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Enhancing Thermoelectric Performance of Sb-Doped $CuInS_2$ Thin Films through Controlled Annealing

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Abstract— **This study investigates the effect of annealing temperature on the thermoelectric performance of 4% Sb-doped $CuInS_2$ thin films deposited via thermal evaporation at substrate temperatures of ambient, 100°C, and 200°C. Using the PhotoThermal Deflection (PTD) method, thermal conductivity was found to increase with annealing temperature, while thermal diffusivity decreased. Electrical conductivity also rose from 4.5 S/cm to 13.9 S/cm. Films annealed at 200°C exhibited the best thermoelectric performance. The research highlights a systematic approach to optimizing Sb-doped $CuInS_2$ thin films, offering insights for future studies on Seebeck coefficients and figure of merit (zT) to advance thermoelectric materials.**

Keywords— **Sb-doped $CuInS_2$, PTD technique, Annealing temperature, Thermal conductivity, Electrical conductivity, Thermoelectric materials, Thin films.**

I. INTRODUCTION

The energy crisis and the associated environmental issues are major challenges in today's rapidly changing world, where the demand for electricity is increasing. To address these challenges, the required energy conversion systems should possess scalability and reliability as their two main desired features. To achieve this goal, ongoing research is being conducted to develop such systems, although it is not as simple as it may seem [1-4].

One of the energy systems that has garnered significant attention in recent years is the thermoelectric generator (TEG). This device is a type of heat engine that has the potential to offer solutions to the aforementioned challenges. The advantages of such devices are numerous: they are environmentally friendly, highly scalable, reliable, and have long lifespans for dependable operation [5-8]. This solid-state device can directly convert thermal energy from sources such as solar systems, factories, power plants, computers, and even human bodies into electrical power using the Seebeck effect [9-16]. However, the practical applications of TEG are hindered by low conversion efficiencies primarily attributed to the intrinsic properties of thermoelectric (TE) materials.

TE materials constitute a large family of materials, including those from semimetals and semiconductors to ceramics, encompassing various dimensions from bulk, films, and wires to clusters. TE materials are considered promising candidates for easing the energy crisis due to their capacity to convert waste heat into electricity based on Seebeck, Peltier, and Thomson effects. Ideally, a good TE material should have low thermal conductivity, high electrical conductivity, and a high Seebeck coefficient [17-19]. Within this context, many researchers have drawn attention to semiconducting materials, particularly those having properties similar to chalcopyrite CuInS2 (CIS) of the I-III-VI2 group. These materials are promising TE materials due to their potential for low environmental impact, high chemical stability, and potential use in solar energy conversion [20-24].

Several studies have shown that the properties of CuInS2 thin films can be improved by optimizing deposition conditions and doping. For instance, Ben Rabeh et al. [21, 22] examined the effects of antimony incorporation on the structural, optical and electrical properties of CuInS2 thin films, revealing that Sb-doped samples post-annealing have a bandgap energy of 1.38-1.51 eV. It was also found that only CuInS2 samples

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doped with a high Sb incorporation (4 wt%) exhibit p-type conductivity. Similarly, Mobarak et al. [24] investigated the electrical and thermoelectric properties of CuInS2 single crystals, characterizing them structurally using X-ray diffraction. Their analysis confirmed that CuInS2 compounds have a tetragonal structure with a single phase, and they identified an energy gap of 1.51 eV, indicating the potential for these single crystals to be utilized as solar energy converters.

Further, Akaki et al. [25] studied the structural, electrical, and optical properties of Sb-doped CuInS2 thin films. They demonstrated that polycrystalline CuInS2 films could be successfully obtained by annealing above 200 °C and found that Sb-doped CuInS2 thin films approached stoichiometry compared to non-doped samples. More recently, Giri et al. [26] explored the thermoelectric properties of CuInS2, revealing a maximum zT value of 1.04 10-4 at 800K and an absolute Seebeck coefficient of -30 μV/K at 300K. This suggests that CuInS2 has high potential for thermoelectric power generation. However, further research is needed to enhance these properties using suitable dopants to achieve a high zT value.

This study focuses on enhancing the thermoelectric performance of $CuInS_2$ thin films through antimony (Sb) doping and optimized annealing. Sb is chosen as a dopant for its ability to improve electrical conductivity and the Seebeck coefficient [27] while maintaining carrier mobility [28]. Additionally, Sb modifies the band structure and reduces thermal conductivity via phonon scattering, enhancing the thermoelectric figure of merit (zT) [29]. The Photothermal Deflection (PTD) method was used to characterize thermal properties, as it provides accurate measurements of thermal conductivity and diffusivity simultaneously. The results demonstrate that Sb-doped $CuInS_2$ thin films annealed at 200°C exhibit the most promising thermoelectric performance, with significant improvements in electrical conductivity and thermal properties. This research contributes to the development of efficient thermoelectric materials and paves the way for future studies on optimizing the Seebeck coefficient and figure of merit (zT) for advanced TEG applications.

II. Photothermal Deflection Technique

The PhotoThermal Deflection (PTD) technique was used to characterize the thermal properties of Sb-doped $CuInS_2$ thin films. This non-destructive, contactless method ensures accurate measurements without damaging the samples, which is crucial for maintaining structural integrity. Unlike techniques such as Photopyroelectric (PPE) [30] and Electropyroelectric (EPE) [31-32], PTD excels in simultaneously measuring thermal conductivity and diffusivity [33-35] and provides reliable results consistent with theoretical models [36].

The PTD technique operates on the principle of the "mirage effect." A modulated light beam heats the sample, generating a thermal wave that propagates through both the sample and the surrounding fluid. This thermal wave induces a temperature gradient, which in turn creates a refractive index gradient in the fluid. As a laser probe beam passes through the fluid, it is deflected due to this refractive index gradient. To quantify the deflection, the temperature at the sample's surface is first determined, enabling the calculation of the refractive index gradient. This approach allows for precise and non-destructive measurement of thermal properties, making it ideal for characterizing Sb-doped $CuInS_2$ thin films.

A. *Theoretical Model*

The theoretical model is constructed by solving the heat equations within distinct media layers: fluid (f), sample (s), and backing (b) (Fig. 1), while ensuring continuity of temperature and heat flow across different interfaces (b/s, s/f).

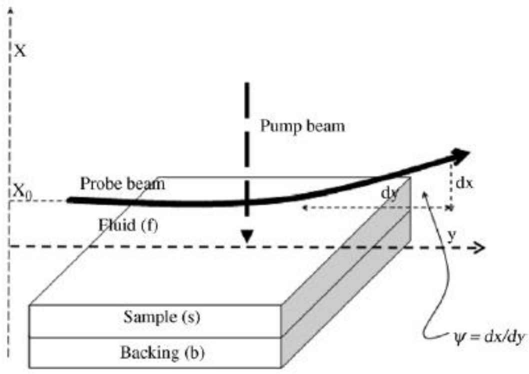

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Fig. 1 Schematic representation of the probe beam deflection.

Given the uniform heating of the probe, our approach considers a one-Dimensional (1-D) heat treatment. The deflection of the probe beam (ψ) can be derived through the Eq. 2.

$$\psi = \frac{dn}{dT_f}|T_0|e^{-\frac{x}{\mu_f}}e^{j(\theta+\frac{\pi}{4}-\frac{x}{\mu_f})}e^{j\omega t} = |\psi|e^{j(\omega t+\varphi)} \quad (1)$$

$|\psi|$ and φ represent the amplitude and phase of photothermal deflection, respectively, and are defined as:

$$|\psi| = \frac{\sqrt{2}L}{n\mu_f}\frac{dn}{dT_f}|T_0|e^{-\frac{x}{\mu_f}} \quad (2)$$

$$\varphi = -\frac{x_0}{\mu_f} + \theta + \frac{5\pi}{4} \quad (3)$$

where:

- $\mu_f = \sqrt{\frac{\alpha}{\pi f}}$ represents the thermal diffusion length, indicating the penetration depth of the thermal wave through the sample,
- x_0 is the distance between the probe beam axis and the sample surface,
- f is the modulation frequency.

The elevation temperature of the sample surface, denoted as T_0, is expressed as:

$$T_0 = |T_0|e^{i\theta} \quad (4)$$

where T_0 and θ represent the amplitude and phase of T_0, respectively, which are functions of the thermal conductivity (k) and thermal diffusivity (D) of the studied samples [37,38].

B. *Experimental Set-Up*

The experimental set-up, depicted in Fig. 2 and detailed in [39], consists mainly of a 100 W halogen lamp, with its light modulated by an SR530 mechanical chopper, which is then focused onto the sample surface. A He-Ne laser probe beam, with a wavelength of 632.8 nm, scans the surface and undergoes deflection.

The deflected beam is measured by a four-quadrant photodetector (QD50T), linked to a lock-in amplifier (EG&G5210). The frequency f of the mechanical chopper can be adjusted via an IEEE bus intermediary, controlled by a PC microcomputer.

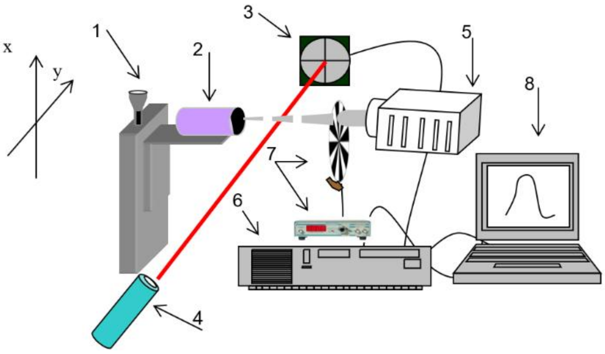

Fig. 2 PTD experimental set-up.

1-Table of horizontal and vertical micrometric displacement, 2-Sample, 3-position photodetector, 4-fixed Laser Source, 5-halogen Lamp, 6-look-in amplifier, 7-Mecanical Chopper, 8-PC.

III. ELECTRICAL CHARACTERIZATION

In addition to thermal characteristics, the electrical properties play a crucial role in the performance of Sb-doped $CuInS_2$ thin films. To evaluate these properties, specific methods were employed to analyze conductivity and resistivity. The hot probe method [40] was used to determine the type of conductivity (p-type or n-type) by placing a heated probe in contact with the film. Meanwhile, the resistivity of the films was measured using a digital universal meter, providing accurate resistance measurements, which were then used

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to calculate the resistivity based on the film's geometry. These electrical measurements are essential for understanding and optimizing the overall thermoelectric performance of the material.

IV. Results and Discussion

The results of this study involved comparing the experimental phase curves obtained from PTD measurements with theoretical curves by varying the values of thermal conductivity (k) and thermal diffusivity (D). The optimal match between the experimental and theoretical curves was achieved by adjusting these thermal properties.

Figs. 3, 4 and 5 depict the experimental (data points) and theoretical (lines) variations of phase against the square root of the modulation frequency (f) for Sb-doped $CuInS_2$ (Ts = Ta), $CuInS_2$ (Ts = 100°C), and $CuInS_2$ (Ts = 200°C). The theoretical phase curves correspond to specific values of thermal properties outlined for each sample.

The uncertainties (U) in the thermal coefficients were estimated by examining the maximum and minimum values until the best agreement between theoretical and experimental data was achieved, by means of the following formula provided in [41]:

$$U = \frac{Max - Min}{2\sqrt{3}} \tag{5}$$

The phase curves clearly display a plateau at low frequencies (f < 15Hz) and a subsequent decline at higher frequencies, consistent with the theoretical predictions for optically opaque samples [42], as indicated by the equation above.

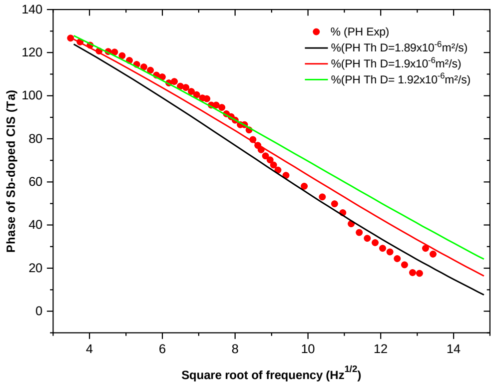

Fig. 3 Variation of PTD signal phase (degree) of Sb-doped $CuInS_2$ (Ts = Ta) as a function of the square root of frequency for k = 0.035 $Wm^{-1}K^{-1}$.

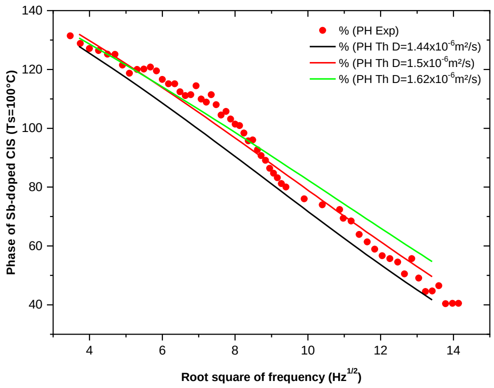

Fig. 4 Variation of PTD signal phase (degree) of Sb-doped $CuInS_2$ (Ts = 100°C) as a function of the square root of frequency for k = 0.055 $Wm^{-1}K^{-1}$.

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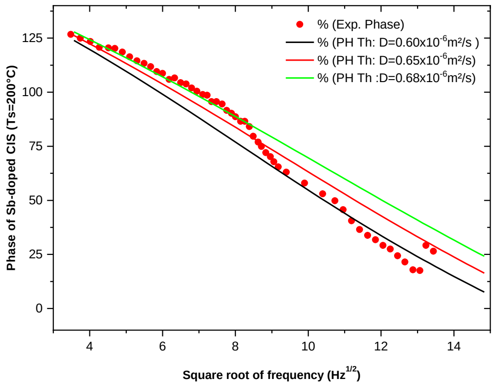

Fig. 5 Variation of PTD signal phase (degree) of Sb-doped $CuInS_2$ (Ts = 200°C) as a function of the square root of frequency for k = 0.075 $Wm^{-1}K^{-1}$.

In this study, we focus on enhancing the thermoelectric performance of $CuInS_2$ thin films through antimony (Sb) doping and optimized annealing processes. The efficiency of thermoelectric (TE) materials principally depends on the figure of merit (zT), which serves as the performance index for a TE material and is defined by:

$$zT = \sigma\alpha^2 T/k \tag{6}$$

where the parameters α, σ, T, and k represent the Seebeck coefficient, electrical conductivity, absolute temperature, and total thermal conductivity that is the sum of the electronic (k_e) and lattice (k_L) parts mainly.

The search for excellent TE materials primarily entails identifying materials with the highest figure of merit (zT). To increase the zT value, it is essential to improve the power factor ($\sigma\alpha^2$) while simultaneously reducing the total thermal conductivity (k). However, these three parameters (σ, α, and k) are connected and depend on carrier concentration, making independent adjustment of these parameters challenging.

The thermal conductivity of undoped $CuInS_2$, as reported in the literature, is approximately 0.4 $Wm^{-1}K^{-1}$ [43]. For the Sb-doped $CuInS_2$ samples, the thermal properties are summarized in Table 1, showing consistent results with previous studies that investigated the impact of doping and annealing on the thermal characteristics of $Sn_xSb_2S_\gamma$ sulfosalt thin films (1≤x≤3, 4≤y≤6) using the Electro-PyroElectric method [44].

The thermal conductivity consists of contributions from both electronic (k_e) and lattice (k_L) components. As electrical conductivity increases, the electronic contribution to thermal conductivity (k_e) also tends to increase, which can explain part of the observed rise in thermal conductivity with higher annealing temperatures. However, the overall thermal conductivity is also affected by lattice vibrations (phonons), which are independent of electrical carriers and can be influenced by doping and microstructural changes induced by annealing.

Table 1: Thermal and electrical properties of annealed Sb-doped $CuInS_2$ thin films.

substrate annealing temperature T_s (°C)	**Ta**	**100**	**200**
Thickness L (µm)	1.44	1.57	1.56
Thermal conductivity k ($Wm^{-1}K^{-1}$)	0.035	0.055	0.075
Thermal diffusivity D (m^2s^{-1})	1.9 10^{-6}	1.5 10^{-6}	0.65 10^{-6}
Electrical conductivity σ (Scm^{-1})	4.5	7.2	13.9
Ratio k/σ ($WK^{-1}S^{-1}$)	7.7 10^{-5}	6.9 10^{-5}	5.4 10^{-5}

The electrical conductivity (σ) of Sb-doped $CuInS_2$ thin films increased significantly from 4.5 S/cm at ambient temperature to 13.9 S/cm at 200°C (Table 1). This improvement is attributed to thermal annealing, which enhances atomic diffusion, reduces defects, and improves crystallinity. The stability of donor and acceptor impurity concentrations during Sb-doping process also contributes to maintaining high conductivity. These findings align with Akaki et al. [25], highlighting the importance of thermal treatment in optimizing electrical performance. Thermal conductivity (k) values, though low compared to electrical conductivity,

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follow a consistent pattern. This is advantageous for thermoelectric applications, as low thermal conductivity combined with high electrical conductivity enhances the figure of merit (zT) of the material. The ratio of thermal to electrical conductivity (k/σ), which is a critical indicator of thermoelectric performance, was notably small for thin films annealed at 200°C, indicating their potential for efficient thermoelectric applications.

It is essential to acknowledge that thermal annealing modifies the atomic structure, influencing defects and atomic disorder. These changes affect thermal heat capacity and electrical resistivity through electron-phonon interactions, underscoring the complex behavior of solid-state materials under varying thermal conditions.

V. Conclusions

This study provides a comprehensive analysis of the impact of annealing temperature on the thermoelectric performance of Sb-doped $CuInS_2$ thin films, utilizing the PhotoThermal Deflection (PTD) technique for precise thermal property measurements. Our results, consistent with prior research, reveal significant improvements in both electrical and thermal conductivity at higher annealing temperatures, emphasizing the critical role of controlled thermal treatment in optimizing thermoelectric materials.

To build on these findings, future work should focus on measuring the absolute Seebeck coefficient to refine the Figure of Merit (zT) and explore time-dependent structural changes and atomic disorder induced by annealing. Advanced characterization techniques could also provide deeper insights into vibrational modes and their relationship with thermoelectric properties, offering a more complete understanding of annealing effects on material behavior.

In conclusion, the enhanced thermoelectric performance of Sb-doped $CuInS_2$ thin films underscores the importance of optimized annealing processes. These findings not only validate the potential of Sb-doped $CuInS_2$ for thermoelectric applications but also lay the groundwork for future research to further refine zT values and advance the development of efficient thermoelectric materials.

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