Preparation and investigation of structure, optical, nonlinear optical and thermoelectric properties of Bi$_2$Se$_3$ thin film

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ABSTRACT

Bi$_2$Se$_3$ thin film was prepared using thermal evaporation. Structure and surface topography were investigated using both of Diffraction Electron Microscope (DEM) and Transmission Electron Microscope (TEM). Optical results confirmed that thin film has a direct energy gap. Moreover, the values for all of oscillating energy (E$_o$), dispersion energy (E$_d$) and ratio of the free carrier concentration on the effective mass (N/m$^*$) were determined optically. The dielectric constant ($\varepsilon'$) and tangent loss ($\varepsilon''$) were calculated. The density of states (DOS) for both of valence band (Nv) and conduction band (Nc) and also position of Fermi level were determined. The nonlinear optical results such as third-order optical susceptibility ($\chi (3)$), refractive index (n2) and absorption coefficient ($\beta_c$) were determined. The influence of temperature on IV results was studied; finally the dependence of all of Dispersion factor (D), parallel inductance (Lp) and Seebeck coefficient (S) values on temperature for this film was studied.

INTRODUCTION

Chalcogenide materials were widely investigated as a result of their electronic applications and also their linear and nonlinear optical properties [1–4]. Among all these binary chalcogenide is Bi$_2$Se$_3$ which is suitable for optical thermoelectric applications [5], solar cell [6], and photosensitive devices, photovoltaic cells, etc. [7–11]. Bi$_2$Se$_3$ thin films have been prepared using various techniques such as electro-deposition [12] and Successive Ionic Layer Absorption and Reaction (SILAR) [13] methods. The structure of Bi$_2$Se$_3$ thin films was studied [14–20]; it was found that these films had polycrystalline structure with rhombohedral structure [17,18], with space group (R-3 m) (166) [20]. Optical properties of Bi$_2$Se$_3$ thin films were investigated [21–27]. It is known that Bi$_2$Se$_3$ had an energy gap around 1.51 eV [21], and it ranges from 1.4 to 2.25 eV [22]. The energy gap of the film decreased with annealing to become 1.10 eV [23]. In addition the substrate temperature influence the energy gap to 1.25 eV [24]. Transmitted values increased with annealing temperature [26]. Many reports about the Bi$_2$Se$_3$ thin films were published and confirmed that the Bi$_2$Se$_3$ thin films have direct optical transitions and the electrical conductivity and current densities affected strongly with temperature and thicknesses [26–31]. Due to excellent thermoelectric properties of Bi$_2$Se$_3$ thin films and its application, some researchers were studied it widely [32–37], and reported that the temperature increased electron-hole generation [33]. Moreover, IV behaviour increased with annealing temperature [38–40].

Still the linear and nonlinear optical properties of Bi$_2$Se$_3$ thin films did not investigated in details, which motivated us to study the optical and nonlinear and thermoelectric properties of Bi$_2$Se$_3$ thin films for exploring the surface charge and the value of second harmonic generation.

In this paper, Bi$_2$Se$_3$ thin film was prepared using vacuum thermal evaporation technique. The structure and surface topography were investigated using both of Diffraction Electron Microscope (DEM) and Transmission Electron Microscope (TEM). Optical properties were measured for the Bi$_2$Se$_3$ thin film. Moreover, optical parameters including; oscillating energy (E$_o$), dispersion energy (E$_d$) and ratio of the free carrier concentration on the effective mass (N/m$^*$) were determined optically using the calculated values of refractive index (n). The dielectric constant ($\varepsilon'$) and tangent loss ($\varepsilon''$) were calculated. The density of states (DOS) and position of Fermi level were determined. The nonlinear optical results such as third-order optical susceptibility ($\chi (3)$), refractive index (n2) and absorption coefficient ($\beta_c$) were determined. The influence of temperature on IV results was studied; finally the dependence of all of Dispersion factor (D), parallel inductance (Lp) and Seebeck coefficient (S) values on temperature for this film was studied.

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inductance \((L_p)\) and Seebeck coefficient \((S)\) values on temperature for this film was studied.

**Experimental Work**

\(\text{Bi}_2\text{Se}_3\) material was prepared from \(\text{Bi}\) and \(\text{Se}\) pure elements with high purity (5 N). Due to some of elements are reactive at high temperatures with oxygen, synthesis was accomplished in evacuated silica tubes \(\sim 10^{-4}\)Torr and then sealed. The inner walls of silica tubes were coated with pure powder graphite, in order to prevent any reactions between the used elements and silica at elevated temperatures. The sealed silica tubes were heated inside a furnace up to 1150 K and kept at this value for about 10 hours. The homogenised melt was quenched in ice-water, to facilitate rapid quenching the specimens were sealed in tubes with a small diameter (about 2–3 mm). Thin films were prepared using vacuum thermal evaporation technique of type Edward – (EDWARDS E 306), the studied films were deposited under vacuum of \(10^{-3}\) torr. The structure of these films was studied using transmission electron microscope of type ”JEOL-TEM -1230-Japan made. The optical transmittance and reflectance spectra of these films were measured using double beam spectrophotometer with of type (JASCO corp., V-570) with wavelength range 300–2500 nm. Current-Voltage properties during illumination with UV-lamp and room light were taken from Virtins Instruments digital-computerised oscilloscope. The electrical measurements were done by Keithley 6517A electrometer.

**Results and discussions**

**Structure**

The structure and surface topography of \(\text{Bi}_2\text{Se}_3\) thin film were studied using both of \(\text{DEM}\) with different values of magnifications (dir. magnif.) as shown in Figure 1(a,b), (dir. magnif. = 2500x for (a) and dir. magnif. = 2000x for (b). The \(q\) spacing between layers for this sample were determined and tabulated in (Table 1), while (TEM) images with different values of direct magnifications (dir. magnif. = 2500x for (a) and dir. magnif. = 2000x for (b)) are shown in Figure 2(a,b). From this Fig it was noticed that, the grains were distributed homogeny over the film.

**Optical results**

The measured optical properties such as transmission \((T)\), Reflection \((R)\) and Absorption \((A)\) for this sample are shown in (Figure 3).

From Figure (3), it was seen that the values of both \((T\) and \(R)\) had a reverse behaviour (500–1000 nm), while these values identified from (1000–2500 nm). The absorption coefficient \((\alpha)\) of this investigated film was determined as [41]

\[
\alpha = \frac{n}{d} \ln \left[ \frac{1-T^2}{1-R^2} \right]
\]

(1)

Where \((d)\) is the film thickness. Figure 4(a) shows the relation between \((\alpha, h\nu^2)\) and photon energy \((h\nu)\) for this film, it is clear that, this film has a direct optical energy gap\((E_{g,dir}\) with value 1.85 eV (Table 2).

Urbach tail for these sample was calculated using the following Equation [42]

\[
\alpha = \alpha_0 e^{\frac{h\nu}{E_{u}}}
\]

(2)

where \((E_u)\) is the Urbach constant (tail). In order to determine the Urbach tail, the relation between \(\ln (\alpha)\) and \((h\nu)\) is shown in Fig. 4(b). The calculated \((E_u)\) value is in (Table 2).

The refractive index \((n)\) for this film was calculated as [43]

\[
n = \frac{(1+\sqrt{R})}{(1-\sqrt{R})}
\]

(3)

Figure 1. DEM Images with dir. magnif. = 2500x for (a) and dir. magnif. = 2000x for (b) for \(\text{Bi}_2\text{Se}_3\) thin films.
Table 1. The d-spacing for Bi$_2$Se$_3$ thin film.

| Spot | d-Spacing (nm) | Spot | d-Spacing (nm) |
|------|----------------|------|----------------|
| 1    | 0.3326         | 1    | 0.3843         |
| 2    | 0.3207         | 2    | 0.3877         |
| 3    | 0.3053         | 3    | 0.3345         |
| 4    | 0.2765         | 4    | 0.3241         |
| 5    | 0.2692         | 5    | 0.3356         |
| 6    | 0.2839         | 6    | 0.3462         |
| 7    | 0.2309         | 7    | 0.3379         |
| 8    | 0.2252         | 8    | 0.2858         |
| 9    | 0.2246         | 9    | 0.2907         |
| 10   | 0.2228         | 10   | 0.283          |

The oscillator strength ($f$) was determined as [47]

$$f = E_o - E_d$$  \hspace{1cm} (8)

Another important parameter is static refractive index ($n_o$), which was determined, as [48]:

$$n_o = \left[\frac{E_o}{E_d} + 1\right]^{-0.5}$$  \hspace{1cm} (9)

The values of both ($\varepsilon'$) and ($\varepsilon''$) for this sample were determined as [49]:

$$\varepsilon' = n^2 - k^2$$  \hspace{1cm} (10)

$$\varepsilon'' = (n^2 + k^2)^2 - (n^2 - k^2)^{0.5}$$  \hspace{1cm} (11)

Figure 4(c) shows the dependence of both ($n$) and extinction coefficient ($k$) on wavelength ($\lambda$). From this figure it is noted that, the ($n$) values decreased with ($\lambda$), while ($k$) values increased ($\lambda$), which give indication that, the absorption ability for this sample increased with wavelength ($\lambda$).

The single oscillator for the Bi$_2$Se$_3$ thin film can be expressed by [44].

$$n^2 - 1 = \frac{E_o - E_d}{E_o - (nh)^2}$$  \hspace{1cm} (4)

The values of ($E_o$) and ($E_d$) are obtained from the intercept on vertical axis ($E_o$) and the slope of ($E_o$, $E_d$)$^{-1}$ resulting from Figure 5(a). The calculated values of ($E_o$) and ($E_d$) for this film is shown in (Table 2).

The relation between ($n^2$) and ($\lambda^2$) for Bi$_2$Se$_3$ thin film is presented in Figure 5(b). The values of ($N/m^*$) for this sample was determined as [45].

$$n^2 - k^2 = \varepsilon L \left(\frac{\alpha}{4\pi^2\varepsilon_0 m^*}\right) \lambda^2$$  \hspace{1cm} (5)

Where ($\varepsilon_L$) is the lattice dielectric constant, ($\varepsilon_0$) is the permittivity of free space, the values of both of ($N/m^*$) and ($\varepsilon_L$) are shown in (Table 2).

The values of both ($M_{-3}$) and ($M_{-5}$) can be derived using the next equations [46]:

$$E_o^2 = \frac{M_{-3}}{M_{-5}}$$  \hspace{1cm} (6)

$$E_d^2 = \frac{M_{-5}}{M_{-3}}$$  \hspace{1cm} (7)

Figure 3. The optical measured parameters such as, Transmittance, Reflectance and Absorbance for Bi$_2$Se$_3$ thin film.

Figure 6 (a) shows the dependence of both ($\varepsilon'$) and ($\varepsilon''$) on ($hv$), from Figure (6) it was found that both ($\varepsilon'$) and ($\varepsilon''$) increase with ($hv$) as a result of increasing electrons mobility's.

Real part of optical conductivity ($\sigma_r$) and imaginary part of optical conductivity ($\sigma_i$) were calculated as [50]

$$\sigma_r = \frac{\varepsilon'' \omega}{2\pi}$$  \hspace{1cm} (12)

Figure 2. TEM Images with dir. magnif. = 2500x for (a) and dir. magnif. = 2000x for (b) for Bi$_2$Se$_3$ thin films.
\( \sigma_2 = \frac{(1-e^{-\chi})}{4\chi} \) \hspace{1cm} (13)

Figure 6(b) shows influence of \((h\nu)\) on both \((\sigma_1)\) and \((\sigma_2)\). It is clear from Figure 6 that both \((\sigma_1)\) and \((\sigma_2)\) increase with \((h\nu)\), while \((\sigma_1)\) increases strongly with \((h\nu)\), this gives indication, that this film had a high ability to absorb photons and increase its conductivity.

Linear optical susceptibility \((\chi^{(1)})\) describes the response of the material to photon energy, which determined as [51]:

\[ X^1 = \frac{\chi^{(1)}}{4\pi} \] \hspace{1cm} (14)

It was found that \((\chi^{(1)})\) increased with \((h\nu)\) as in Figure 6(c), also the values of \((\chi^{(1)})\) have a maximum

Table 2. The calculated results for Bi\(_2\)Se\(_3\) thin film.

| Fermi level Position (eV) | \(N_s\) | \(N_C\) | \(N/m^4\) | \(n_0\) | \((f) (eV)^2\) | \(M_{-3}\) (eV) | \(M_{-1}\) (eV) | \(E_d\) (eV) | \(E_a\) (eV) | \(E_o\) (eV) |
|--------------------------|--------|--------|-----------|-------|-------------|-------------|-------------|-------------|-------------|-------------|
| 0.17                     | 7.9E+21| 2.2E+22| 2.3E+51   | 01.48 | 21.92       | 02.28       | 04.68       | 05.12       | 02.22       | 04.20       | 1.85        |

Figure 5. (a) The dependence of \((n^2-1)^{-1}\) on \((h\nu)^2\) and \((b)\) the dependence of \(n^2\) on \(\lambda^2\) for Bi\(_2\)Se\(_3\) thin film.
value higher than \((E_g)\), this means that this material is a promise material for optical devices.

**Nonlinear optical properties**

The third-order nonlinear optical susceptibility \(\chi^{(3)}\) was determined as [52]:

\[
X^{(1)} = A \left\{ \frac{E_x \times E_y}{4\pi \varepsilon_0 (E_x^2 - h\nu^2)} \right\}^2
\]

(15)

Where \(A = 1.7 \times 10^{-10}\) c.s.u [53]. \(\chi^{(3)}\) increase with \((h\nu)\) as in Figure 6(c), this could attribute to the variation of free carrier concentration which leads to the increase of electrons mobility, while \((n_2)\) which resulted from nonlinear effects and determined as [54,55]

\[
n_2 = \left(12\pi X^{(3)}\right)/n_o
\]

(16)

The dependence of \((n_2)\) on \((\lambda)\) for this thin film is in Figure 7(a). The values of \((n_2)\) decrease with \((\lambda)\). Also \((\beta_c)\) was determined as [56]:

\[
\beta_c = \frac{4\pi^2 X^{(3)}}{n_2 h\nu^2}
\]

(17)

the values of \((\beta_c)\) increase with \((h\nu)\) as in Figure 7(b), as a result high number of excited electron which overcome band gap.

Both real and imaginary parts of the third-order nonlinear optical susceptibility \(\chi^{(3)}\) were determined as follows [57]

\[
\text{Re} \chi^{(3)}(\epsilon_{us}) = 10^{-4} \left\{ \frac{\varepsilon_0 \chi_n^2 \lambda_n^2}{\pi} \right\}
\]

(18)

\[
\text{Im} \chi^{(3)}(\epsilon_{us}) = 10^{-2} \left\{ \frac{\varepsilon_0 \chi_n^2 \lambda_n^2}{4\pi} \right\}
\]

(19)

The relation between both of \(\text{Re} \chi^{(3)}\) and \(\text{Im} \chi^{(3)}\) is shown in Figure 7(c), from the Figs it is clear that, both of \(\text{Re} \chi^{(3)}\) and \(\text{Im} \chi^{(3)}\) increase \((h\nu)\), this could be attributed to the increase of electorn mobility which gives advaced to high resopnse for changing optical properties.

Electrical susceptibility \(\chi(\epsilon)\) was determined using the following relation [58]:

\[
X_c = \frac{n^2 - k^2 - \varepsilon_r}{4\pi}
\]

(20)

Also relative permittivity \(\varepsilon_r\) was calculated as [59]:

\[
\varepsilon_r = (X_c + 1)
\]

(21)

The dependence of both of \(\chi(\epsilon)\) and \(\varepsilon_r\) on \((h\nu)\) for this thin film is shown in Figure 7(d). It is clear that the
values of both $\chi(e)$ and $\varepsilon_r$ with $h\nu$; this could be attributed to the increasing of electron mobility with $h\nu$.

**Semiconducting and electronic results**

The density of states (DOS) are calculated as follow [60]:

$$N_V = 2 \left( \frac{2nKTm^*_{h}}{h^2} \right)^{\frac{3}{2}}$$

$$N_C = 2 \left( \frac{2nKTm^*_{e}}{h^2} \right)^{\frac{3}{2}}$$

Where $N_v$ and $N_C$ are the density of states for both valence and conduction bands, respectively, effective mass of holes in (Bi$_2$Se$_3$) $m^*_{h} = 0.24m_0$ [61], effective mass of electrons in (Bi$_2$Se$_3$) $m^*_{e} = 0.12m_0$ [62].

The position of Fermi level was determined as [53]:

$$E_f = \left( \frac{KT}{q} \right) \ln \frac{N_C}{N_V}$$

Figure 7. (a) the dependence of $n_2$ on $\lambda$, (b) the influence of $h\nu$ on $\beta_c$, (c) the dependence of both of real $\chi^{(3)}$, $\text{Im}\,\chi^{(3)}$ on $h\nu$ and (d) the dependence of both of $\chi(e)$, $\varepsilon_r$ on $h\nu$ for Bi$_2$Se$_3$ thin film.

Figure 8. The influence of temperature on both of resistivity (a) and capacitance (b) for Bi$_2$Se$_3$ thin film.
The values of Fermi level position for this investigated thin film are shown in (Table 2).

**Electrical and I-V characterisation results**

The electrical resistivity and capacitance were studied for this sample. The dependence of both electrical resistivity ($\rho$) and capacitance ($C$) on temperature is shown in Figure 8 (a,b). From this figure it is noted that, ($\rho$) decreases with temperature with in temperature range (298 – 312 k), this is due to increase of electron mobility with temperature which leads to decrease of resistivity, while within temperature range (328 – 348 k) ($\rho$) increase with temperature due to increase the random motion electrons, which leads to increase the impedance for current passes. On the other hand the capacitance ($C$) increases with temperature as a result of increase of the electron mobility with temperature within temperature range (298–350 k), and decreases within temperature range (353–388 k).

The studied IV characterisation is shown in Figure 9, from this figure it is clear that the IV increases linearly with temperature, this due to increase of the electrons mobility’s with temperature which leads to increase the electric current. The influence of temperature on measured values of both of ($D$, $L_p$ and $S$) with different frequencies are shown in Figure 10 (a,b,c). From this figure it is noted that, ($D$) increases with both of temperature and frequency as a result of increasing the electron mobility’s with temperature, while ($L_p$) decreases with temperature at low frequencies, while at high frequencies, ($L_p$) increase with temperature due to increase of electron mobility. Finally ($S$) increased with temperature as a result of decreasing the electrical resistivity for this film with temperature.

![Figure 9](image-url) The influence of temperature on IV characterisation for Bi$_2$Se$_3$ thin film.

![Figure 10](image-url) (a) The relation between frequency and $D$, (b) frequency and $L_p$ at different temperature, (c) relation between $S$ and temperature for Bi$_2$Se$_3$ thin film.
Conclusion

The structure and surface topography for this sample were investigated using both of DEM and TEM photos, it is found that, the grains were distributed all over the film. Bi2Se3 thin film had a direct energy gap with value 1.98 eV. The determined parameters \((E_g)\) and \((E_0)\) had a values of 4.20 and 5.12 eV respectively, while the values of both of \(M_1, M_2\) and \(f\) had a values of 4.68 eV, 2.28 eV and 21.92 (eV)\(^2\) respectively. The \((N/m^2)\) had a value of 2.3 \(\times 10^{51}\). The determined dielectric parameters such as \((\varepsilon')\) and \((\varepsilon'')\) increase with \((h\nu)\) as a result of increasing the electron mobility’s, the same behaviour was obtained for \((\sigma_1, \sigma_2)\) with \((h\nu)\). The density of states \((DOS)\) for both of \((N_d)\) and \((N_s)\) determined with values of \(7.9 \times 10^{21}\) and \(2.2 \times 10^{22}\) respectively. The values of nonlinear results such as \((\chi^{(3)})\) and \((\beta)\) increase with \((h\nu)\) as a result of increase electron mobility and electron response to absorb light beam, also the same results were obtained for both of \((\chi, \varepsilon)\) with \((h\nu)\). The temperature affected strongly on both of electrical resistivity and capacitance for this film. The \(IV\) results increase with temperature as a result of increase electron mobility’s with temperature. The temperature affected strongly on all of \((D, L_p\) and \(S)\) which give an advanced to control and change these properties either by change frequency or change temperature.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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