Broadband photocurrent spectroscopy and temperature dependence of band gap of few-layer indium selenide (InSe)

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Abstract
Understanding broadband photoconductive behaviour in two-dimensional layered materials is important in order to utilize them for a variety of opto-electronic applications. Results of photocurrent spectroscopy measurements performed on few-layer indium selenide (InSe) flakes are presented here. Temperature (T)-dependent (40 K < T < 300 K) photocurrent spectroscopy was performed in order to estimate the band gap energies \( E_g(T) \) of InSe at various temperatures. Measurements indicate that room temperature \( E_g \) value for InSe flake is \( \sim 1.254 \) eV, which increased to a value of \( \sim 1.275 \) eV at low temperatures. The estimation of Debye temperatures by analysing the observed experimental variation of \( E_g \) as a function of T using several theoretical models is presented and discussed.

Keywords 2D semiconductors · Indium selenide · InSe · Photoconductivity · Photocurrent spectroscopy · Debye temperature

1 Introduction

Since the discovery of graphene [1], it has been predicted that there could be numerous layered materials that can be isolated to a single- or few-layer form. These include transition metal dichalcogenides (TMDs), metal oxides and single-element materials such as silicene and phosphorene [2]. Isolating thin layers of these materials from their bulk counterpart imparts them with exotic properties that could potentially lead to several applications [3–7]. Specifically, it is predicted that several thin two-dimensional (2D) layered semiconductors could possibly lead to multi-functional opto-electronic applications due to their exotic photo-electronic properties [8–10]. Initial studies, which favoured such predictions, heavily investigated molybdenum (Mo)- and tungsten (W)-based binary chalcogenides [11, 12], since it was found that 2D MoS\(_2\) can exhibit a photoresponsivity as high as 880 AW\(^{-1}\) [13] and multilayer WS\(_2\) can act as good photosensors [14].

Among many of the 2D materials that were initially investigated, several of them, which include MoS\(_2\) and WS\(_2\), are direct band gap materials in single layer form. The possibility of a single atomic layer to be viable for key opto-electronic processes that demand enhanced optical absorption [15], therefore, seems unlikely. As such, 2D layered materials which possess direct band gap in their few-layered form could perhaps provide ample optical absorption, leading to a variety of suitable applications, such as high sensitivity photo detectors, photo switch, and active materials for solar cells [15]. Several group III–VI layered compounds belong to this category in which direct band gap persists even if the material is few layers thick [3]. These classes of materials, which include InSe, show promising electronic and opto-electronic properties [3, 9]. These Se-based systems due to their high photoresponsivity and wide spectral response can be used for photovoltaics and photodetector applications. InSe was reported to have a small band gap of 1.3 eV, thus having a broadband spectral response [16, 17]. Investigators have also shown broad spectral response as well as high performance of flexible photodetectors using few layers of InSe [16, 18].
initial investigations seem very promising and indicate the prospect of InSe to become one of the choice materials for a wide variety of opto-electronics applications. Thus, understanding the spectral response of these materials is important from both fundamental and technical point of view. The photocurrent spectral response of few-layered InSe flake over a wide range of temperatures (40 K < T < 300 K) is presented here. Band gap values (E_g) for InSe flake as a function of temperature were estimated by method similar to Tauc plot. Analysis of the variation of E_g as a function T was performed using several theoretical models such as Bose-Einstein function [19], Double Bose-Einstein function [20] and Manoogian-Leclerc equation [21] in order to understand the effects of electron-phonon interaction as well as lattice dilation. This analysis will be presented and discussed in light of Debye temperature (Θ_D) of InSe flakes.

2 Materials and methods

2.1 Indium selenide crystal synthesis and exfoliation

Few-layer InSe devices were fabricated from thin flakes obtained through mechanical exfoliation of bulk crystals, grown using thermal treatment of a nonstoichiometric mix of indium (> 99.99%, Alfa Aesar Co.) and selenium (> 99.99%, Sigma-Aldrich Co.) with a molar ratio of 52:48 in a sealed quartz tube under millitorr vacuum [18]. At the beginning, the system was heated to 685 °C. This temperature was maintained for few hours to ensure a complete reaction between In and Se. Thereafter, the temperature was raised to 700 °C and was maintained at this temperature for another 3 h. The system was then cooled down to 500 °C at 10 °C per hour rate. Once the system was cooled down to 500 °C, it was allowed to cool down naturally to the room temperature to acquire high-quality InSe crystals. A detailed structural characterization of as-produced bulk InSe single crystals was previously reported in ref. [18, 22, 23].

2.2 Device fabrication

Detailed device fabrication method and electrical transport characterization is presented in previous reports [22, 23]. Briefly, the devices were fabricated on silicon/silicon dioxide (SiO2) wafers with a (SiO2) thickness of 1000 nm. A typical device fabrication routine involved evaporating metal contacts on top of a suitable flake residing on the Si/SiO2 wafer through shadow masking. Either nickel or gold transmission electron microscopy grid was used for the masking process. After securing TEM grid carefully on top of the flake, system was mounted inside the thermal evaporator chamber. The chamber was then pumped down to 10^-6 torr and was held at this pressure overnight. A thin layer of chromium (Cr) and gold (Au) electrodes was deposited through the mask by evaporating them using a tungsten (W) boat. Typically, about 40 nm of Cr layers and about 160-nm-thick Au layer were deposited. After metal deposition, the system was cooled down to room temperature before the devices could be taken out from the metal deposition chamber. The height profile of the device was measured using the contact mode atomic force microscopy (AFM) (Fig. 1a). Height of the device was measured to be ~ 34 nm, which corresponds to ~ 40 InSe layers (Fig. 1b). The metal contacted flakes were then placed on a ceramic chip holder and gold connecting wires were bonded to the metal contacts using a wire bonder. The chip was then mounted on a closed cycle helium cryostat for with an optical window for desired opto-electronic measurements.

Fig. 1 (a) Atomic force microscopy (AFM) image of InSe device measured. (b) AFM height profile taken along the red line from (a). (c) Schematic of experimental setup that was used for measuring photocurrent spectroscopy. (d) Typical photocurrent spectroscopy data (photocurrent as a function of wavelength)
2.3 Photocurrent spectroscopy measurements

Broadband photoelectronic conduction (PC) characterization was performed using in-house built photocurrent spectroscopy measurement setup. Xenon lamp was used as a light source to generate broad spectrum of wavelengths (300 nm ≤ λ ≤ 1000 nm) and was passed through a monochromator. These monochromatic light sources were guided on to the device using an optical wave-guide and focused on to the device using a convex lens with a focal length of 15 cm. Kiethley 2400 series source meters controlled with in-house developed LabVIEW module was utilized for measuring the photo response. Schematic diagram of the experimental setup and typical data obtained is shown in Fig. 1c and d respectively.

3 Results and discussions

Photocurrent spectroscopy, in the past, has been used widely in order to estimate the broadband optical absorption behavior of a variety of semiconductor materials [24, 25] including, InSe [17, 18], MoS2 [26], MoSe2 [27] etc. The generation and/or variation of current as a function of wavelength of light radiation impinging on the material is the core information that can be obtained using this technique. The data obtained using photocurrent spectroscopy setup is shown in Fig. 2a. From Fig. 2a, it can be seen that the photocurrent slowly increases for decreasing wavelengths close to the band gap (or E > 1.26 eV), and shows a broad peak around 2.5 eV. Similar photocurrent peaks are reported for other photoconductive materials in the past [28–31] and can be explained as follows. Initially, the photocurrent increases slowly and then starts to increase sharply from ∼ 2 eV until ∼ 2.66 eV. This could be generally attributed to increased photon absorption, which leads to an increase in the photo-generated carriers. As the wavelength decreases further the carriers generated increases (at higher energies). The carriers generated at higher energies (lower wavelengths) tends to recombine with surface states, and therefore do not contribute to the photocurrent and hence at lower wavelengths photocurrent decreases sharply (as seen in Fig. 2a for E > 2.5 eV).

In previous investigation [18] related to photocurrent spectroscopy measurement of similar InSe samples (measured within the wavelength of interval 400 nm < λ < 800 nm) it was noted that, in case of InSe, electronic transition from P_{x,y}-like orbital to the bottom of conduction band will occur between 2.25 eV (550 nm) and 3.1 eV (400 nm) and electronic transition from P_{z}-like orbital to the bottom of conduction band will occur between 1.55 eV (800 nm) and 2.25 eV (550 nm) [18]. Peaks occurring at similar energy values were observed for sample studied here as well, as seen from the deconvoluted peaks, summarized in Table 1.

where A is constant, λ_0 is peak position and σ is standard deviation. Values of λ_0 and σ for all five peaks from Fig. 2b are listed in Table 1.

Table 1 Parameter for peak fitting for photocurrent spectra. Here, λ_0 is peak position and σ is standard deviation of Gauss distribution (1)

| Peak # | λ_0 (nm) | σ  | E (λ_0) (eV) |
|--------|----------|----|--------------|
| Peak 1 | 466.56 ± 1.08 | 78.09 ± 1.06 | 2.658 |
| Peak 2 | 540.40 ± 1.07 | 16.77 ± 0.88 | 2.294 |
| Peak 3 | 580.79 ± 1.00 | 16.55 ± 0.75 | 2.134 |
| Peak 4 | 703.68 ± 3.74 | 125.89 ± 13.42 | 1.762 |
| Peak 5 | 861.10 ± 1.24 | 5.26 ± 1.38 | 1.439 |
| Peak 6 | 918.94 ± 2.14 | 65.53 ± 3.21 | 1.349 |

For λ between 800 and 1000 nm, a broad shoulder around 900 nm (∼ 1.4 eV; deconvoluted to two peaks) as seen in Fig. 2b. Generally, the higher wavelength peaks observed near the band edge are attributed to deep level impurities [32]; thus, such impurities in samples are responsible for some of the less intense peaks observed in the higher wavelength region. Photocurrent spectra at various temperatures is shown in Fig. 3a and band edge used to extract band gap is shown in Fig. 3b.
3.1 Band gap determination from photocurrent spectroscopy

In order to estimate the band gap from photocurrent spectra, a method similar to extracting band gap from UV-Vis absorption spectroscopy was utilized. Since the photocurrent generated in a semiconductor is proportional to the number of absorbed photons and the absorption coefficient ($\alpha$) of a direct band gap semiconductor is proportional to the square root of the difference of the photon energy ($E_{ph}$) and the band gap energy ($E_g$), or in other words $\alpha \sim (E_{ph} - E_g)^{0.5}$. Therefore, it can be assumed that $(I_{ph}) \sim (E_{ph} - E_g)^{0.5}$ as well and one can generate a plot similar to Tauc plot [33] for extracting the band gap values. In the past several investigators have utilized the aforementioned technique and used photocurrent spectroscopy for extraction and estimation of band gap [18, 24, 25, 27]. For this purpose, the quantity $[I_{ph} \times E]^2$ as a function of $E$ was plotted as shown in Fig. 4. Estimation of the band gap from this plot was performed by extrapolating the straight line portion of the data to x-axis intercept. In Fig. 4 such extrapolation for various temperatures is presented. The room temperature band gap value for the measured sample was found to be $\sim 1.254$ eV. Here, it should be noted that band tail at the edge of conduction band and valence band might result in photocurrent edge and it might interfere in band gap calculation. Analysis presented here is based on peak at 918.94 nm ($\sim 1.35$ eV, peak #6) which emerge due to band to band absorption and not from band tail absorption. The band gap value extracted here is similar to the values obtained for multi-layered InSe flakes from experimental investigations [16, 17, 34–37] employing a variety of other techniques. For example, Mudd et al. showed that $\gamma$-InSe shows direct band gap of $E_g = 1.2635$ eV by using layer-dependent photoluminescence (PL) and modelling it using a square quantum well potential of infinite height [17]. Gürbulak et al. reported direct band gap of $E_g = 1.280$ eV by optical absorption measurement [35]. Isik et al. showed band gap of $E_g = 1.22$ eV by absorption coefficient and derivative spectrophotometry analyses [36]. Zelewski et al. reported direct band gap of $E_g = 1.23$ eV by photoacoustic (PA) and modulated reflectance (MR) spectroscopy [37].

3.2 Temperature dependence of band gap

Temperature (T) dependence of $E_g$ derived from photocurrent spectroscopy of InSe (Fig. 4) is presented in Fig. 5. The information pertaining to the variation of $E_g$ in semiconductors as a function of temperature is of significance since, such information can be analyzed in order to have fundamental insights about core materials property. Generally, the
Fig. 5 Temperature dependence of the band gap energy of the InSe, fitted to (a) Bose-Einstein function (blue), (b) Double Bose-Einstein function (green) and (c) Manoogian-Leclerc equation (red)

variation of $E_g$ as a function of temperature is caused due to the combination of electron-phonon interaction and/or lattice dilation. In the past, several semi-empirical equations [19–21, 38] are developed in order fit the experimental data obtained. One of the most popular approaches is to fit the experimental data with Varshani’s equation [38]. However, it has been argued that in some cases where lattice dilation effects could be significant, a good fit to the experimental data using Varshni equation was not achievable [39]. Several other equations, for example Bose-Einstein (BE) function [19], Double Bose-Einstein (DBE) function [20] and Manoogian-Leclerc (ML) [21] equation, are also used widely to fit the variation of $E_g$ as a function of temperature. Below, each of these equations is described briefly and the results obtained by fitting them to the data are discussed in detail.

Bose-Einstein function [19] takes into consideration the interaction between electron and phonon and can be represented as shown in Eq. 2.

$$E_g(T) = E_g(0) - \frac{2a_B}{\exp\left(\frac{\Theta_E}{T}\right) - 1}$$

In this equation, $E_g(0)$ is a band gap at absolute zero (0 K), $a_B$ is a measure of strength of the electron-phonon interaction coupling within the crystal and $\Theta_E$ is the Einstein characteristic temperature. $\Theta_E$ is defined as the average temperature of the phonons which are interacting with the electrons. It is deduced that Debye phonon spectrum with the Debye temperature $\Theta_D$ is equivalent to an Einstein oscillator with a temperature $\Theta_E$, with $\Theta_D = 4/3 \times \Theta_E$ [19]. From fitting of Eq. 2 in Fig. 5a, a value of $\Theta_D$ is extracted to be 782.2 K. This value of $\Theta_D$ is significantly higher than theoretical value of $\Theta_D = 190$ K [40] as well as experimentally determined maximum possible $\Theta_D$ of 275 ±15 K for layered InSe [41]. One reason for such discrepancy could arise from the fact that Bose-Einstein function uses only one Einstein oscillator. In presence of one Einstein oscillator, band gap energy will either monotonically increase or saturate at constant value as temperature $T \to 0$ [20].

For the experimental data presented here, relatively small increase in band gap energy (∼25 meV) over the measured range of temperatures was observed. Similar small changes in band gap (∼30 meV) over a wide range of temperatures have been previously observed in samples of AgGaSe$_2$ [20]. Further, this particular study also observed a decreasing trend in the band gap values at lower temperatures. Similar behaviour at low temperatures ($T \leq 100$ K) was also observed (Fig. 5) for the InSe flake that is being reported here. This behaviour could be elucidated by considering a contribution from low energy (secondary) phonon with opposite weight, leading to decrease in band gap energy as $T \to 0$ [20]. Such contributions can be incorporated by using Double Bose-Einstein function [20] of the form shown in Eq. 3 was used for fitting.

$$E_g(T) = E_g(0) - \frac{2a_{B1}}{\exp\left(\frac{\Theta_{E1}}{T}\right) - 1} + \frac{2a_{B2}}{\exp\left(\frac{\Theta_{E2}}{T}\right) - 1}$$

where $E_g(0), a_{B1}, a_{B2}, \Theta_{E1}$ and $\Theta_{E2}$ have same meaning as previously described. The second Einstein oscillator (third term in Eq. 3) carries a opposite weight to that of the first oscillator. From fitting of Eq. 3 in Fig. 5b, a value of $\Theta_D$ is extracted to be 261.3 K, which is much closer to experimentally determined maximum possible Debye temperature of 275 ±15 K for layered InSe [41]. The frequencies of the two Bose-Einstein oscillators correspond to optical and acoustic phonons [20].

Past investigations have also shown that consideration of lattice dilation effects is crucial in order to explain the temperature variation of band gaps in a variety of semiconductors [21]. In order to determine if there is any lattice dilation effect along with electron-phonon
The data obtained was used to estimate Debye temperature ($\Theta_D$) values of InSe using established theoretical models. The $\Theta_D$ of $\sim$ 260 K obtained using Double Bose-Einstein equation is similar to the values obtained for this quantity for layered InSe. This strongly suggests that Double Bose-Einstein equation is perhaps the best model for explaining the variation of band gap of thin InSe flakes. This would mean (a) that band gap shifts observed in few-layer InSe samples presented in this study depend on electron-phonon interactions, which includes low energy secondary phonon, and (b) lattice dilation effects are perhaps negligible in samples studied. Most importantly, the variation of the band gap of InSe with temperature presented here is extremely valuable for strengthening the fundamental understanding needed for developing a variety of technological applications using optically active 2D materials.

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**Declarations**

**Conflict of interest** The authors declare that they have no conflict of interest.

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**Table 2** Values of parameters obtained by fitting of the experimental band gap energy data. Here $E_g(0)$ is band gap at 0 K, $\Theta_E$ is Einstein Temperature and $\Theta_D$ is Debye temperature. In case of double Bose-Einstein function, 1 and 2 correspond to Einstein oscillators associated with electron interacting with primary and secondary phonons respectively.

| Fitting               | $E_g(0)$ (eV) | $\Theta_E$ (K) | $\Theta_D$ (K) |
|-----------------------|--------------|----------------|----------------|
| Bose-Einstein         | 1.276 ± 0.002 | 588.1          | 782.2          |
| Double Bose-Einstein   | 1.274 ± 0.038 | 1196.5         | 1261.3         |
| Manoogian-Leclerc      | 1.272 ± 0.029 | 303.4          | 403.5          |

4 Conclusion

In summary, broadband photoconductive behavior was observed in 34-nm-thick flakes of layered InSe. The variation of band gap as a function of temperature was investigated by performing photocurrent spectroscopy measurements. Interactions that might influence the variation of $E_g$ with temperature in sample, fitting the data to Manoogian-Leclerc equation, which incorporates the lattice dilation effect term, was also considered. Equation 4 shows the form of Manoogian-Leclerc equation [21].

$$E_g(T) = E_g(0) - UT^4 - V\Theta_E \left[ \cosh \left( \frac{\Theta_E}{2T} \right) - 1 \right]$$

In Eq. 4, $E_g(0)$ is a band gap at absolute zero (0 K) and U, V, and s are temperature-independent constants. Second and third terms on right-hand side of equation correspond to lattice dilation term and electron-phonon interactions respectively. From fitting of Eq. 3 in Fig. 5c, a value of $\Theta_D$ is extracted to be 403.5 K. This value of $\Theta_D$ is substantially higher than experimental values of $\Theta_D$ previously reported for layered InSe [41]. Observing the $\Theta_D$ values obtained specifically from all the fits and comparing it to typical values of $\Theta_D$ reported for layered InSe [41], we believe that Double Bose-Einstein function is the closest model that can explain the band gap shift as a function of temperature observed in the sample. Various parameters obtained from all the fits are summarized in Table 2. From the information presented in Table 2, it is to be noted that although the estimated values of $E_g(0)$ for all the models used to fit the experimental data yielded reasonably consistent value of $E_g(0)$ $\sim$ 1.27 eV, this was not the case for values obtained for $\Theta_D$. One reason for this could be that $\Theta_D$ for a material is often dependent on multiple factors involving the thermodynamic and mechanical properties of material [42] and hence it becomes extremely difficult to have an accurate and consistent estimation of this value.
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