Layer-dependent ultrafast dynamics of α-In$_2$Se$_3$ nanoflakes

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Abstract
Photodetectors based on α-phase In$_2$Se$_3$ ultrathin films demonstrate unusually high photoresponsivity comparing to those based on other two-dimensional (2D) materials, such as MoS$_2$. To understand the underlying mechanism, we investigate the ultrafast dynamics of In$_2$Se$_3$ ultrathin films ranging from 11 nm to 40 nm on mica and Au substrates, respectively, analogous to the practical layout of a photodetector. Our results show that the carrier lifetime of α-phase In$_2$Se$_3$ on mica is nearly independent of thickness and comparable to that of MoS$_2$, and the efficient charge carrier separation occurs on Au substrate. Because all of the key parameters of In$_2$Se$_3$ nanoflakes that determine its photoresponsive behavior are of similar values to those of MoS$_2$, we suggest that the interface effect, i.e. photogating effect and contact resistance, should be responsible for the dramatic photoresponsivity reported for field-effect transistor-type optoelectronic devices.

Introduction
Indium selenide (In$_2$Se$_3$) is an intriguing III–VI compound layered semiconductor that attracts substantial attention due to its direct bandgap (~1.4 eV for bulk) and efficient absorption, which are appealing for optoelectronic applications [1–7]. Optoelectronic devices based on single-crystal α-In$_2$Se$_3$ thin layers demonstrated ultrahigh photoresponsivity [4, 5, 8–10], which is approximately 10$^2$–10$^3$ times higher than those photodetectors based on other 2D materials, such as MoS$_2$ [11], WS$_2$ [12], WSe$_2$ [13], SnSe [14] and SnS monolayer or few layers [15]. It is also suggested that the photoresponsivity of α-In$_2$Se$_3$-based photodetectors depends on sample thickness, bias voltage, incidence light power, and so on [10].

The photoresponsivity is defined as the ratio of the photocurrent to the incident illumination power. For a photodetector based on semiconductor materials with Ohmic contact, photocurrent is generally expressed as $eG\tau_p (\mu_n + \mu_p) AE$, where $G$ is the generation rate of carrier, which is related to absorption coefficient and pump fluence. $\tau_p$ is the carrier lifetime, $e$ is the electric charge, $\mu_n$ ($\mu_p$) represents the electron (hole) carrier mobility, $A$ is the cross-sectional area of the device and equal to channel length times width, and $E$ is the bias voltage. For α-In$_2$Se$_3$ thin film, its mobility (2.5 cm$^2$ V$^{-1}$ s$^{-1}$) [16] and absorption coefficient (4 × 10$^{-5}$ cm$^{-1}$ at 400 nm) [17] are comparable to those of bare monolayer MoS$_2$ (0.1–10 cm$^2$ V$^{-1}$ s$^{-1}$, 8 × 10$^{-5}$ cm$^{-1}$ at 400 nm) [18, 19]. The cross-sectional area $A$ is in the order of $\mu$m$^2$. Thus, the cross-sectional area $A$, the generation rate $G$ and the carrier mobility $\mu$ of these two materials have similar values. Despite of the fact that these parameters are not significantly different, α-In$_2$Se$_3$ exhibits an intriguingly high photoresponsivity [10]. Hence, it is natural to consider the effect of the carrier dynamics and the applied drain-source bias voltage based on the formula of photocurrent.

Carrier dynamics is an underlying mechanism that plays an important role in understanding the optoelectronic performance of devices [20–22]. The
carrier dynamics is related to the electron–hole separation, electron–electron scattering, electron–phonon and phonon–phonon interaction in the material. So far, the carrier dynamics of In$_2$Se$_3$, including its thickness dependence behavior, is still incomplete. Here, we employ ultrafast spectroscopy to investigate the carrier dynamics of single-crystal $\alpha$-In$_2$Se$_3$ nanoflakes of various thickness on mica and Au substrate. This configuration is relevant to real device architectures, whereby we aim to reveal the underlying mechanism of its extraordinary photoresponsive characteristics.

**Results and discussion**

In figure 1, we show that the transfer curves of our $\alpha$-In$_2$Se$_3$ phototransistor on a SiO$_2$ capped Si substrate. It is clearly seen that the device exhibits p-type conduction for few layers ($\leq$22 nm) and n-type-dominated ambipolar conduction for thicker films. The main reason for this behavior could be attributed to sample doping stemming from surface oxide or charge transfer at the interface (figure SI). Our results confirm the important role of film thickness on the device performance, which agrees with the previous reports[23, 24].

Since the ultrahigh photoresponsivity of $\alpha$-In$_2$Se$_3$ phototransistor we mentioned is on the mica substrate[10], we measure the differential reflectivity of the same batch of In$_2$Se$_3$ nanoflakes on mica using collinear ultrafast time-resolved pump-probe setup. Details of the setup are provided in the methods section. In this configuration, a mechanical chopper is employed to modulate the pump beam at ~2.1 kHz, and a lock-in amplifier collects the change in the reflection of the probe beam from the sample induced by the pump (i.e. $\Delta R$). Such a quantity is then normalized to provide a differential reflection, i.e. $\Delta R/R_0 = (R - R_0)/R_0$, where $R$ and $R_0$ are the reflection of the probe beam from the sample with and without the presence of the pump pulse, respectively. $\Delta R/R_0$ is confirmed to be proportional to the carrier density in the condition of unsaturated absorption regime[25]. Hence, by measuring the differential reflection as a function of the time delay, we can obtain the information of carrier dynamics. All the measurements are conducted at room temperature unless otherwise specified.

Figure 2(a) shows the typical fluence-dependent (6–30 $\mu$J cm$^{-2}$) carrier dynamics for $\alpha$-In$_2$Se$_3$ nanoflake with a thickness of 29 nm. The conversion between pump power and pump fluence/injected carrier density in the front surface are displayed in the table 1 of supplementary material (stacks.iop.org/TDM/6/035034/mmedia). The magnitude of $\Delta R/R_0$ represents the change of the complex refractive index in the presence of pump-induced carrier excitation. The possible sign of $\Delta R/R_0$ indicates that the absorption coefficient of In$_2$Se$_3$ decreased via the band-filling effect of the pump-induced carriers. The experimental result can be fit very well with single- or two-exponential function, depending on the pump fluence, i.e. injected carrier density (figure 2(a)). As displayed in figure 2(a), the signal decays with a time constant of 290 ± 10 picosecond (ps) at pump fluence of 6 $\mu$J cm$^{-2}$; all the other curves under the excitation of higher pump fluence are fit with a two-exponential function, as $\Delta R/R_0 = A_1 \exp (-t/\tau_1) + A_2 \exp (-t/\tau_2)$, where $A_1$ ($A_2$) and $\tau_1$ ($\tau_2$) are the amplitude and time constant of the fast (slow) decay component, respectively. The fitting parameters ($A_1$, $\tau_1$, $A_2$, $\tau_2$) as a function of pump fluence are summarized in figures 2(b) and (c), respectively. $\tau_1$ is on the order of several tens of ps, and $\tau_2$ is on the order of several hundred of ps. For example, under the highest pump fluence of 30 $\mu$J cm$^{-2}$, the signal decay can be fitted very well with two-time constants, 50 ± 2 ps and 340 ± 20 ps, respectively. $\tau_1$ decreases with increasing pump fluence, and it is much shorter than the typical radiative recombination time (longer than sub-nanosecond) of direct-gap semiconductors.
Thus, it could be attributed to the Auger type carrier–carrier scattering. This is also directly confirmed by the amplitude ($A_1$) dependence on the fluence as shown in figure 2(c) [26]. The Auger process is the dominant non-radiative process in semiconductors and happens in the several tens of ps, and it affects the efficiency of the electron–hole separation owing to the competing with carrier recombination process as mentioned in the below and the long-term heat accumulation in the samples. The occurrence of carrier–carrier scattering (i.e. Auger process) generally needs a carrier density larger than $10^{19}$ cm$^{-3}$. However, under the pump fluence of $6 \mu$J cm$^{-2}$, the carrier density is at the level of $10^{18}$ cm$^{-3}$, i.e. Auger scattering is too weak to be detect in the case of $6 \mu$J cm$^{-2}$. On the other hand, $\tau_2$ decreases with increasing fluence could be explained by the radiative carrier recombination process. The carrier recombination time ($\tau_2$) directly influences the electron–hole separation process. This is the first time to obtain the characteristic recombination decay time for $\alpha$-In$_2$Se$_3$ nanoflake. The inset of figure 2(c) shows the relative amplitude with pump fluence, which demonstrated that the ratio of Auger process in whole decay process increases with pump fluence due to the stronger carrier–carrier scattering at higher pump fluence. These two sub-processes compete and play an important role in the performance of the device based on the $\alpha$-In$_2$Se$_3$ nanoflake.

We further investigate the ultrafast carrier dynamics of In$_2$Se$_3$ on Au substrate: this configuration is to simulate the region in close contact to a metal electrode. The typical ultrafast relaxation dynamics of In$_2$Se$_3$ films with thickness of 21 nm on Au is plotted in figure 3(a). The negative sign of $\Delta R/R_0$ indicates that photo-bleaching occurs because of band-gap renormalization [27, 28]. We also find that $\tau_1$ (fast decay) and $\tau_2$ (slow decay) are similar to those of the samples on mica, as summarized in figure 3 (b). At the lowest pump fluence of $2 \mu$J cm$^{-2}$, the signal obeys a single exponential decay ($\tau_2$), and an additional relaxation component ($\tau_1$) appears with the pump fluence increases. $\tau_1$ is attributed to Auger recombination [29]. The fluence-dependent $A_1$ further confirms the process of Auger recombination, as displayed in figure 3(c). The slow decay ($\tau_2$) allows the efficient charger transfer at the interface of In$_2$Se$_3$/Au electrode.
before electron and hole recombined. The figure 3(c) shows the amplitude of the two sub-process (i.e. Auger and recombination process). Here we need to point out that due to the high reflection of Au substrate, the injected carrier density is higher than that of mica substrate under the same pump fluence. This is the reason that we can observe Auger process even with a low pump fluence of 8 μJ cm⁻².

As the thickness of two-dimensional material reduces to a few layers, their electronic structure may change dramatically, such as the case of direct bandgap for monolayer MoS₂. Therefore, the sample thickness could have a significant effect on the performance of the optoelectronic device. We further investigate the thickness-dependent ultrafast dynamics of In₂Se₃ nanoflakes with thickness varying from 40 nm to 11 nm. The differential reflectivity ΔR/R₀ of In₂Se₃ with various thicknesses is displayed in figure 86. It is found that the relaxation dynamics of all the samples has a similar behavior with that of the 29 nm thick sample under the same condition. The analysis of lifetime and amplitude of excited-state carrier are summarized as a function of thickness at the pump fluence of 30 μJ cm⁻², as shown in figure 4. Figure 4(a) shows that the carrier recombination time τ₂ is approximately 370 ± 60 ps and has no apparent dependence on thickness, and the Auger time constant τ₁ slightly increases with increasing thickness because the carrier density of thicker samples is smaller under the same pump fluence. The behavior of Auger time constant is consistent with the carrier density dependent Auger process and has been observed by previous report [22]. Since the Auger effect is directly related to the non-radiative process, it is necessary to choose a proper film thickness to minimize the effect of Auger process. In figure 4(b) we explicitly plot the thickness-dependence of the two amplitudes, which both exhibit a U-shaped dependence on the layer thickness. The amplitude ratio A₁/(A₁ + A₂) reflects the weighting of Auger in the whole decay process, which is shown in figure 4(c). The lower weighting of Auger effect is favorable for optoelectronic device applications. The carrier dynamics of In₂Se₃ may be very different when the thickness is reduced to a few layers owing to the thickness-dependent dielectric constant [30] and strong thickness-dependent shift of the optical band [31]. For the thickness range of our sample (11–40 nm), the carrier dynamics shows no notable difference.

In view of the fact that the carrier lifetime of In₂Se₃ on mica is approximately 370 ± 60 ps, which is comparable to that of MoS₂ (~200 ps) [32]. It is unlikely that the carrier lifetime is the dominant factor responsible for the ultrahigh photoresponsivity. The bias voltage on the channel, which is affected by the quality of contact, is another important parameter according to the photocurrent equation eGτ₂(µ₁ + µ₂)AE for the device with Ohmic-like contact. As we know, the In₂Se₃ photodetector yielded an ultrahigh external photoresponsivity of ~1650 AW⁻¹ at 5 V bias based on the Ohmic-like contact [10], and the monolayer MoS₂ photodetector with Ohmic-like contact only exhibited a photoresponsivity of 0.42 mAW⁻¹ at 1 V bias [33]. However, even for the Ohmic-like contact, the contact resistance is not equal, such as for monolayer MoS₂ transistors with the same metal contact, the contact resistances could have two orders of magnitude difference [34]. Therefore, it is reasonable to suggest that the unequal efficient bias voltage on the channels results from unequal contact resistance partly leads to the huge difference of their photoresponsivity.

The main mechanisms of the photoresponse of the phototransistor based on 2D layered materials are photocurrent (PC), photoconductive (PC), and photothermal (PTE) effect. Since the laser spot and the device channel have the similar size, it is not expected sharp thermal gradient that would lead to any appreciable contribution from thermal mechanisms to the total photocurrent, the PTE effect can be neglected under the operational conditions for both the In₂Se₃ [35] and MoS₂ [36–38] transistor. Hence, in general two mechanisms (PV and PC effect) give rise to the photocurrent, of which the PV effect is dominant mechanism in the ON state for both two materials [35, 38].

For layered-material-based phototransistors, due to the large surface to volume ratio of the materials, the PV effect often manifests itself as photogating effect, which is mediated by long-lived states from surface and interface traps [35, 38]. Under illumination, the photo-injected carriers charge the trap states leading to the formation of a Coulomb potential (i.e. the photo-induced gating effect), which is different from the gate voltage [39, 40] and has the same effect as applying gate voltage [39]. It has been reported that the photoresponsivity can be tuned by several orders of magnitude through controlling the gate voltage [41]. The gate voltage modulates the photoresponsivity strongly since the gate voltage influences the Fermi energy level of the 2D material, which leads to the dramatic change of the distribution of carrier obeys the Fermi–Dirac distribution. The change of the carrier distribution leads to huge difference of the photocurrent, i.e. giant different photoresponsivity. In MoS₂ transistor, PV effect arises from charge trapping by molecules, including few layers of surface-bound water underneath the MoS₂ sheet. The noticeable increase of photogating effect has been observed due to additional surface water and other adsorbents for MoS₂ device [35, 42]. While in the case of In₂Se₃, traps are also likely associated with the natural surface oxide [43], strengthening the photogating effect. It needs to point

5 It is need to point out that it is hard to identify thin flakes on mica substrate in the ultrafast dynamics’ measurement just through the weak optical contrast, and the pulsed laser leads to the sample burning and phase change for thin sample. Therefore, our discussion focused on the flakes with thicknesses between 11 nm and 40 nm.
out that the photoresponsivity and response time are competitive owing to these long-lived trap states. The larger responsivity implies a longer response time [44].

Conclusions

In summary, we investigate the ultrafast dynamics of α-In$_2$Se$_3$ nanoflakes on mica and Au substrate to understand the underlying mechanism of the optoelectronic performance of α-In$_2$Se$_3$. Our results indicate that the key parameters of the carrier dynamics of In$_2$Se$_3$ are comparable with MoS$_2$. The ultrahigh photoresponsivity of α-In$_2$Se$_3$ phototransistor should be closely reexamined, as we suggest that it might be associated with the interface effect, i.e. the photoinduced gating effect results from the long-lived trap states and the contact quality of metal electrodes.

Methods

Pump-probe setup

In the measurement, the femtosecond laser pulse from Ti:sapphire laser system (Mira with 6 W pump, Coherent Inc.) with 800 nm central wavelength,
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Conflict of interest

The authors declare no conflict of interest.

Supplementary data

Supporting information available; sample information and XPS of α-In2Se3 flakes; the calculation of pump fluence and carrier density; ultrafast carrier dynamics of In2Se3 flakes with different thickness on mica and Au substrate.

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