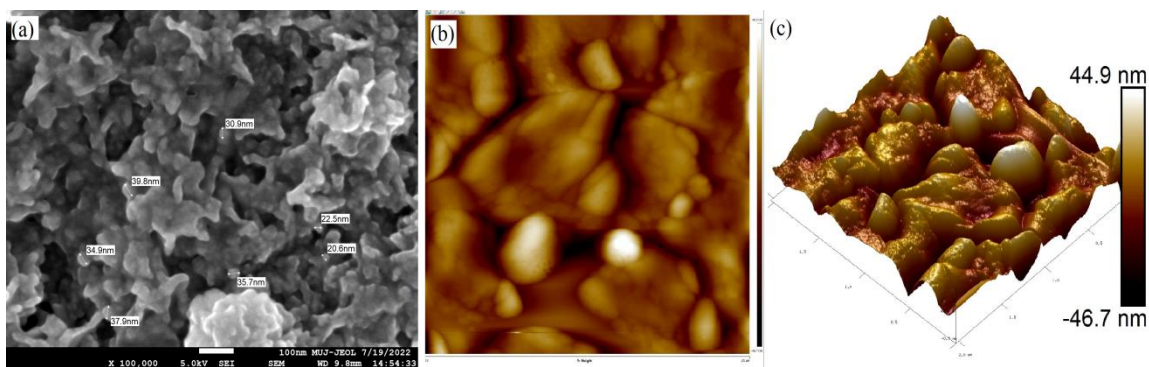


Figure 3: (a) SEM image, (b) 2D AFM micrograph, and (c) 3D AFM micrograph for $\text{Cu}_2\text{ZnSnS}_4$ thin film

The surface topography of synthesized thin films has been investigated by atomic force microscopy (AFM). Fig.3 (b) and 3 (c) display the 2D and 3D micrographs of the synthesized CZTS thin film samples, respectively. The 2D micrograph shows that CZTS thin film consists of almost spherical grains. The 3D micrograph shows a spike-like structure over the entire thin film surface with a vertical height between $+44.9 \text{ nm}$ and -46.7 nm . On the basis of AFM images, the average and root mean square (RMS) roughness have been estimated to be 0.146 nm and 0.203 nm , respectively.

6. Conclusion

In the research work, CZTS thin films were effectively synthesized by the sol-gel spin coating method on glass substrates. The XRD analysis revealed a kesterite phase with an intense peak at 28.35° representing the (112) plane. Raman spectrum displays a single intense peak at 329 cm^{-1} . The synthesis of the single-phase CZTS thin film is verified by the absence of additional Raman peaks. As a consequence, the Raman analysis of CZTS thin films validates the X-ray diffraction observations. The UV-

Visible spectroscopy revealed that the direct band gap energy of the thin film is around 1.52 eV, which is in the optimal band gap range for absorber material. The SEM image depicts the homogeneous, compact, and dense surface morphological thin films having grain sizes around 32 nm. The AFM images show that CZTS thin film contains almost flat surfaces. With these characteristics, the fabricated CZTS films are suitable for absorber layers in photovoltaic utilization.

7. Future Scope

The suggested sol - gel spin coating technique may provide an appropriate way to fabricate CZTS thin films with improved crystallinity and advantageous optical properties for solar applications. In the future, cheaper and easier synthesis process will be able to form non - polluting CZTS thin films, which may be utilized to fabricate effective thin film solar cells.

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