



Structural and optical properties of Bi- and Sn-doped Sb_2Se_3 thin films prepared by thermal evaporation

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ABSTRACT

In this study, Sb_2Se_3 -based semiconductor thin films, both pure Sb_2Se_3 and those doped with Bi and Sn, were produced using the thermal evaporation method. The structural, morphological, and optical properties of the obtained films were investigated using XRD, SEM, and UV–Vis spectroscopy. While films without annealing exhibited an amorphous structure, significant crystallization was observed after annealing at 350 °C, especially at high doping levels. SEM analyses showed significant changes in surface morphology and grain structure depending on the doping level. Optical measurements revealed that absorption increased and the band gap changed with increasing doping level. The band gap for the undoped Sb_2Se_3 thin film was found to be 1.54 eV. An increase due to the Burstein–Moss effect was observed at low additive ratios, while band narrowing was observed at high additive ratios. The results obtained indicate that Bi and Sn additives improve the optoelectronic properties of Sb_2Se_3 thin films and hold potential for photovoltaic applications.

1 Introduction

Over the past half-century, rapid changes in semiconductor technology have led to significant improvements in the functional characteristics of all kinds of technological devices used in our daily lives, as well as making it easier to produce devices that are quite small in size [1]. Large switches used in electronic assemblies have been replaced by electronic circuit systems in very small packages that can automatically and flawlessly perform switching tasks based on stimuli such as

light, heat, pressure, and humidity [1, 2]. Due to these technological developments, the importance of operating conditions is quite high in order for the electronic components within the system (photodiodes, solar cells, sensors, detectors, etc.) to perform their tasks flawlessly and further improve the functionality of the devices [3]. External factors in the environment, such as humidity, temperature, pressure, and speed, cause negative effects on the components in the system, such as wear, corrosion, and roughness, which reduce the performance of the electronic circuit components [4].

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To prevent such situations, it is very important that the components used in the circuits are resistant to these effects and are not affected by them. Recent studies have focused particularly on p-type and magnetic semiconductors, nano- and ZnO-based materials [2]. Today, doping or coating methods are generally used in the production methods of these components. Among these methods, thermal vapor deposition offers distinct advantages over other coating methods, such as providing a more homogeneous distribution, enabling control over coating thickness, eliminating the need for any chemical solvents for coating, and achieving a smoother surface [5–7].

Particularly in recent scientific studies, antimony triselenide (Sb_2Se_3) and antimony sulfide (Sb_2S_3) compounds are widely used in photovoltaic cells–solar cells due to their advantages such as being easily accessible and inexpensive, recyclable, non-toxic, as well as their absorptive properties in solar cells and the ability to adjust the forbidden energy band [8–11]. Many recent studies have shown that thin-film coatings made with selenide compounds such as (Sb_2Se_3) and (Sb_2S_3) cause an increase in the existing crystal grain size of the material and bring about positive changes in the material's structure by providing advantages such as easy controllability of these structures [11, 12]. Among these compound types, the Sb_2Se_3 compound has attracted increased interest due to its excellent properties in terms of switching, thermoelectricity, and photovoltaics, owing to its orthorhombic crystal structure [13]. It should be noted that thermal treatments performed below 200 °C in selenization-type compounds result in an irregular structure in long-range structures of such materials, depending on the distance between the substrate and the coating, and that this causes the forbidden energy gap (E_g) value to be high [14]. It should be noted that the ease of application of the thermal vapor deposition method used, its ability to control the thickness of the coating applied, and the fact that it exhibits a change similar to a structural change in an alloy depending on the heat treatment performed increase the importance of this technological coating method.

In a thin-film study conducted by Ghosh and his colleague, they stated that the thin-film coating they made with the Sb_2S_3 compound, which has a crystalline structure and an amorphous structure, created continuity in the regularity of the coating, strengthened the bond structures of p electrons with a weak bond structure, increased the number of electronic

transitions occurring between bands, and caused a significant change in the existing optical properties of the material [15]. In another study, Kamruzzaman and colleagues reported that a coating made with antimony triselenide (Sb_2Se_3) had an amorphous structure under normal conditions, but after a half-hour heat treatment at 290 °C, the thin-film structure transformed from an amorphous structure to a polycrystalline structure. They found that this process resulted in an increase in the amount of light absorbed and that this caused a significant decrease in the amount of reflected light, leading to a notable improvement in optical properties [16]. It is clearly stated that the most significant limitations of Sb_2Se_3 and similar chalcogenide thin films are low carrier density, limited electrical conductivity, and high defect levels. Doping with a third metal is commonly used to address these issues. For example, Sn doping has improved electrical conductivity by increasing carrier density in Sb_2Se_3 thin films and enabled the adjustment of the conduction type [17]. Furthermore, Sn doping has been reported to reduce the effect of intrinsic defects that limit the photovoltaic performance of Sb_2Se_3 [18]. Bi doping contributes to narrowing the bandgap, increasing the optical absorption coefficient, and improving crystal quality in chalcogenide thin films [19]. Similarly, it has been demonstrated that the optoelectronic properties of Bi-doped semiconductor thin films obtained by thermal evaporation are significantly improved [20]. This study investigates the effect of Bi and Sn dopants and thermal treatment on Sb_2Se_3 thin films produced by thermal evaporation and aims to achieve bandgap tuning, enhanced absorption, and controlled crystallinity with suitable Bi and Sn dopants.

2 Experimental procedure

This study involved the synthesis of Sb_2Se_3 -based semiconductor alloys doped with Bi and Sn, resulting in the production of thin films. Bi and Sn were chosen as dopants because their atomic radii (Bi 143 nm; Sn 145 nm) are similar to those of Sb (133 nm), and they can significantly change the band structure. Using the general formula and an unalloyed Sb_2Se_3 reference sample, we generated the alloys $(\text{Sb}_{1-y}\text{Bi}_y)_2\text{Se}_3$ and $(\text{Sb}_{1-y}\text{Sn}_y)_2\text{Se}_3$ ($y = 0.01$ and 0.05). Sample codes are given here according to the following order: $(\text{Sb}_{1-y}\text{Bi}_y)_2\text{Se}_3$ group: $y = 0.01 \rightarrow 1\%\text{Bi}$, $y = 0.05 \rightarrow 5\%\text{Bi}$ and $(\text{Sb}_{1-y}\text{Sn}_y)_2\text{Se}_3$ group: $y = 0.01 \rightarrow 1\%\text{Bi}$,

$y = 0.05 \rightarrow 5\% \text{Bi}$. The element selenium is stable at room temperature; however, it can oxidize in strong oxidizing agents or biological environments and exhibits significant volatility (sublimation or evaporation) at high temperatures. Therefore, an additional 2% Se has been added to compensate for possible thermal losses during the production process. The study used Sb, Se, Bi, and Sn elements with 99.99% purity as the starting material. They mixed the measured elemental powders together for half an hour to make them all the same. The mixtures were heated in a closed system in a vacuum, first to 350 °C for 6 h, then to 600 °C at a rate of 3 °C/min. and kept at that temperature for 3 h. The samples were carefully cooled in the furnace after being heated, and the resulting alloy ingots were ground into powder to produce thin films. Thermal evaporation was used to make thin films. Under a vacuum of 6×10^{-5} mbar, alloy powders were placed in a tungsten pot and coatings were deposited onto glass substrates using a 240 A current. The substrates were washed in an ultrasonic bath with distilled water and acetone prior to coating. They were then dried in an oven. The thickness and evaporation rate were controlled during film evaporation. The thickness of the thin films is approximately 500 nm. The films were coated and then heated to 350 °C for 4 h in an vacuum to make the crystals grow better and the optoelectronic properties better. Therefore, it is known that selecting the annealing temperature within the range of 200 to 400 °C causes the Sb_2Se_3 thin film to crystallize from its amorphous phase. For this reason, the annealing temperature was selected as 350 °C [16, 21]. X-ray diffraction (XRD, Rigaku MiniFlex) was employed to study the structure of the produced alloys and thin films, focused ion beam–scanning electron microscopy (FIB-SEM, Thermo Fisher Scientific Scios 2 DualBeam) was used to examine their surface morphologies, and UV–Vis spectroscopy (Shimadzu UV-2600) was utilized to investigate their optical properties.

3 Results and discussion

Figure 1a illustrates the X-ray diffraction patterns of untreated Sb_2Se_3 alloy thin films compared with those of Bismuth (Bi)- and Tin (Sn)-doped alloys. The X-ray diffraction patterns of the heat-treated Sb_2Se_3 alloy and thin-film alloys doped with 5% Bi and Sn are illustrated in Fig. 1b. All measurements were conducted at ambient temperature, within the 0–80° scanning

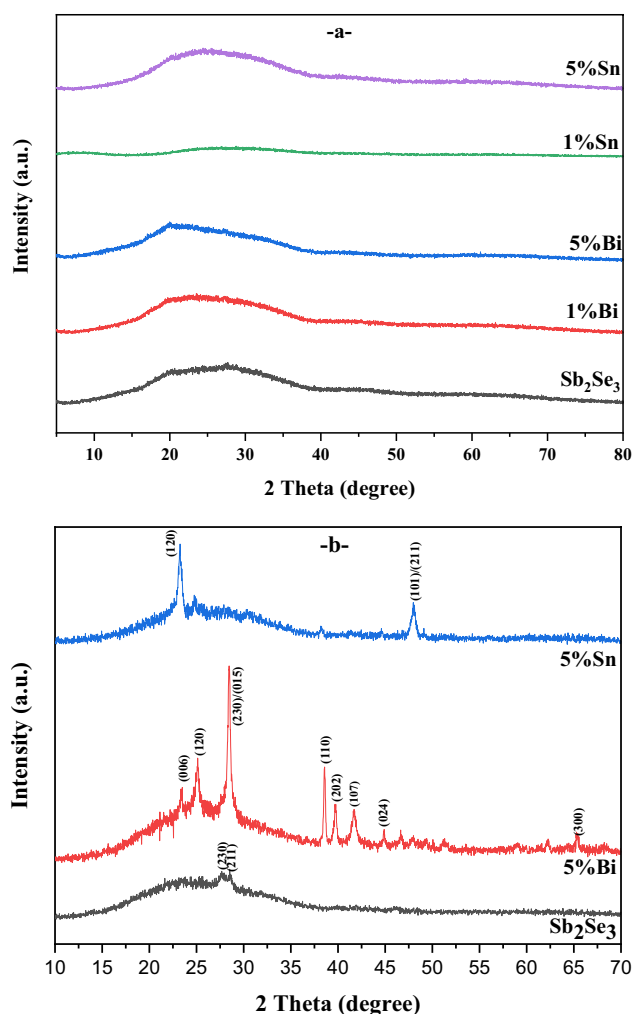


Fig. 1 X-ray diffraction (XRD) patterns of Sb_2Se_3 -based thin films: **a** as-deposited (non-heat-treated) and **b** heat-treated samples

range, and at a scanning rate of 6°/min. The absence of clear and defined crystalline diffraction peaks in the X-ray diffraction patterns of the primary chalcogenide Sb_2Se_3 alloy thin film, prepared without annealing, as well as in the thin-film alloys doped with bismuth (Bi) and tin (Sn), clearly shows that the synthesized thin films possess an amorphous structure. The non-crystalline XRD patterns have a predominant single-step, broad hump shape. This extensive and low-intensity peak arises from the amorphous nature of the Sb_2Se_3 alloy applied to the thin sheets [22, 23]. X-ray diffraction patterns of Sb_2Se_3 alloy thin films containing the highest levels of bismuth (Bi) and tin (Sn) and Sb_2Se_3 thin films subjected to annealing at 350 °C revealed new diffraction peaks indicating the presence of a

crystalline structure, in contrast to the broad hump characteristic of an amorphous structure. The formation of these peaks indicates that annealing facilitates the rearrangement of the atomic structure, promoting the formation of the crystalline phase. The increase in the intensity of these peaks after annealing reveals that the degree of crystallinity in the alloy thin films has increased and the atomic order has improved significantly. This is associated with the increased mobility of atoms with rising temperature and the overcoming of energy barriers, allowing atoms to settle into more stable and regular crystalline positions [24]. The indexing of the peaks appearing in the X-ray diffractograms of these heat-treated samples was performed using JCPDS cards found in the literature [25–31]. The primary purpose of post-production annealing is to promote the transformation of amorphous or weakly crystallized Sb_2Se_3 precursor films into well-crystallized films. This is done to achieve large grain sizes and vertical orientation. Research has shown that it is necessary to adjust the annealing temperature and annealing time to achieve the ideal balance between

crystallinity and compositional homogeneity [21]. For example, Wang and colleagues studied the thermally induced structural transformations of Sb_2Se_3 . They found that the number and intensity of X-ray peaks increased when examining Sb_2Se_3 thin films at low temperatures (approximately 100 °C) and those heat-treated at high temperatures (400 °C) [32]. This result emphasizes that the structural change of Sb_2Se_3 is strongly dependent on the annealing temperature.

The surface morphologies of pure Sb_2Se_3 and Bi- and Sn-doped Sb_2Se_3 -based samples were examined using focused ion beam scanning electron microscopy (FIB-SEM). The FIB-SEM image of the pure Sb_2Se_3 thin film (Fig. 2a) shows that the film surface has a homogeneous and continuous morphology on the substrate. The observed packet-like morphology, consisting of small grains, is indicative of an amorphous structure [33]. Figure 2b shows FIB-SEM images of bismuth and tin-doped Sb_2Se_3 -based thin films and chemical mapping images showing the Bi-Sn content. These small grains observed in the pure Sb_2Se_3 thin film have coalesced with a one percent bismuth addition,

A

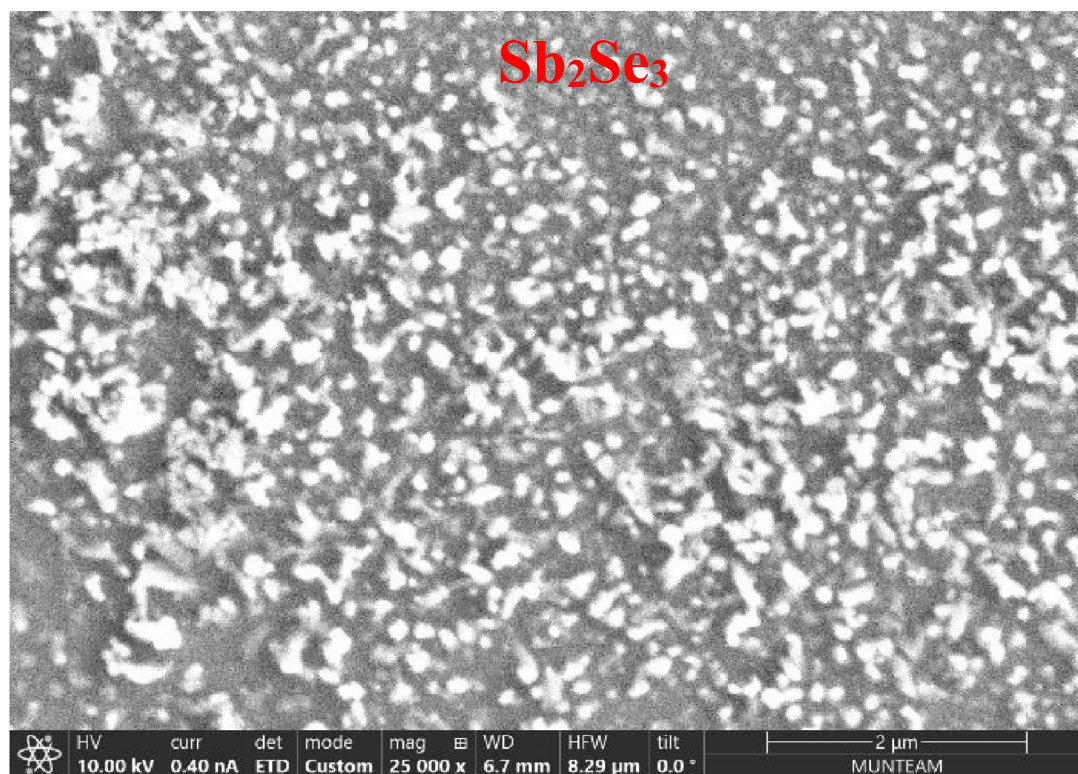
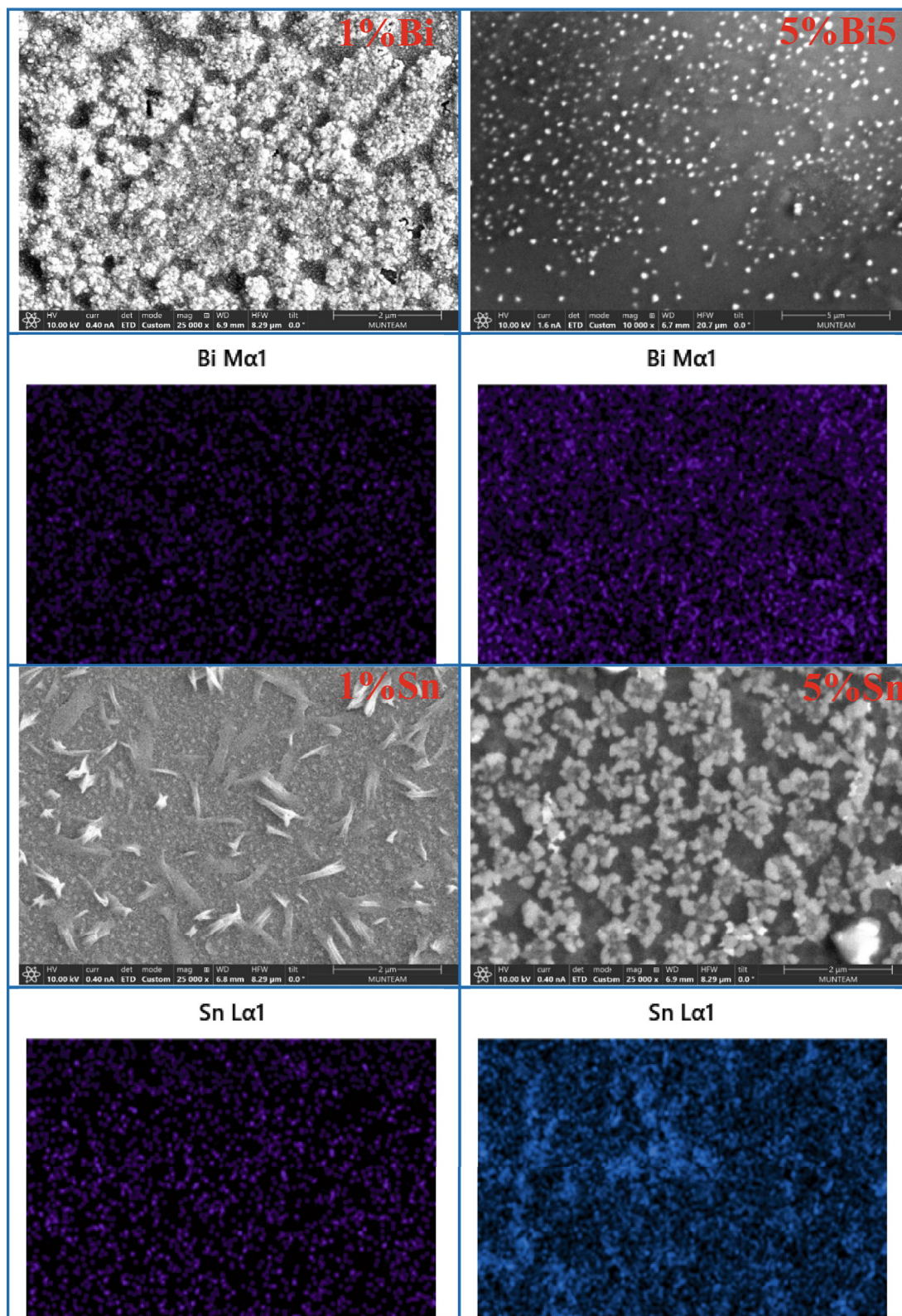


Fig. 2 **a** Focused ion beam scanning electron microscopy (FIB-SEM) image of the Sb_2Se_3 alloy thin film. **b** Focused ion beam–scanning electron microscopy (FIB-SEM) image and corresponding elemental mapping of the Bi/Sn-doped Sb_2Se_3 alloy thin film

B**Fig. 2** continued

and agglomerations have begun to form. In addition to the bismuth addition, the annealing temperature is also thought to be effective [24]. The 5% bismuth addition is thought to contribute to a more regular and smoother microstructure by limiting grain growth in the surface morphology. This can be attributed to the substitution of Bi atoms at Sb lattice sites, which alters the crystal growth kinetics. Yaseen and colleagues doped Sb_2Se_3 thin-film alloys with varying amounts of bismuth and observed that the surface became smoother and more isotropic; they suggested that this smoother surface could be more suitable for optical devices as it could reduce optical losses caused by scattering. Furthermore, it was observed that as the concentration of the additive particles increased, their density also increased, and consequently, the compactness of the films increased [34]. The grain development in Sn-doped Sb_2Se_3 thin films is affected by the dopant concentration and the post-deposition annealing procedure. At little Sn content (1%), restricted nucleation leads to rod-like and poorly crystalline structures. Elevating the Sn concentration facilitates nucleation and atomic diffusion, thereby improving crystallinity. Additionally, thermal annealing supplies adequate energy for grain coalescence and expansion. Consequently, the morphological alteration arises from the complementary influence of Sn incorporation and heat processing [21, 35]. A study in which Sn was added

to a ZnO thin film also found this structure, which looks like a nanorod. As the Sn concentration in the main thin film goes up, the nanorods start to cross-link strongly, which changes the surface morphology of the thin film [36]. The 5% Sn sample, on the other hand, seems like it has a granular surface because the little particles show out more and the shape looks like a group of them. This change illustrates that the increased Sn content makes it easier for atoms to move around, which makes nucleation density higher and crystallization happen faster. The literature says that adding more additive elements can make grain boundaries stronger, make the surface rougher, and make secondary phases arise in thin-film systems. So, the shift in form demonstrates that Sn atoms dissolve into the matrix when there are not many additives, but they promote phase separation and grain expansion when there are a lot of additives [37]. In addition, for both bismuth and tin additions, it was observed that as the addition ratios increased, the distinctiveness of these elements in the mapping images increased, and it was concluded that a homogeneous distribution was achieved.

Figures 3a and b show the change in absorbance and transmittance of Sb_2Se_3 thin films and Sb_2Se_3 matrix thin films doped with increasing amounts of bismuth and Sn. In these curves, there is an inverse relationship between absorbance and transmittance. Absorbance

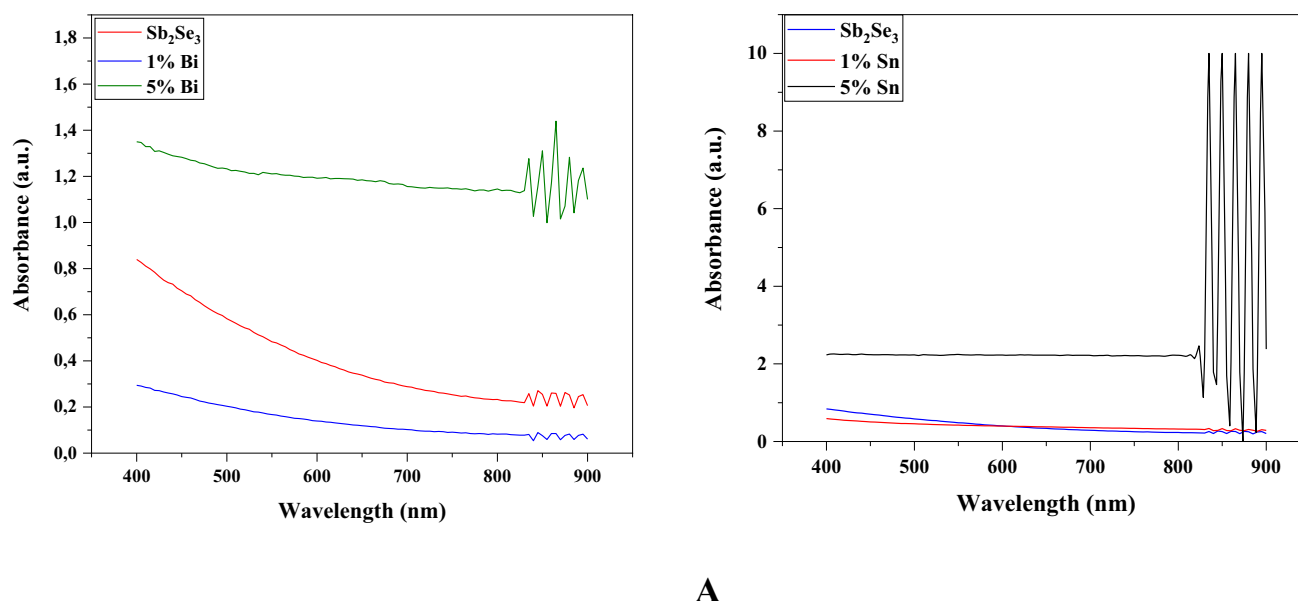


Fig. 3 **a** Optical absorbance spectra of Sb_2Se_3 thin films and Bi/Sn-doped Sb_2Se_3 thin films as a function of wavelength. **b** Optical transmittance spectra of Sb_2Se_3 thin films and Bi/Sn-doped Sb_2Se_3 thin films as a function of wavelength

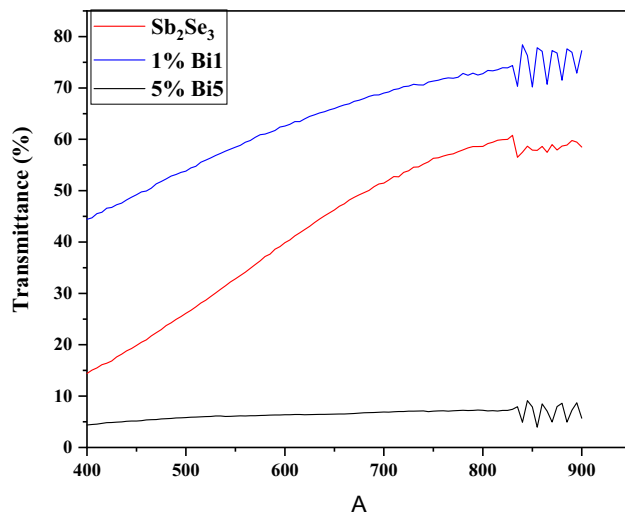
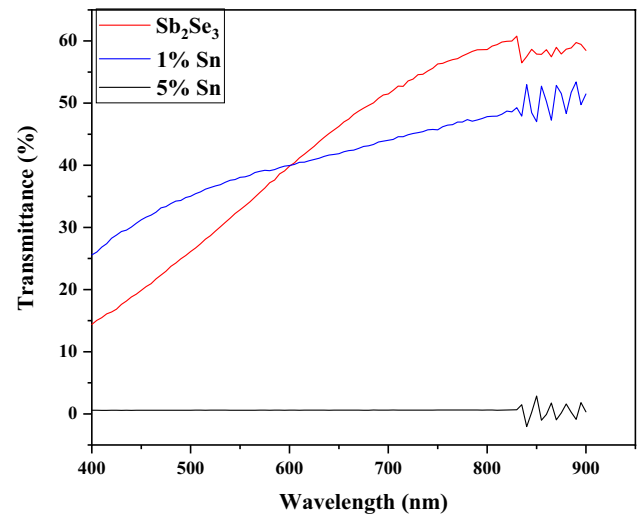


Fig. 3 continued

decreases with increasing wavelength, while transmittance increases with increasing wavelength. The reason for this is that the decrease in transmittance is due to the dopants increasing the absorption power of the film. This decrease in optical transmittance is due to the amorphous-to-crystalline phase transformation, as reported in various other studies. It is the destructive interference of light transmitted through the high and irregular grains of films that create light-scattering non-flat surfaces. Therefore, the decrease in optical transmittance can be attributed to the larger grain size of the films [38]. Transmittance spectra show interference bands with a sharp drop at the band edge in the medium and weak absorption regions. Optical absorbance is highly sensitive to particle size changes and crystallinity improvement [39]. Another noteworthy feature in the absorbance and transmittance curves is the fluctuations observed after 800 nm. In the long-wavelength region with a low absorption coefficient, these reflections interfere with each other, resulting in periodic absorbance fluctuations (interference fringes) in the UV-Vis spectra. These fluctuations originate from dangling bonds [40].

The Tauc method was used on the absorbance data to calculate the optical band gap (E_g) and learn about the electronic transitions. Using the absorbance data obtained from UV-Vis measurements, the optical bandgap of semiconductor thin films was calculated using the Tauc approach [41]:

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

The Tauc parameter, denoted by 'A,' is a constant that quantitatively expresses the disorder within the crystal structure. It is influenced by the probability of electrical transitions and the distribution of band density tails. Here, $n = 2$ is used for indirect transitions, while $n = 0.5$ is used for direct transitions in the calculation of the forbidden energy band of semiconductors. The Tauc plot is a graphical representation of the relationship between the square of the absorption coefficient $(\alpha h\nu)^n$ and the photon energy ($h\nu$). The band gap energy (E_g) of a material can be determined by the point where the slope of the Tauc plot intersects the x-axis (Fig. 4). A change in the slope indicates a corresponding change in the band gap energy (E_g). UV-Vis spectroscopy provides critical information for evaluating the effect of dopant elements on the band structure and optical transitions of Sb_2Se_3 thin films. The band gap (E_g) and optical absorption properties of Bi- and Sn-doped thin films have also been determined for indirect transitions, and the effect of the type and ratio of the dopant element on the band structure has been investigated. The E_g value for pure Sb_2Se_3 thin film was found to be 1.54 eV. This value is consistent with the "suitable band gap" window reported for Sb_2Se_3 thin-film photovoltaic absorbers. The literature emphasizes that the band gap in Sb_2Se_3 and $\text{Sb}_2(\text{S},\text{Se})_3$ systems can be adjusted over a wide range

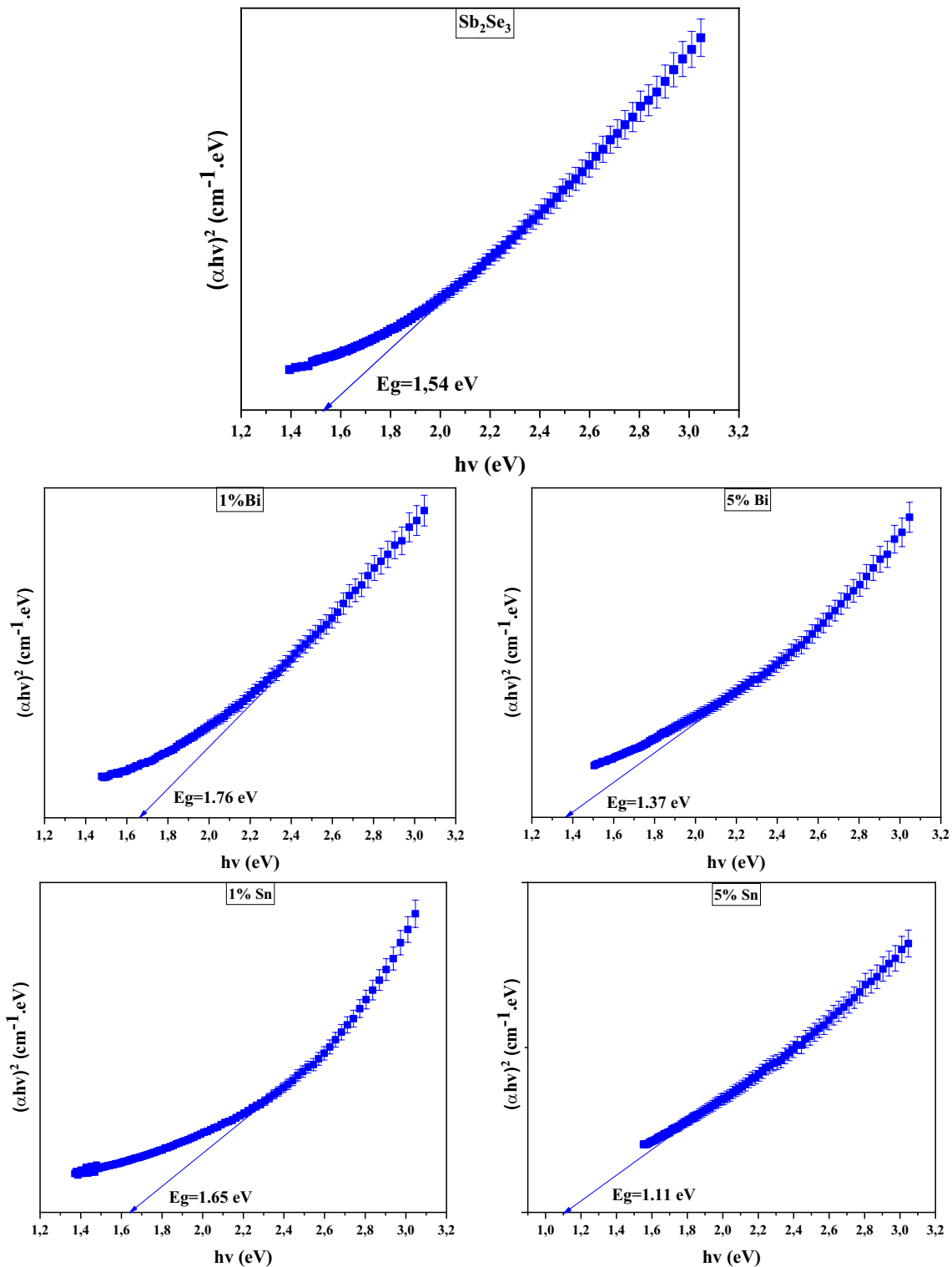


Fig. 4 Tauc plots of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) for Bi/Sn-doped Sb_2Se_3 thin films

(approximately 1.1–1.8 eV) depending on the composition, production conditions, and heat treatment temperature, and that it is one of the fundamental

parameters determining device performance [25, 42, 43]. Muiva and colleagues added increasing amounts of antimony to selenium and determined the forbidden

energy band gap of the thin films they produced. They found an E_g value of around 1.90 eV in thin films with a high selenium content [44]. Bi contribution at low levels (1%Bi) raises the E_g to 1.76 eV, while at high levels (5%Bi) the E_g drops to 1.37 eV. The increase in band gap observed at low doping levels occurs because electrons occupy states within the conduction band, raising the Fermi level to a higher energy. This results in a slight increase in the band gap observed in optical studies. This phenomenon is called the Burstein–Moss effect or Burstein–Moss shift [17, 45]. Similar changes have been observed in Sb_2Se_3 thin films containing low-concentration tin and high-concentration tin. A comparable methodology has been employed, indicating that element substitution influences the electrical structure in doped Sb_2Se_3 thin films. The reduction in the band gap correlates with enhanced absorption, especially in the longer wavelength (lower energy) region of the spectrum. Enhanced absorption of lower energy markedly elevates gadget performance. The reduced bandgap allows the material to absorb a broader photon spectrum from incident sunlight. Increased photon collection over a broader spectral range leads to a higher electron–hole pair generation rate. As a result, an increasing number of charge carriers accumulate, leading to a high short-circuit current density in the solar energy device. Ali AK. Bakly and colleagues also doped Sb_2S_3 thin films with calcogenic properties, such as Sb_2Se_3 , with barium and reported that the barium doping altered the optical bandgap behavior and that the bandgap shifted to higher energies with increasing carrier concentration, consistent with the Burstein–Moss effect [46]. M. S. Abo Ghazala and colleagues attributed this behavior to a change in the crystal's ionic nature as the second reason, suggesting that the substitution of the alloying element for the main alloying element in the crystal lattice of the main alloy causes an observable change in E_g [47]. Increased absorption, particularly at low photon energies, leads to an increase in external quantum efficiency over a broader spectral range, meaning that incoming photons are converted into electric current with higher efficiency. The narrowing of the bandgap observed with Bi and Sn dopants can enhance device performance by facilitating more efficient light absorption, leading to increased photon carrier production and ultimately improved current output in potential optoelectronic applications [48]. As a second approach, this decrease in E_g is also explained by the Mott and Davis model. The decrease in E_g observed with an increase

in Bi percentage can be well explained by the Mott and Davis model: the increase in regional density of thin films produced on glass is effective. Defects in thin films caused by some dangling bonds (band tails) are responsible for the formation of regional states. Most studies have shown that amorphous materials transform into crystalline materials under thermal annealing [23, 24, 49]. Therefore, some dangling bonds form during the crystallization process. The formation of defects associated with these dangling bonds causes the bands to extend toward the band gap region. In other words, thermal annealing increases the number of dangling bonds by separating larger crystals into smaller ones. Wang and colleagues investigated the effect of annealing temperature on Sb_2Se_3 thin films and observed that the width of the band tail decreased as the degree of film ordering increased, as determined by Raman analysis. They explained this decrease using the Mott and Davis state model, suggesting that the increase in Sb content, resulting from a decrease in the band tails and band gap due to Sb's lower electronegativity compared to Se in the thin film, causes an increase in the concentration of localized states [32]. Consequently, heat treatment of films causes an increase in the energy width of local states and thus reduces the optical energy gap. In short, the heterogeneous growth of crystalline phases and the increase in metallic behavior reduce the optical gap in these films by increasing the density of defect states associated with the surface of the crystallites [50].

4 Conclusions

In this study, it has been demonstrated that thermal treatment applied with Bi and Sn doping can effectively control the structural order and band structure of Sb_2Se_3 thin films. The results obtained significantly increase the potential for Sb_2Se_3 -based thin films to be used as low-cost photovoltaic absorber materials with high absorption coefficients and tunable band gaps. XRD analyses revealed that the unannealed doped and undoped films were predominantly amorphous in structure. However, after thermal treatment at 350 °C, distinct crystalline phase formation was observed, particularly in samples with high Bi and Sn doping. This indicates that annealing plays a critical role in enhancing crystallization and atomic order. SEM examinations showed that unalloyed Sb_2Se_3 thin films have a homogeneous

and continuous surface morphology. With increasing alloying ratio, grain growth, surface roughness, and distinct morphological transformations occurred. Elemental mapping results confirmed that Bi and Sn alloying elements were homogeneously distributed within the Sb_2Se_3 matrix. UV–Vis measurements have revealed that absorption increases and transmittance decreases with increasing dopant concentration. This behavior has been attributed to structural transformations and an increase in particle size. The optical bandgap of the undoped Sb_2Se_3 thin film was determined to be 1.54 eV. While band gap widening was observed at low dopant ratios due to the Burstein–Moss effect, band narrowing occurred at high dopant levels. It was concluded that the narrowing of the band gap is related to increased defect levels, band tailing, and developing crystallization. It was determined that the change in the band gap was more pronounced in tin (Sn)-doped films, and the highest band gap values were obtained with Sn doping. This indicates that Sn doping is more effective than Bi doping in improving the optoelectronic properties of Sb_2Se_3 thin films. In conclusion, Sn doping was found to be more successful in terms of structural order, crystallization degree, and bandgap engineering, making it a more promising dopant element for Sb_2Se_3 -based photovoltaic applications.

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Author contributions

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Data availability

Data available for peer-reviewers under request.

Declarations

Conflict of 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 this paper.

Ethical approval Ethical approval was not required, as the study conducted did not involve any ethical concerns or issues.

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