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Study of structural and electrical properties of $CuInSe_2$ thin films prepared by spray pyrolysis technique

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Abstract:
In this study, copper indium selenide ($CuInSe_2$) thin films were successfully prepared on glass substrate using the spray pyrolysis technique, and the structural and electrical properties of films were systematically investigated. The motivation for this research stems from the growing demand for efficient, low-cost absorber materials in thin-film photovoltaic and optoelectronic applications. The structural analysis using X-ray diffraction revealed that the prepared films possess a chalcopyrite crystalline phase (JCPDS card No. 40-1487) with a crystallite size of approximately 55.79 nm, indicating good crystallinity. Field Emission Scanning Electron Microscopy (FESEM) analysis showed that the surface morphology of the films is smooth and uniform, which is desirable for device fabrication. Energy-dispersive X-ray spectroscopy (EDS) confirmed the elemental composition, indicating the presence of copper (Cu), indium (In), and selenium (Se) without any secondary phases. Electrical characterization revealed that the films exhibit a resistivity of 4.93625 Ω·m for a film thickness of 44 nm. The temperature coefficient of resistance (TCR) was found to be negative (-0.00538 /°C), confirming the semiconducting nature of the films. Activation energy values were calculated as 0.2779 eV in the high-temperature region (HTR) and 0.0138 eV in the low-temperature region (LTR), suggesting thermally activated conduction mechanisms. These results demonstrate that spray-deposited $CuInSe_2$ thin films possess promising structural and electrical characteristics, making them suitable candidates for future optoelectronic and photovoltaic applications.

Keywords: Copper indium selenide, spray pyrolysis, conduction mechanisms, semiconducting nature, photovoltaic.

1. INTRODUCTION

Thin film technology is a highly versatile and widely adopted field in materials science that involves the deposition of extremely thin layers of material typically ranging from a few nanometers to several micrometers onto a substrate surface [1, 2]. These thin layers can be composed of metals, semiconductors, insulators, or organic materials, depending on the intended application. The significance of thin film technology lies in its ability to precisely tailor material properties such as electrical conductivity, optical transparency, magnetism, and surface reactivity [3, 4]. This precision makes it indispensable in a variety of industries, including microelectronics, photovoltaics, optics, sensors, and protective coatings. In the realm of

renewable energy, thin film solar cells such as those using $CuInSe_2$ offer advantages like reduced material consumption, lightweight and flexible form factors, and cost-effective large-area fabrication. Common deposition techniques include physical vapor deposition (PVD), chemical vapor deposition (CVD), sputtering, and solution-based methods like spray pyrolysis. As device miniaturization and energy efficiency become increasingly important, thin film technology continues to evolve as a vital enabler of next-generation electronic, photonic, and energy-harvesting systems [4-6].

Spray pyrolysis is a simple, cost-effective, and scalable thin-film deposition technique widely used for fabricating metal oxide and chalcogenide semiconductor films. In this method, a precursor solution containing metal salts or organometallic compounds is atomized into fine droplets using a spray nozzle and directed onto a heated substrate. As the droplets reach the hot substrate surface typically maintained at temperatures between 300°C and 500°C they undergo rapid evaporation and thermal decomposition, resulting in the formation of a solid thin film. The properties of the resulting film such as thickness, crystallinity, and surface morphology can be tailored by adjusting process parameters like spray rate, substrate temperature, carrier gas flow, and solution concentration [7, 8]. It is particularly advantageous because it does not require vacuum systems, can be performed in open air, and is well-suited for large-area coatings and multilayer structures. Its ability to produce uniform, adherent, and compositionally tunable films makes it an attractive technique for fabricating materials used in photovoltaics, gas sensors, optoelectronics, and transparent conductive coatings [6, 7].

Physicochemical and electrical properties of $CuInSe_2$ make it a highly attractive material for thin-film photovoltaic and optoelectronic applications. It is a ternary chalcogenide semiconductor that crystallizes in the chalcopyrite structure with a direct bandgap of approximately 1.04 eV, which is ideal for absorbing a broad spectrum of solar radiation. It exhibits high absorption coefficients ($>10^5$ cm^{-1}), allowing efficient light absorption in films as thin as a few micrometers. The material naturally exhibits p-type conductivity, primarily due to copper vacancies, and its electrical resistivity can range from 10^{-3} to 10^{0} Ω·cm depending on synthesis conditions and post-treatment. The thermally activated electrical behavior is often characterized by a negative temperature coefficient of resistance (TCR), indicating semiconductor-like conduction [9, 10]. It has good thermal and chemical stability under ambient conditions and a low toxicity profile compared to other semiconductors like CdTe, making it environmentally favorable. Its physicochemical robustness, high photoconversion efficiency, and ease of alloying with gallium or sulfur (to form $Cu(In,Ga)Se_2$ or $CuIn(S,Se)_2$) allow for bandgap tuning and further optimization for solar energy and infrared detection applications.

$CuInSe_2$ NPs have been extensively explored due to its remarkable optoelectronic and semiconducting properties. $CuInSe_2$ NPs could be synthesized using a variety of methods, each offering control over its structural, morphological, and electrical characteristics. Common synthesis techniques include spray pyrolysis, solvothermal and hydrothermal methods, hot-injection synthesis, chemical vapor deposition (CVD), and electrodeposition. Among these, spray pyrolysis is favored for its simplicity, cost-effectiveness, and ability to produce large-area thin films without requiring a vacuum. The resulting $CuInSe_2$ NPs or thin films typically exhibit a direct bandgap of ~1.04 eV, high absorption coefficients, and natural p-type conductivity due to copper vacancies. These properties make $CuInSe_2$ a highly suitable candidate for thin-film solar cells, where it serves as an efficient light-absorbing layer, particularly in $Cu(In,Ga)Se_2$ (CIGS) solar technologies [10-12]. Beyond photovoltaics, $CuInSe_2$ NPs is used in infrared photodetectors, photoelectrochemical (PEC) cells for water splitting, thermoelectric devices, and non-volatile memory applications. Its tunable optical properties, high thermal stability, and environmental compatibility continue to drive research into its integration in flexible electronics, gas sensors, and next-generation optoelectronic devices [12, 13].

The aim of the present study is to investigate the structural and electrical properties of $CuInSe_2$ thin films prepared using the spray pyrolysis technique, with a focus on understanding their suitability for optoelectronic and photovoltaic applications. By employing techniques like XRD, FESEM, EDS, and electrical measurements, the study aims to correlate the microstructural features with electrical performance metrics, such as resistivity, TCR, and activation energy in both high- and low-temperature regions.

2. EXPERIMENTAL WORK

In this study, high-purity AR (Analytical Reagent) grade chemicals were utilized for the preparation of $CuInSe_2$ thin films without any further purification, ensuring the consistency and reliability of the chemical composition. The thin films were deposited onto clean glass substrates using the spray pyrolysis technique, a cost-effective and scalable method well-suited for the fabrication of large-area thin films. In this method, stoichiometric amounts of high-purity copper, indium, and selenium salts were accurately weighed and dissolved in 100 mL of distilled water to prepare a homogeneous precursor solution. The mixture was thoroughly stirred to ensure complete dissolution of the salts. This solution was then loaded into a spray system equipped with a fine nozzle [14, 15]. During deposition, the precursor solution was atomized into fine droplets using compressed air as a carrier gas and directed toward a pre-cleaned glass substrate. The substrate was mounted on a heater, with the nozzle-to-substrate distance carefully fixed to maintain uniform spray coverage. The substrate was heated to an optimized temperature to facilitate the pyrolytic decomposition of the sprayed droplets upon contact, resulting in the formation of $CuInSe_2$ thin films. The spray flow rate was precisely maintained at approximately 0.5 mL/min to ensure controlled deposition. Key parameters such as spray rate, substrate temperature, solution concentration, and spray duration were optimized to obtain uniform, well-adherent films with desirable structural and electrical properties suitable for device applications [14-16].

3. RESULT AND DISCUSSION

The structural characterizations such as FESEM, EDAX and XRD were carried out at CIF, SPPU, Pune and SAIF Kochi while the electrical characterizations were carried out using half bride method. The electrical characterization of the $CuInSe_2$ thin films was carried out using the half-bridge method, a widely used technique for measuring the temperature-dependent resistance of semiconducting thin films. In this method, the film is connected in one arm of a Wheatstone bridge circuit, allowing precise measurement of resistance variations as a function of temperature. This setup is particularly effective in minimizing errors due to contact resistance and lead resistance, thereby ensuring accurate determination of the film's electrical behavior. The resistance values were recorded over a controlled temperature range, enabling the calculation of important parameters such as the temperature coefficient of resistance (TCR) and activation energy, which reveal the semiconducting nature of the material. The thickness of the $CuInSe_2$ thin films was estimated using the mass (or weight) difference method. In this approach, the weight of the substrate is measured before and after film deposition. The difference in mass, combined with the known area of the substrate and the density of the material [17, 18].

3.1 Structural characterizations

3.1.1 Field Emission Scanning Electron Microscopy (FESEM) Analysis

It was employed to examine the surface morphology and microstructural features of the $CuInSe_2$ thin films. Figure 1 shows the Field Emission Scanning Electron Microscopy image of the $CuInSe_2$ thin films at a high magnification of 200000×. The microstructure reveals a highly agglomerated morphology, composed of dense clusters of fine, nearly spherical nanoparticles.

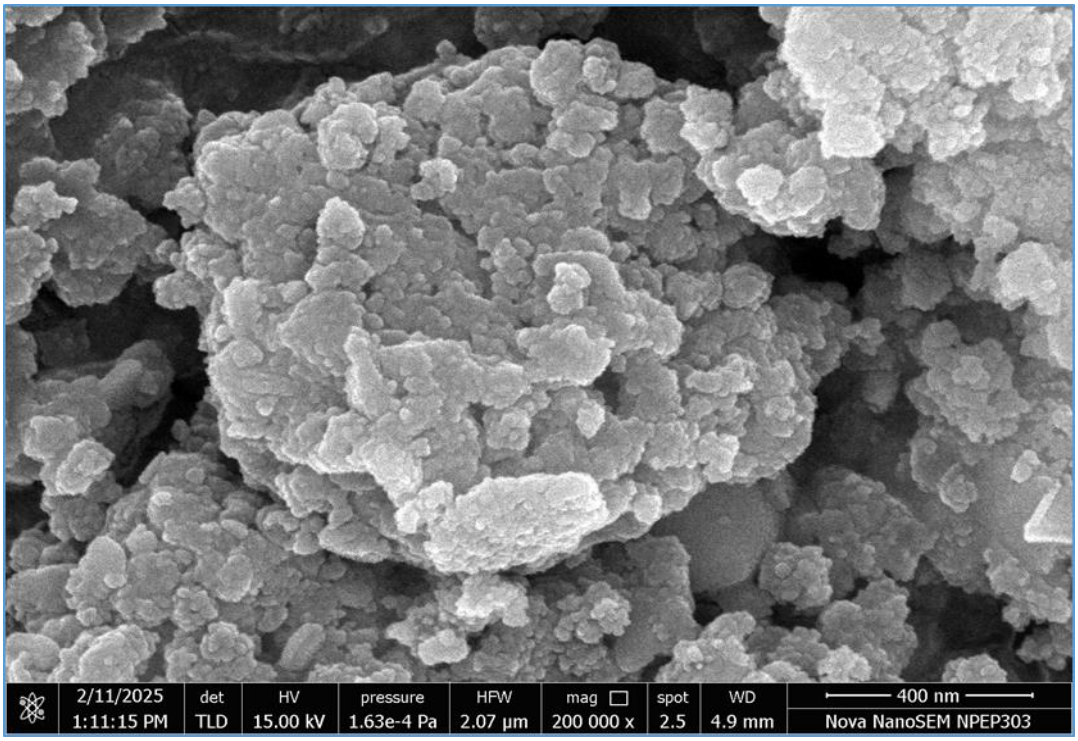

Figure 1. FESEM Image of $CuInSe_2$ thin films

Figure 1 shows the grains are interconnected and exhibit a rough surface texture, indicating a porous and compact structure, which is beneficial for applications like photovoltaics and gas sensing due to increased surface area [18, 19].

$$S_w = \frac{6}{\rho d} \quad \text{(Eq. 1)}$$

Where, Sw - the specific surface area, d - the diameter of the spherical particles, and ρ - the density.

The particle sizes appear to be in the nanometer range, with larger agglomerates forming due to the coalescence of smaller crystallites. The well-developed grain boundaries and uniform distribution suggest that the spray pyrolysis deposition technique facilitated homogeneous film growth. This nanostructured morphology can significantly influence the electrical and optical properties of the $CuInSe_2$ thin films by enhancing charge transport pathways and light absorption efficiency [19, 20]. The specific surface area was estimated using BET method [Eq. 1] and it was found to be 3.68 m^2/g.

3.1.2 Energy Dispersive X-ray Spectroscopy (EDS) Analysis

It was employed to determine the elemental composition of the synthesized $CuInSe_2$ thin films. Figure 2 presents the Energy Dispersive X-ray Spectroscopy (EDS) analysis and elemental mapping of the $CuInSe_2$ thin films.

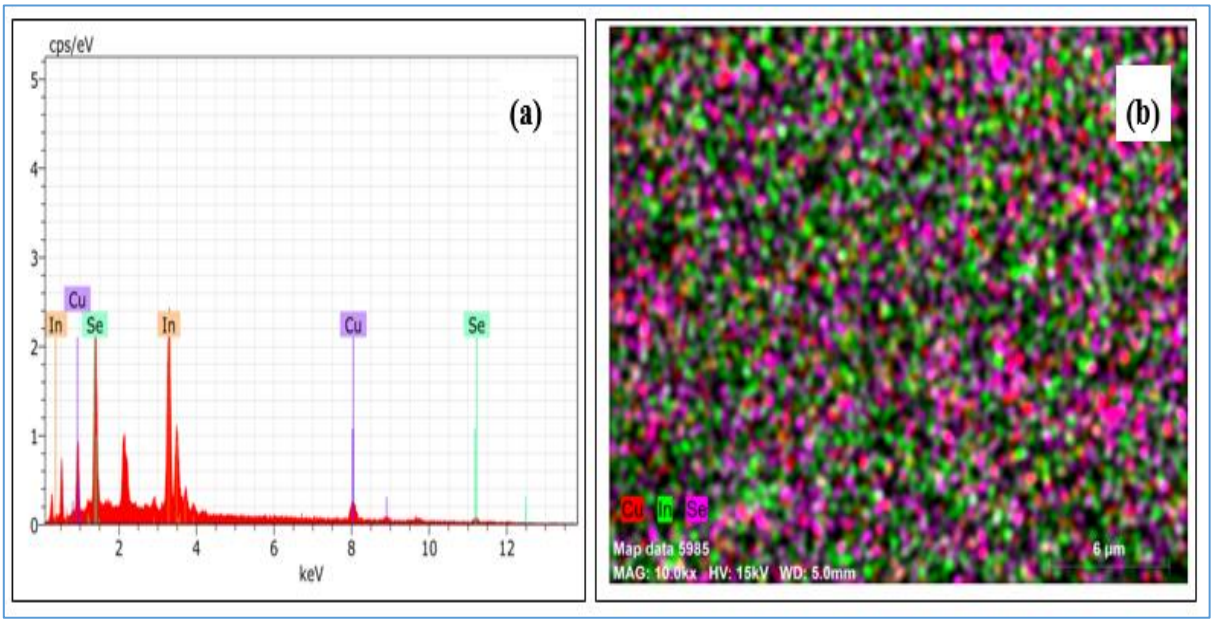

Figure 2. (a) EDS spectra, (b) Elemental mapping of $CuInSe_2$ thin films

In Figure 2 (a), the EDS spectrum clearly shows prominent peaks corresponding to indium (In), copper (Cu), and selenium (Se), confirming the successful formation of the $CuInSe_2$ phase. The presence of sharp and distinct peaks with no significant impurities indicates high purity and good stoichiometric incorporation of the elements. According to the quantitative EDS data, the normalized weight percentages of In, Se, and Cu are approximately 57.05 wt.%, 22.91 wt.%, and 20.04 wt.%, respectively, corresponding to atomic percentages of 45.07% In, 26.32% Se, and 28.60% Cu. This composition aligns reasonably well with the expected stoichiometry of the $CuInSe_2$ compound, although a slight excess of indium is observed, which may influence the electrical conductivity and defect structure. Figure 2(b) displays the elemental mapping, with red, green, and pink dots representing the spatial distribution of Cu, In, and Se, respectively. The uniform and dense distribution of all three elements across the scanned area confirms the homogeneity of the thin film and successful co-deposition of the elements during the spray pyrolysis process [21, 22]. This elemental uniformity is crucial for achieving consistent optoelectronic and structural properties in $CuInSe_2$ thin films, especially for device applications such as photovoltaics and sensors.

3.1.3 X-ray Diffraction (XRD) Pattern Analysis

Figure 3 displays the X-ray Diffraction (XRD) pattern of the synthesized $CuInSe_2$ thin films.

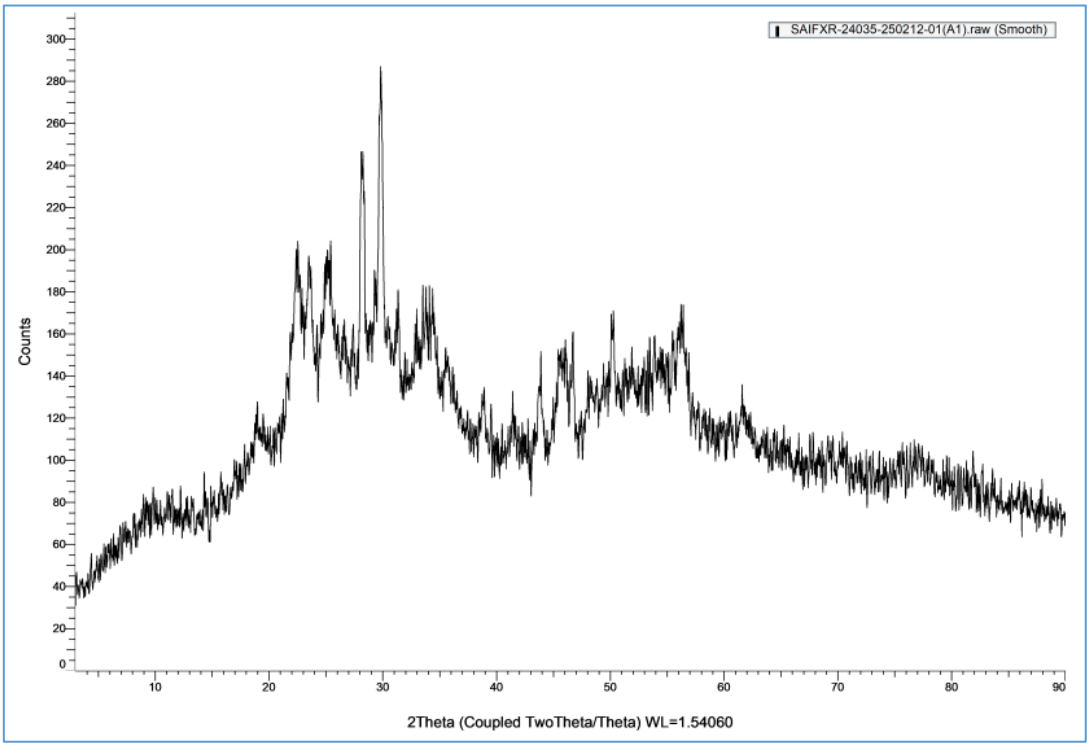

Figure 3. XRD pattern of $CuInSe_2$ thin films

The diffraction peaks observed correspond well to the chalcopyrite phase of $CuInSe_2$ and the pattern is in good agreement with the standard data provided by the JCPDS card No. 40-1487 [23, 24]. This confirms the successful formation of a single-phase chalcopyrite $CuInSe_2$ structure with good crystallinity. The major diffraction peaks are observed at 2θ values around 26.6°, 44.2°, and 52.4°, which correspond to the crystal planes indexed as (112), (220)/(204), and (312)/(116), respectively. Among these, the (112) peak is the most intense, indicating that the film has a strong preferential orientation along the (112) plane, which is a characteristic feature of well-crystallized chalcopyrite $CuInSe_2$ thin films. The sharpness and intensity of the peaks suggest good crystallinity of the material, while the absence of impurity peaks confirms the phase purity of the deposited films [24, 25]. The crystallite size was determined by Scherrer formula [Eq. 2]

$$D = \frac{K\lambda}{\beta Cos\theta} \qquad \text{(Eq. 2)}$$

D - Crystallite size, K - Scherrer constant (0.9), β - full width at half maximum (FWHM), and λ - wavelength of the X-ray source.

The presence of multiple diffraction peaks matched to standard Miller indices further supports the polycrystalline nature of the film. The observed pattern confirms the tetragonal structure of $CuInSe_2$ with lattice parameters consistent with reported values. This crystalline structure is essential for the material's applications in optoelectronics and solar energy conversion, as it directly affects charge carrier mobility and absorption properties.

3.2 Electrical characterizations

The electrical characterizations were conducted based on resistivity, temperature coefficient of resistance (TCR), and activation energy in both high and low temperature ranges. The electrical characteristics of $CuInSe_2$ thin films were carried out utilizing the half bridge method. The resistivity, temperature coefficient of resistance (TCR), and activation energy were calculated using equations 3, 4, and 5, respectively [26, 27].

$$\rho = \left(\frac{R \times b \times t}{l}\right)\Omega - m \qquad \text{(Eq. 3)}$$

Where, ρ - resistivity of the produced film, R -resistance at ambient temperature, b -width of the film, and t - the thickness of the film, and l- the length of the film.

$$TCR = \frac{1}{R_o}\left(\frac{\Delta R}{\Delta T}\right) \ /^{O} C \qquad \text{(Eq. 4)}$$

Where, ΔR - change in resistance, ΔT - temperature difference, and R_o - initial resistance of the film.

$$\Delta E = \frac{log\, R}{log\ Ro} \times KT \qquad \text{(Eq. 5)}$$

Where, ΔE -Activation energy, R -Resistance at raised temperature, R_0 -Resistance at room temperature. The thickness of the $CuInSe_2$ films was determined using the weight difference method as per equation 6.

$$t = \Delta m/\rho A \qquad \text{(Eq. 6)}$$

Where,

Δm - mass difference before and after deposition, ρ -density, and A – area of the film.

Figures 4 and 5 illustrate the temperature-dependent electrical behavior of $CuInSe_2$ thin films. Figure 4 shows the variation of resistance with temperature (R–T) in the range of 300–470 K. The resistance decreases exponentially with increasing temperature, demonstrating a negative temperature coefficient of resistance (TCR). This behavior confirms that the $CuInSe_2$ thin films exhibit semiconducting properties, where thermal activation of charge carriers leads to enhanced electrical conductivity. The recorded TCR value is -0.00538 $°C^{-1}$, indicating moderate sensitivity of resistance to temperature, which is beneficial for temperature sensor or thermistor applications [27, 28].

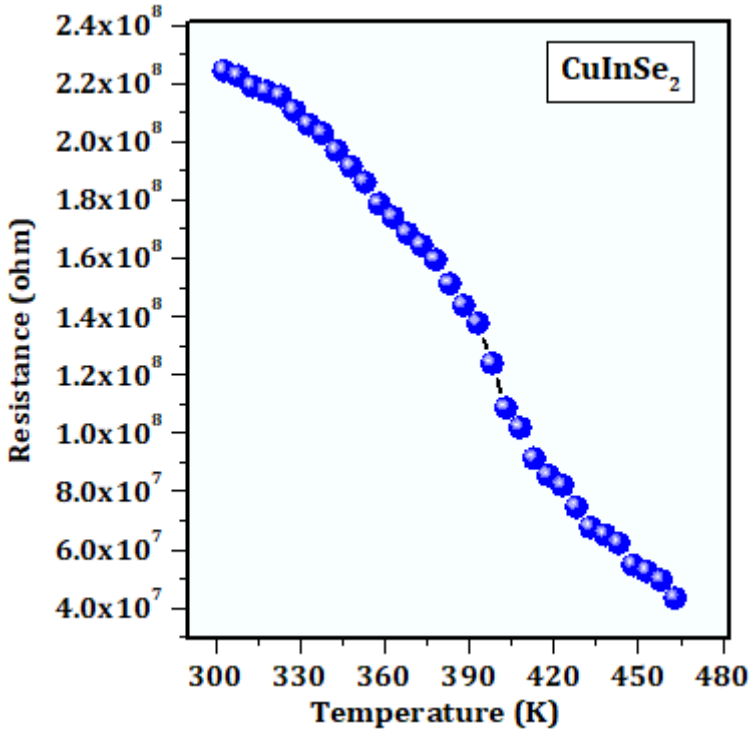

Figure 4. Resistance versus temperature plot of $CuInSe_2$ thin films

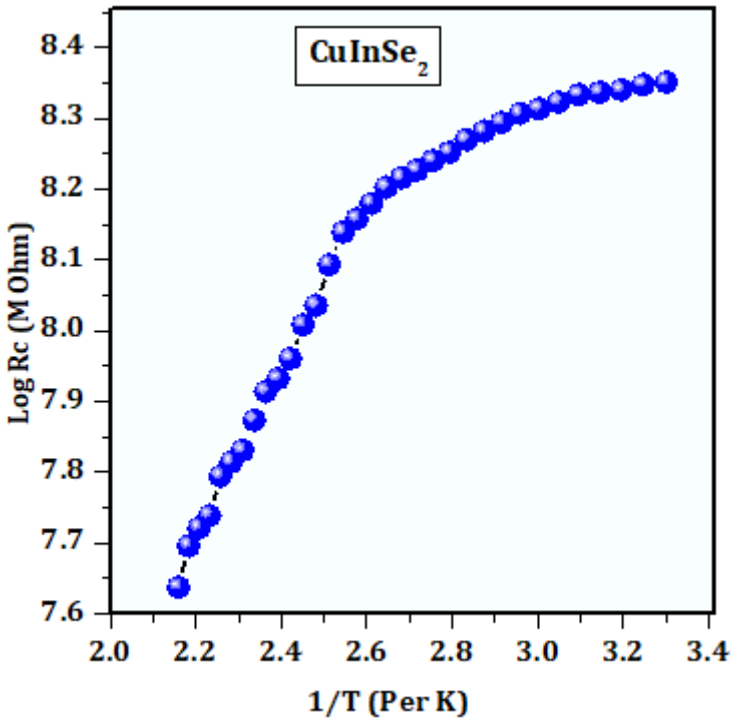

Figure 5. Log Rc versus inverse of temperature plot of $CuInSe_2$ thin films

Figure 5 presents a plot of logarithmic resistance (log Rc) versus inverse temperature (1/T). This Arrhenius-type plot follows the equation [Eq. 5]. The slope of the linear region yields the activation energy of charge carriers. The plot clearly shows two linear regions, corresponding to two different conduction regimes: high-temperature region (HTR) and low-temperature region (LTR). From the slope analysis, the activation energies were found to be 0.2779 eV (HTR) and 0.0138 eV (LTR). The higher value at elevated temperatures suggests thermal excitation of charge carriers across the bandgap, while the lower energy at low temperatures indicates possible defect-related hopping conduction [26, 28]. The electrical outcomes and thickness of $CuInSe_2$ thin films are tabulated in Table 1.

Table 1. Electrical outcomes of $CuInSe_2$ thin films

Thin film sample	**Thickness (nm)**	**Resistivity (Ω.m)**	**TCR (/°C)**	**Activation Energy (eV)**	
				HTR	**LTR**
$CuInSe_2$	44	4.93625	-0.00538	0.2779	0.0138

CONCLUSIONS:

The $CuInSe_2$ thin films were successfully synthesized using the spray pyrolysis technique on glass substrates, employing high-purity AR-grade precursors. The morphological analysis using FESEM revealed a highly porous and agglomerated nanostructure composed of fine spherical grains, which is advantageous for enhancing surface-related phenomena such as light absorption and gas interaction. Elemental composition and uniformity were confirmed by EDS and elemental mapping, showing a homogeneous distribution of Cu, In, and Se throughout the film with no significant impurities. The atomic percentages closely match the stoichiometric ratio of $CuInSe_2$, validating the effective incorporation of all constituent elements. XRD analysis confirmed the formation of the tetragonal chalcopyrite phase of $CuInSe_2$, in agreement with JCPDS card No. 40-1487. Major diffraction peaks indexed to (112), (220)/(204), and (312)/(116) planes indicated polycrystalline nature with preferential orientation along the (112) plane and good crystallinity. Electrical

characterization showed that the resistance of the films decreases with increasing temperature, indicating semiconducting behavior. The negative temperature coefficient of resistance (TCR = -0.00538 $°C^{-1}$) and the Arrhenius plot revealed two conduction regions, with activation energies of 0.2779 eV in the high-temperature region and 0.0138 eV in the low-temperature region. The film exhibited a resistivity of 4.93625 Ω·m and a thickness of 55.79 nm. The structural, morphological, compositional, and electrical results confirm that the prepared $CuInSe_2$ thin films possess desirable qualities for applications in photovoltaics, thermistors, and optoelectronic devices, owing to their phase purity, uniform nanostructure, and thermally activated semiconducting properties.

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Declaration of competing interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in the present research paper.

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