

Article

Effects of Post-Thermal Annealing on the Structural, Morphological, Optical and Electrical Properties of Co-Evaporated Sb_2Se_3 Thin Films

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Abstract

This study investigates the effects of post-annealing on the properties of thermally evaporated antimony selenide (Sb_2Se_3) thin films. The prepared samples were annealed at different temperatures of 150, 200, 250 and 300 °C for half an hour. Different analytical techniques were used to investigate the properties of the annealed samples. XRD analysis confirmed the formation of orthorhombic Sb_2Se_3 in all samples, while variations in diffraction peak intensity and preferred orientation revealed a strong dependence of crystallographic texture on annealing temperature. EDX results indicated progressive selenium loss with increasing annealing temperature, particularly for the film annealed at 300 °C. AFM analysis showed that the 300 °C sample exhibited increased surface roughness compared with the films annealed at lower temperatures. All films demonstrated good optical absorption in the visible region with optical band-gap values ranging from approximately 1.0 to 1.3 eV, making them suitable for photovoltaic applications. Hall-effect measurements revealed significant changes in carrier concentration, mobility, and resistivity as a function of annealing temperature. Among the investigated conditions, the film annealed at 250 °C exhibited the most favorable combination of structural quality and electrical performance, indicating that this temperature provides an optimal annealing condition for thermally evaporated Sb_2Se_3 thin films considered for optoelectronic and photovoltaic applications.

Keywords: Sb_2Se_3 ; thin films; post-annealing; optical properties

1. Introduction

Antimony selenide (Sb_2Se_3) is an abundant and nontoxic material that has various properties that allow it to be considered a promising absorber layer for thin-film photovoltaic applications. Sb_2Se_3 displays a good band gap of around 1.1 to 1.3 eV direct and 1.03 eV indirect, an absorption coefficient that is larger than 10^5 cm^{-1} and a carrier lifetime of around 60 ns [1–4]. Theoretically, studies predict that Sb_2Se_3 -based solar cells could achieve power conversion efficiency exceeding 30% [5]. In practice, the conversion efficiency cell have rapidly increased and has reached 9.2%, demonstrating the significant potential of Sb_2Se_3 for next-generation photovoltaic technologies [2,3].

Different fabrication methods have been employed to deposit Sb_2Se_3 thin films, such as electrodeposition [6,7], e-beam evaporation [8] and chemical bath deposition [9,10]. Thin-film deposition with solution methods is easy and cheap [11–20], but utilizes toxic solvents and chemicals while contaminating the films through applying the procedures in a non-vacuum environment. In contrast, physical deposition techniques are widely



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recognized for their reliability, reproducibility, and industrial applicability, as demonstrated by their successful implementation in Cu(In,Ga)Se₂ (CIGS) and CdTe solar-cell manufacturing [21,22].

Recently, several physical vapor deposition techniques, including molecular beam epitaxy (MBE), pulsed laser deposition (PLD) and magnetron sputtering [23–25] have been employed for the growth of Sb₂Se₃ thin films. While these methods can produce high-quality films, they generally require more advanced equipment, higher processing costs or more complex deposition procedures. In contrast, thermal evaporation is a simple, cost-effective and scalable deposition technique that is well appropriate for large-area thin-film fabrication [22].

However, thermally evaporated Sb₂Se₃ films often require post-deposition treatment to improve crystallinity, grain growth and overall film quality. Several approaches, including post-annealing treatments and doping engineering, have been explored to enhance the quality and performance of thin films [26–30]. Optimizing the annealing conditions of thermally evaporated thin films is of particular interest, as it has proven to be an effective strategy for improving the performance of commercially successful thin-film photovoltaic technologies, such as CIGS and CdTe solar cells [31]. In this study, the effects of post-annealing temperature on the morphological, structural, optical and electrical properties of Sb₂Se₃ thin films deposited by a dual thermal evaporation system are investigated.

2. Experiment

Near stoichiometric Sb₂Se₃ thin-film samples were prepared by the thermal evaporation method at a high vacuum of 10^{−5} mbar at room temperature. In this method, two separated boats were employed, with selenium (Se: 99.99%) powder placed in boat 1 and antimony (Sb: 99.99%) shot (1–2 mm) placed in boat 2. The applied current was 50 A to evaporate Se atoms, and 100 A to evaporate Sb atoms. The boats were heated at the same time to grow films on the glass substrates. The samples were then post-annealed in a nitrogen atmosphere, each at different temperatures of 150, 200, 250 and 300 °C for half an hour.

A desktop scanning electron microscope (JCM-6000, JEOL, Tokyo, Japan) was used to determine the elemental compositions of the deposited and annealed films. Shimadzu XRD-6000 diffractometer (Shimadzu, Kyoto, Japan) was used to determine the crystal structure of the annealed films, applying a radiation source generated at an X-ray tube voltage of 40 kV, and a current of 30 × 10^{−3} A. Also, Bruker Senterra Raman spectroscopy (Bruker, Billerica, MA, USA) was employed to study the structure of the annealed thin films, via an excitation laser (λ = 532 nm) with a power of 5 mW. The surface morphology of the annealed films was characterized by Veeco CP-II atomic force microscopy (Veeco, Plainview, NY, USA), applying non-contact mode with a silicon tip at a scan rate of 1 Hz. The thickness of the annealed Sb₂Se₃ thin films was measured using a Dektak 150 Stylus profilometer (Veeco, Plainview, NY, USA). The measured thickness values for the films deposited at 150 °C, 200 °C, 250 °C, and 300 °C were 227 nm, 200 nm, 190 nm, and 150 nm, respectively. The optical properties were investigated by using a Shimadzu 3150 UV–VIS–NIR double-beam spectrophotometer (Shimadzu Kyoto, Japan) with a resolution of 0.1 nm. Also, the electrical resistivity, carrier type, carrier concentration and mobility of the films were measured using a Ecopia HMS 3000 Hall measurement system (Ecopia Corporation, Anyang-si, Republic of Korea).

3. Results and Discussion

The prepared thin films were examined with EDX to reveal their chemical compositions. Four samples with near Sb₂Se₃ stoichiometric composition (Se/Sb is around 1.5)

were determined as shown in the EDX spectra in Figure 1. The selected samples were then annealed at different temperatures to investigate the effects of post-annealing on the properties of the films. The EDX results indicate a decrease in selenium content and a corresponding increase in antimony content after annealing. This trend is attributed to the higher volatility of selenium compared with antimony, leading to partial selenium evaporation during thermal treatment. The compositional change is relatively small for the films deposited at 150–250 °C but becomes significant for the 300 °C sample, where the Se content decreases from 59.81% to 47.17%. This behavior suggests enhanced selenium loss at higher temperatures, resulting in a deviation from the stoichiometric Sb_2Se_3 composition and the formation of relatively Sb-rich films. Such stoichiometric variations can affect the concentration of native defects and may contribute to the observed changes in the structural and optical properties of the films.

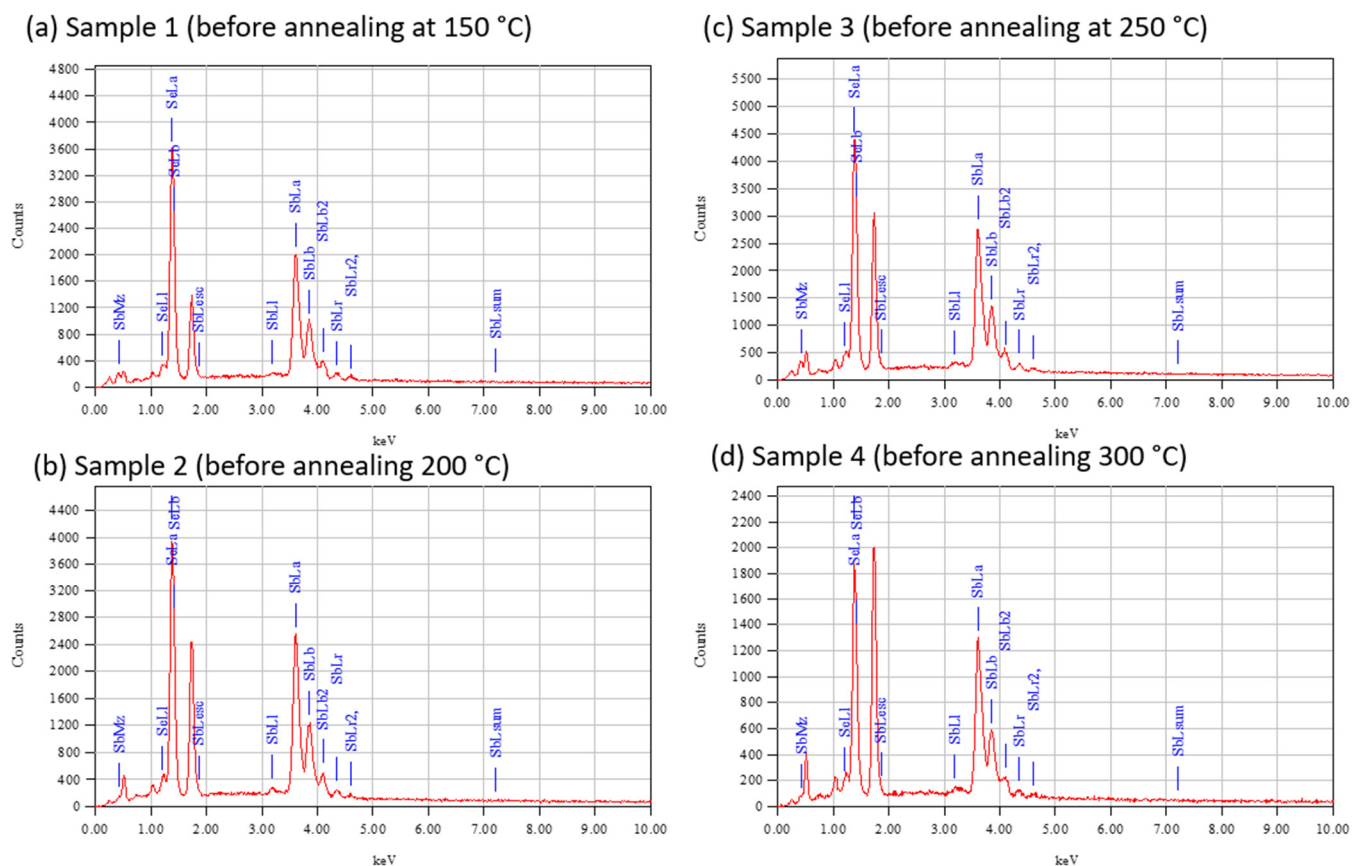


Figure 1. EDX spectra of the deposited Sb_2Se_3 thin films.

The crystal structure of the post-annealed samples was examined by XRD, as shown in Figure 2. Generally, the annealed films at different temperatures exhibit diffraction peaks that match the Sb_2Se_3 thin films' orthorhombic phase (JCPDS 00.015–0861). In particular, the samples annealed at 150 °C, 200 °C and 250 °C display almost the same diffraction peaks but with different intensities. The crystal orientation of [130] is dominant in the diffraction patterns of these samples. However, the sample annealed at 200 °C displays high peaks under 20°, which are the [020] and [120]. These peaks are recognized to negatively affect the solar-cell devices' performance [31]. In contrast, the peaks' intensities reduced with the sample annealed at 250 °C, and the preferred crystal orientation of [211], [221] and [212] clearly appear. The peaks disappeared on films annealed at 300 °C, and different peaks appeared with the crystal faces of [230]. In addition, a diffraction peak at $2\theta \approx 46^\circ$ appeared, which may be associated with Sb-rich phases [32]. Together with the reduction

in selenium content revealed by EDX analysis (Table 1), these results suggest structural changes induced by high-temperature annealing.

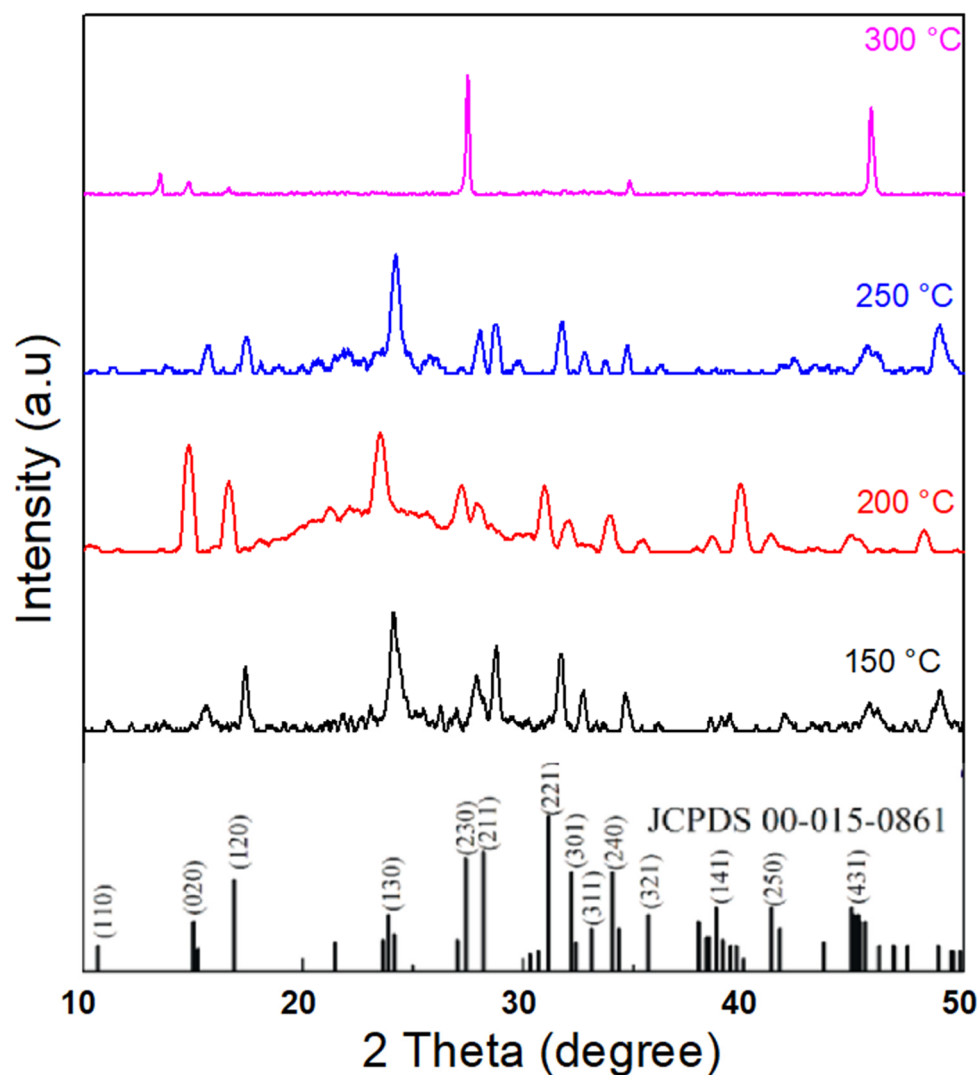


Figure 2. XRD patterns of Sb_2Se_3 thin films annealed at different temperatures.

Table 1. Elemental compositions of the samples before and after annealing.

Samples	Atomic Composition			
	As Deposited Films		After Annealing	
	Se %	Sb %	Se %	Sb %
150 °C	62.3	37.7	62.1	37.9
200 °C	59.6	40.4	57.2	42.8
250 °C	64.5	35.5	63.3	36.7
300 °C	59.8	40.2	47.2	52.8

Scherrer's equation was used to calculate the crystallite size of the annealed films:

$$D = 0.9\lambda / \beta \cos\theta$$

where D is the crystallite size, λ is the wavelength of X-rays, β is the FWHM of the corresponding peak in radians, and θ is the diffracted angle.

The full width at half maximum (FWHM) values of the principal diffraction peaks were analyzed and are summarized in Table 2 together with the calculated crystallite sizes. In general, the FWHM decreased with increasing annealing temperature, indicating enhanced crystallinity and grain growth. The films annealed at 150, 200, and 250 °C exhibited similar crystallite sizes, ranging from 16.6 to 17.4 nm. In contrast, the sample annealed at 300 °C showed a larger crystallite size of 55.8 nm. This increase is consistent with enhanced grain coarsening at elevated temperature and may also be associated with the structural changes observed in the XRD patterns. These findings are supported by the AFM results shown in Figure 3. The surfaces of the films annealed at 150, 200, and 250 °C were relatively smooth, with RMS roughness values of approximately 5, 9 and 6 nm, respectively. However, the film annealed at 300 °C exhibited a significantly rougher surface, with an RMS roughness of approximately 28 nm, accompanied by the appearance of larger surface grains. The AFM observations, therefore, corroborate the crystallite growth inferred from the XRD analysis.

Table 2. FWHM, crystallite size, and RMS roughness of the annealed Sb₂Se₃ thin films.

Annealed Films	FWHM	Grain Size D (nm)	RMS (nm)
150 °C	0.46800	17.35	4.9
200 °C	0.48400	16.62	9.4
250 °C	0.47330	17.23	5.61
300 °C	0.14710	55.81	28.3

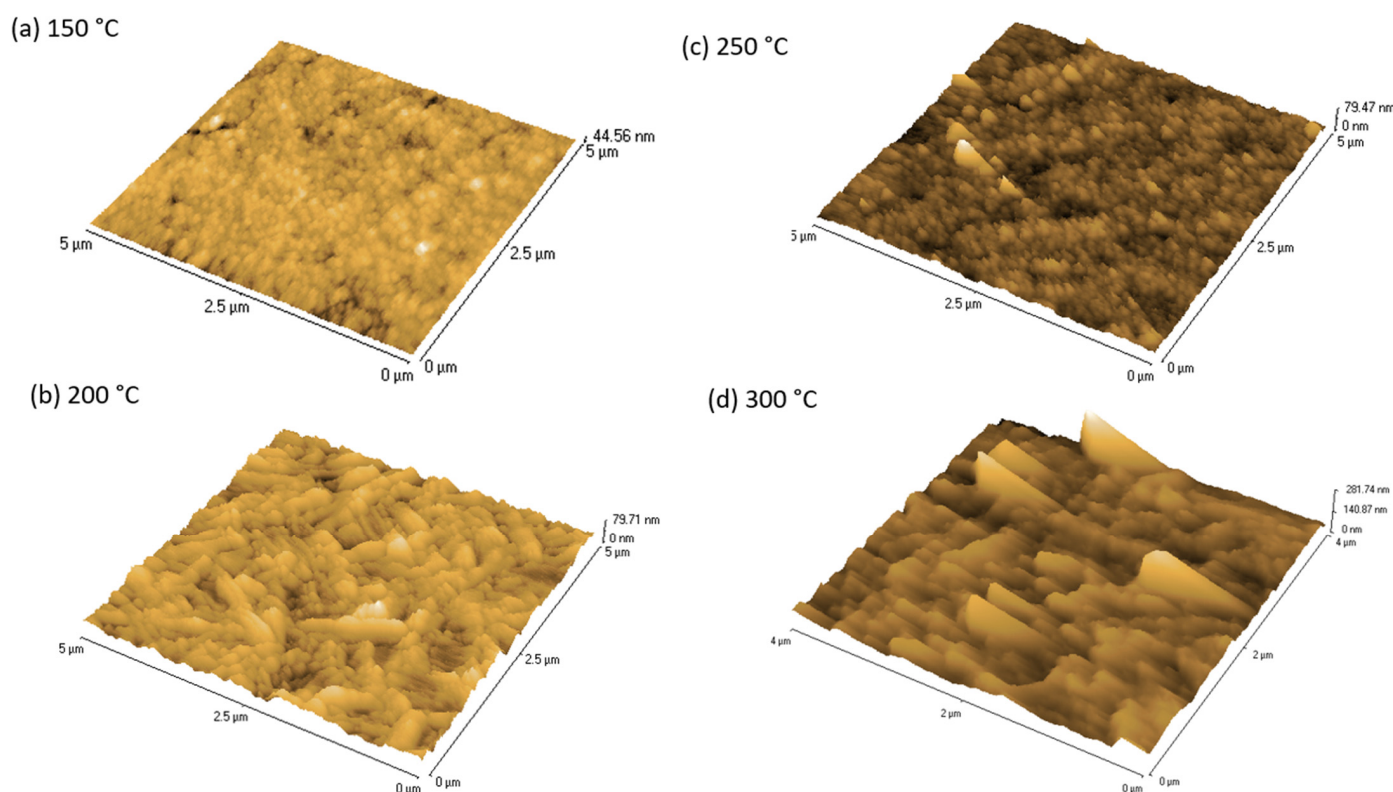


Figure 3. AFM images of Sb₂Se₃ thin films annealed at different temperatures.

Raman spectra were collected on the annealed samples at different temperatures, as shown in Figure 4. All samples exhibit a dominant vibrational mode at $\sim 190 \text{ cm}^{-1}$, characteristic of Sb₂Se₃, confirming its presence in all films [22,33,34]. An additional Raman peak at $\sim 253 \text{ cm}^{-1}$ appears only in the sample annealed at 300 °C, indicating a temperature-

induced structural change, which is consistent with the phase evolution suggested by XRD and EDX results.

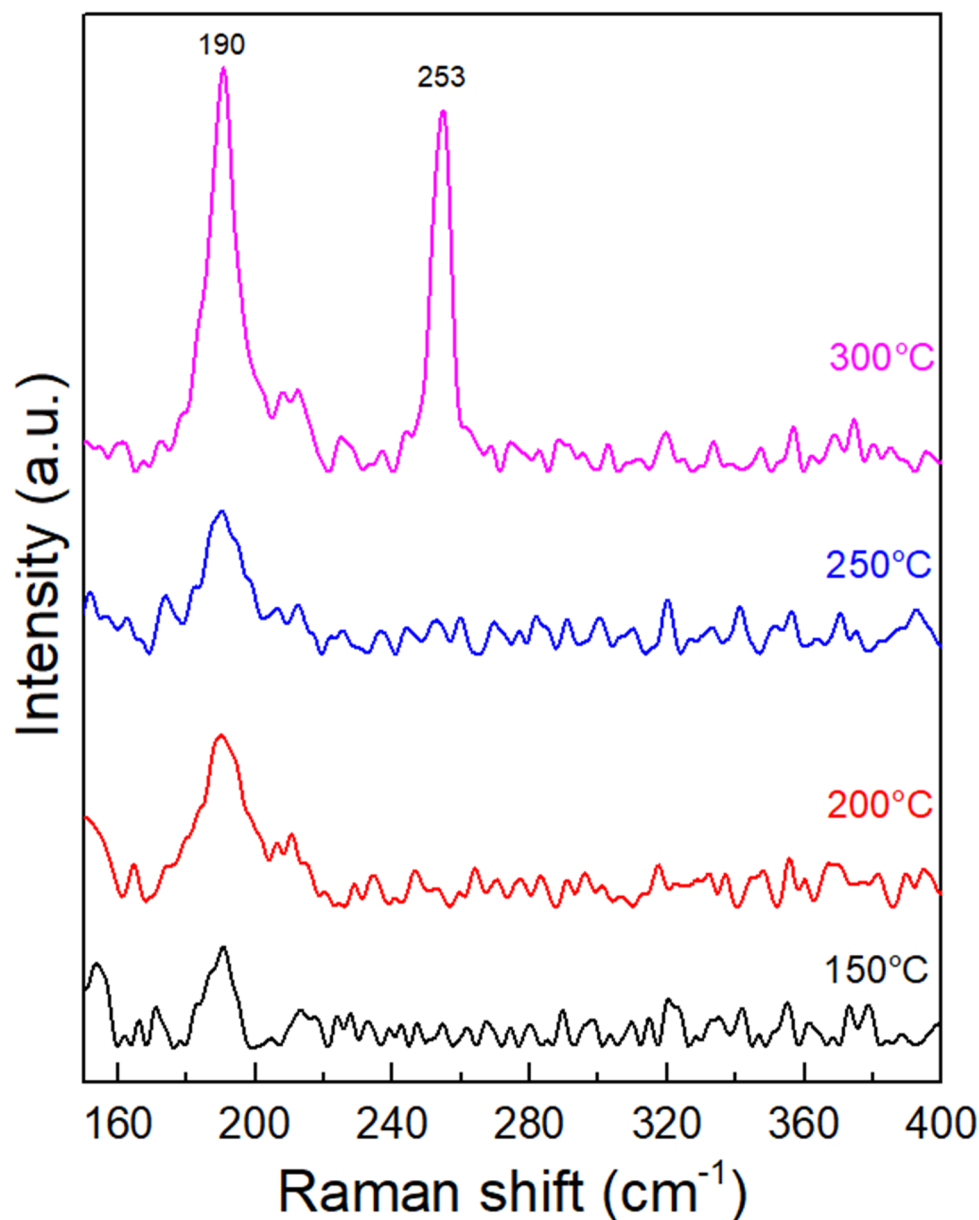


Figure 4. Raman spectra of Sb_2Se_3 thin films annealed at different temperatures.

The UV–VIS–NIR spectrometer was used to study the optical properties of the annealed Sb_2Se_3 films. Figure 5 shows these films' reflectance spectra. Generally, the annealed films displayed a constant value of reflectance in the visible regions. This value is around 45% of the annealed films at temperatures less than 300 °C. However, it is around 15% of the films annealed at the highest temperature of 300 °C. This can be explained in terms of roughness. As the roughness increases, the reflectance reduces because of the light scattering effects in the visible region [35,36]. The annealed films' absorbance spectra are presented in Figure 6. The annealed films notably absorb in the visible regions with the same value of around 50%, except for the sample annealed at the highest temperature of 300 °C, where the value is around 80%. The absorption spectra show weak peaks in the 900–1000 nm region. These features are attributed to thin-film interference effects arising from multiple reflections within the films rather than to intrinsic electronic transitions. Since the absorbance was calculated from the measured transmittance and reflectance

spectra according to ($A = 1 - (T + R)$), interference-induced oscillations in (T) and (R) can appear as peaks in the calculated absorbance spectra. The magnitude and position of these features depend on film thickness.

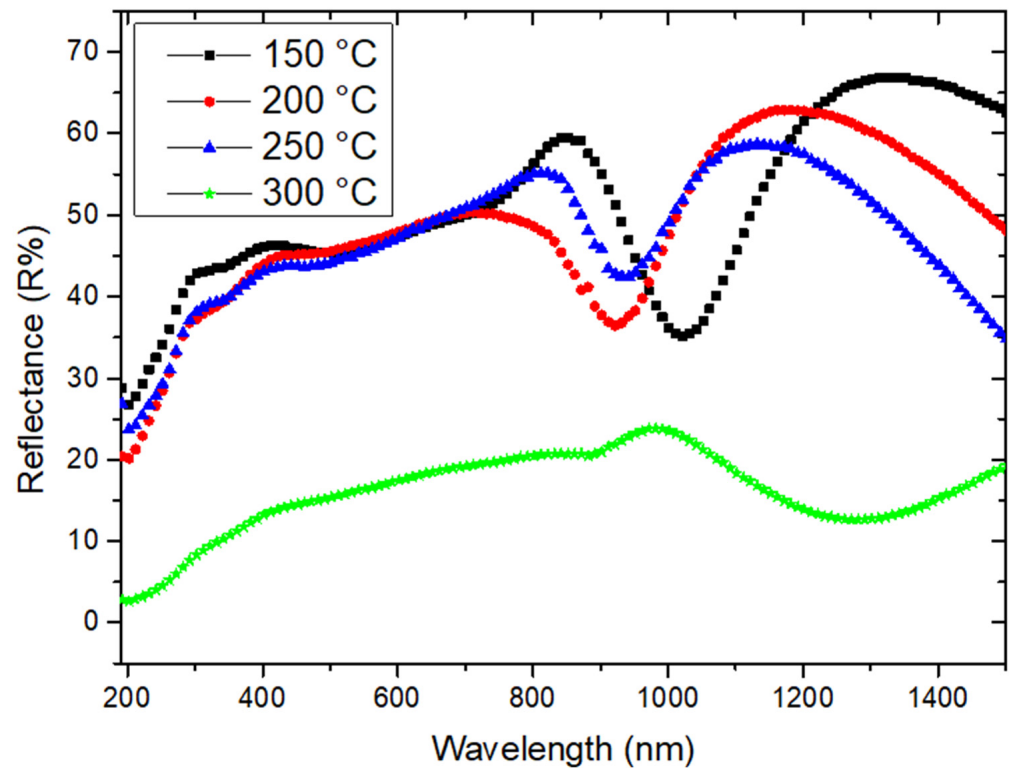


Figure 5. Reflectance spectra of Sb_2Se_3 thin films annealed at different temperatures.

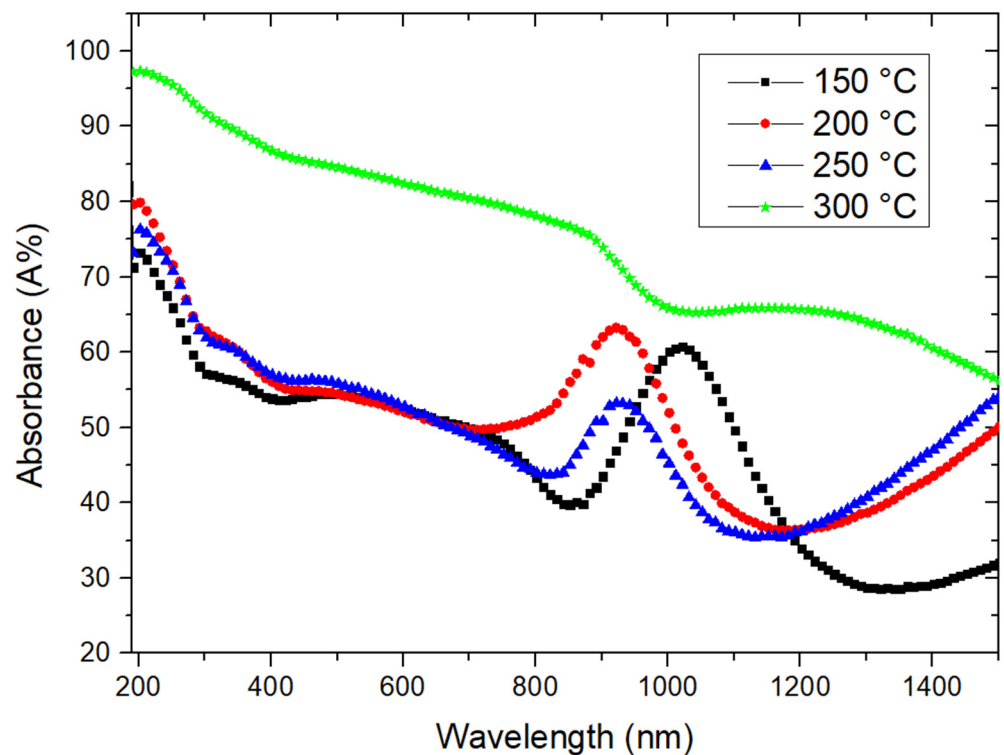


Figure 6. Absorbance spectra of Sb_2Se_3 thin films annealed at different temperatures.

The following formula was used to calculate the annealed films' absorption coefficient (α):

$$\alpha = \frac{1}{d} \ln \left\{ \frac{(1-R)^2}{2T} + \left[\frac{(1-R)^4}{4T^2} + R^2 \right]^{1/2} \right\}$$

where d is the thickness of the annealed film, and T is the optical transmission [37]. The annealed films generally display a high coefficient α of 10^5 cm^{-1} , which agrees with the stated value in scientific papers [2,4]. The annealed films' optical energy gap (E_g) was estimated using Tauc's relation:

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

where ν is the light frequency, and A is a constant. Figure 7 illustrates the plot of $(\alpha h\nu)^2$ against $h\nu$ of the annealed films at different annealing temperatures. The optical band-gap (E_g) values were estimated to be 1.25 eV, 1.00 eV, 1.30 eV, and 1.21 eV for films annealed at 150 °C, 200 °C, 250 °C, and 300 °C, respectively. These values are generally consistent with the reported band-gap range of stoichiometric Sb_2Se_3 thin films (1.1–1.3 eV) [1–4,8]. The slight deviation observed, particularly for the sample annealed at 200 °C, may be attributed to annealing-induced variations in crystallinity and defect density, which can influence localized band tailing and thus affect the optical absorption edge. Although the 150 °C sample exhibits the longest apparent absorption edge in the raw absorbance spectra (Figure 6), the absorption coefficient and band-gap analysis presented in Figure 7 account for film-thickness effects, providing a more reliable evaluation of the optical transition energy.

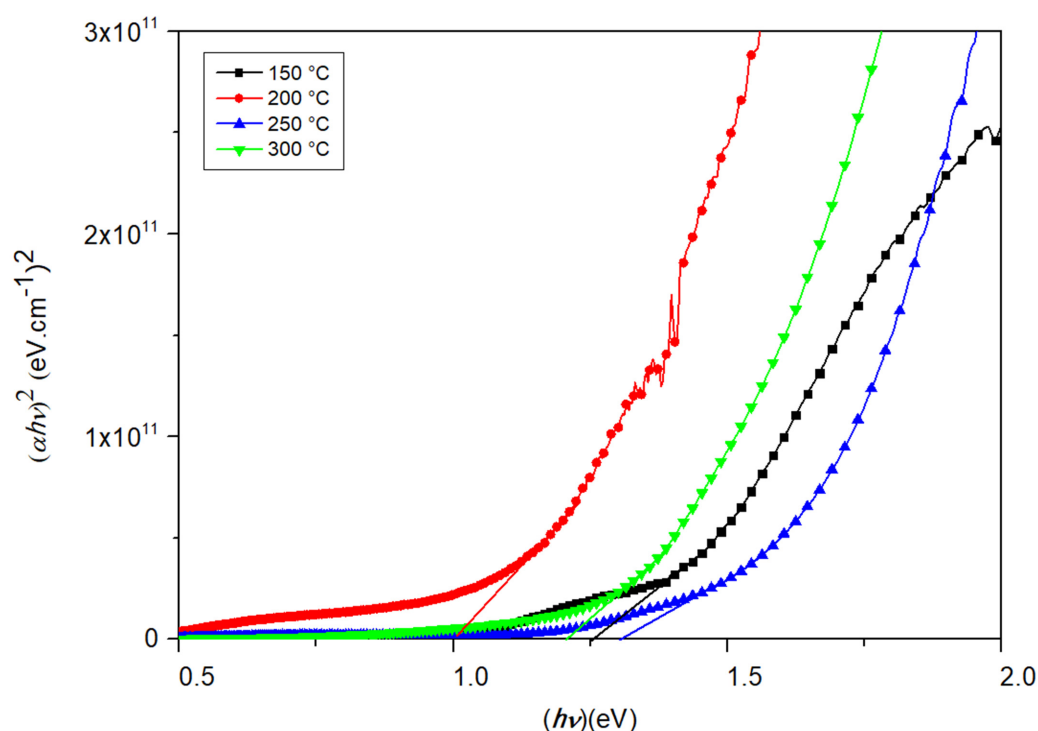


Figure 7. Annealed films' plot of $(\alpha h\nu)^2$ against $h\nu$ at different annealing temperatures.

The Hall-effect measurements of samples annealed at different temperatures are summarized in Table 3. The electrical properties show a strong dependence on annealing temperature, indicating that thermal treatment significantly modifies the carrier transport characteristics of the material. The sample annealed at 150 °C exhibited an electron-

dominated conduction mechanism, as indicated by the negative Hall coefficient, with a carrier concentration of $7.15 \times 10^{17} \text{ cm}^{-3}$ and a mobility of $1.63 \times 10^3 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. In contrast, samples annealed at 250 °C and 300 °C showed positive Hall coefficients, indicating hole-dominated (p-type) conduction. The carrier concentration varied substantially with annealing temperature, ranging from $2.29 \times 10^{14} \text{ cm}^{-3}$ for the 300 °C-annealed sample to $7.15 \times 10^{17} \text{ cm}^{-3}$ for the 150 °C-annealed sample. Such variations suggest that annealing alters defect populations, impurity activation, and charge compensation mechanisms within the material.

Table 3. Hall-effect parameters of the annealed thin films measured at room temperature.

Annealed Films	Carrier Concentration (cm^{-3})	Mobility ($\text{cm}^2/\text{V}\cdot\text{s}$)	Resistivity ($\Omega\cdot\text{cm}$)	Hall Coefficient (cm^3/C)	Type
150 °C	7.15×10^{17}	1.63×10^3	1.79×10^{-2}	-2.12×10^1	<i>n</i>
250 °C	1.17×10^{15}	3.88×10^3	7.50×10^{-1}	1.39×10^3	<i>p</i>
300 °C	2.29×10^{14}	3.34×10^2	1.60×10^2	6.18×10^4	<i>p</i>

The highest mobility was observed for the sample annealed at 250 °C, indicating improved carrier transport and reduced scattering. Meanwhile, the sample annealed at 300 °C exhibited a lower mobility and higher resistivity, which may be associated with increased defect scattering, grain-boundary effects, or annealing-induced structural changes. Overall, the Hall-effect measurements demonstrate that annealing temperature strongly influences carrier type, carrier concentration, mobility, and resistivity. The optimum annealing condition for carrier transport appears to occur near 250 °C, where the highest mobility was obtained.

4. Conclusions

In this study, four Sb_2Se_3 thin-film samples were successfully fabricated, each annealed at a different temperature (150 °C, 200 °C, 250 °C and 300 °C) to investigate the influence of thermal treatment on their structural, morphological, optical and electrical properties. XRD confirmed that orthorhombic Sb_2Se_3 remained the dominant phase in all films. Variations in diffraction peak intensity and preferred orientation revealed a strong dependence of crystallographic texture on annealing temperature. Additional diffraction features observed in the film annealed at 300 °C, together with Raman and EDX results, suggest structural changes associated with selenium loss at elevated temperatures. EDX analysis revealed a gradual selenium loss with increasing temperature, particularly obvious at 300 °C, attributed to the higher volatility of selenium. This compositional deviation may promote the formation of Sb-rich regions and influence the defect chemistry and carrier behavior. Optically, all films exhibited strong absorption in the visible region with absorption coefficients on the order of 10^5 cm^{-1} . The optical band-gap values ranged from 1.00 eV to 1.30 eV, consistent with the optimal range for photovoltaic applications. Theoretically, the optical response is governed by electronic transitions described by Tauc's relation, while variations in band gap can be associated with changes in crystallinity, defect density, and band tailing effects induced by annealing. Electrical characterization using Hall-effect measurements revealed a strong dependence of carrier transport on annealing temperature, including a transition in conduction type and significant variation in carrier concentration, mobility, and resistivity. Among all samples, the film annealed at 250 °C exhibited the highest mobility and the most favorable balance of electrical properties, indicating this temperature as the optimal annealing condition for enhanced charge transport.

When compared with typical thin-film semiconductor processing conditions, the identified optimal annealing temperature of 250 °C lies within a moderate processing temperature range, making it compatible with low-cost and substrate-sensitive photovoltaic fabrication processes. However, despite these promising properties, several practical limitations remain, including long-term environmental stability under ambient conditions and further process optimization for scalable deposition and device integration [38]. Furthermore, the annealing time was fixed at 30 min in this study to isolate the effect of annealing temperature on the film properties. We acknowledge that annealing duration is also a critical parameter that can significantly influence crystallinity, grain growth, and defect evolution in Sb₂Se₃ thin films. A systematic investigation of annealing time effects was beyond the scope of this work, but it is recommended for future studies to further optimize film quality and performance.

Overall, the combined structural, optical, and electrical analysis demonstrates that Sb₂Se₃ thin films exhibit promising properties for optoelectronic and photovoltaic applications. However, further work involving device fabrication, stability assessment, and process optimization (including annealing time and large-area uniformity) is required to fully establish their technological applicability.

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