Thin-Film Transistors from Electrochemically Exfoliated In$_2$Se$_3$ Nanosheets

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1. Introduction

Two dimensional semiconductors have promoted the rapid development of electronics and optoelectronic devices due to their excellent charge transport properties and mechanical character [1,2]. The wafer-scale fabrication of well-ordered and uniform 2D semiconductor thin films are indispensable for large-area electronics and crucial to the practical application and development of 2D semiconductors [3–5]. High-quality 2D semiconductor thin films can be grown by chemical vapor deposition (CVD) [5–7]. However, the reaction conditions are stringent, and arduous transferring procedures are required to realize the transfer of thin films from the growth substrates to the targeted substrates. The transfer process is not only cumbersome and time-consuming, but it may also cause irreversible damage to the semiconductor device performance and reduce device yield.

Alternatively, uniform 2D semiconductor thin films can be economically prepared by a solution method from solution-processed 2D semiconductors colloidal inks [8,9]. Solution-processable 2D semiconductor electronics is an emerging research area, and substantial progress has been made. It has been reported that the electrochemical intercalation of quaternary ammonium ions is powerful in preparing stable 2D semiconductor inks such as graphite, black phosphorus, MoS$_2$, In$_2$Se$_3$, and NbSe$_2$ [10–14]. Their thin films have a wide range of applications in the fields of superconductors, field-effect transistors (FETs), photodetectors, and spin electronics. The spin-coated and LbL-assembled MoS$_2$ thin films
from electrochemically exfoliated MoS$_2$ nanosheets used as FETs channels show mobilities of $\approx 10$ cm$^2$.V$^{-1}$.s$^{-1}$ and on/off ratios of $>10^5$ [13,15]. Such device performance is superior to other solution-processed 2D semiconductor thin-film devices. The vacuum-filtrated In$_2$Se$_3$ thin films with thicknesses of 10 µm show ultrafast response, with rise and decay times of 41 and 39 ms, respectively, and efficient photoresponsivity (1 mA W$^{-1}$) [16]. However, conventional, solution-based, thin-film deposition approaches confront the problems of uncontrollable film thickness, uneven deposition, and the coffee ring effect.

In this work, we propose LbL assembly as an effective method of fabricating scalable 2D thin films from electrochemically exfoliated nanosheets. LbL assembly is based on the alternating assembly of two species with complementary interactions (such as electrostatic attraction, hydrophobic interactions, or hydrogen bonds) and can prepare thin films, patterns, and heterostructures on any substrate. Various low-dimensional electronic nanofilms, including gold nanoparticles, single-walled carbon nanotubes, boronitride, clay nanosheets, and MoS$_2$, have been successfully assembled by LbL assembly with precise thickness control [15,17–21]. Here, we assembled uniform In$_2$Se$_3$ thin films by electrostatic adsorption between poly(diallyldimethylammonium chloride) (PDDA) and electrochemically exfoliated In$_2$Se$_3$. The single In$_2$Se$_3$ flake and LbL-assembled In$_2$Se$_3$ thin films, serving as active channel materials in FETs, possessed excellent device performance. The mobility and on/off ratio of the LbL-assembled In$_2$Se$_3$ thin films were even better than the CVD-grown In$_2$Se$_3$ thin film, showing the robustness of solution-processed electronics.

2. Materials and Methods

2.1. Materials

Bulk In$_2$Se$_3$ was purchased from Six Carbon Technology (Shenzhen, China). Tetraheptylammonium bromide (THAB) was purchased from Aladdin (Shanghai, China). Polyvinyl pyrrolidone (PVP) was purchased from Energy Chemical (Shanghai, China). Poly(diallyldimethylammonium chloride) (PDDA) was purchased from Sigma-Aldrich (Shanghai, China). Polyurethane was purchased from Yantai Wanhua Polyurethane Co., Ltd. (Yantai, China). Cr and Au metals were purchased from Vnano Vacuum Technology Co., Ltd. (Beijing, China). Carbon rod was purchased from Tianjin Aida Hengsheng Technology Development Co., Ltd. (Tianjin, China). Other solutions, including Acetonitrile, N,N–Dimethylformamide (DMF), acetone, and ethanol and isopropyl alcohol were purchased from Tianjin Chemical Reagent Company (Tianjin, China). Ultrapure water (20 MΩ. cm$^{-1}$) was prepared by a Sartorius Arium pro UF system made by sartorius (Göttingen, Germany).

2.2. Synthesis of In$_2$Se$_3$ Nanosheets

In$_2$Se$_3$ nanosheets were synthesized by the electrochemical intercalation of quaternary ammonium ions. The electrochemical intercalation was performed in a 5 mg mL$^{-1}$ tetraheptylammonium bromide (THAB) acetonitrile solution, with the bulk In$_2$Se$_3$ and carbon rod serving as cathode and anode, respectively. The intercalation voltage was 8 V. After the intercalation, the THAB-intercalated In$_2$Se$_3$ was collected and sonicated in a 0.2 M polyvinyl pyrrolidone (PVP) solution (PVP: molecular weight of about 10,000) for 30 min to form a brown dispersion of In$_2$Se$_3$ nanosheets. The In$_2$Se$_3$ dispersion was subsequently centrifuged and washed with DMF several times to remove excessive PVP. The final In$_2$Se$_3$ dispersion was centrifuged at 1000 rpm for 5 min, and precipitates were discarded. The supernatant was concentrated in DMF for characterization and thin-film assembly.

2.3. Fabrication of In$_2$Se$_3$ Thin Films

The In$_2$Se$_3$ thin films were assembled by LbL assembly. Before LbL assembly, the SiO$_2$/Si substrates were pre-cleaned with acetone as well as ethanol and isopropyl alcohol. The substrates were treated with oxygen plasma at 100 W for 5 min to produce a superwetting surface. The substrates were firstly immersed in a positively charged PDDA solution (0.1 wt %) for 2 min to deposit single-layer PDDA chains and were then rinsed by
ultrapure water and gently dried with the use of an air gun. The substrates attached with PDDA chains were then immersed in negatively charged In$_2$Se$_3$ dispersion for 5 min, and In$_2$Se$_3$ nanosheets were assembled in order on the substrates by the electrostatic interaction between the PDDA and In$_2$Se$_3$. Finally, the substrates were rinsed by ultrapure water to remove the loosely attached In$_2$Se$_3$ nanosheets and were dried by air gun. The first cycle of the LbL assembly of the In$_2$Se$_3$ thin film was then completed. It is worth noting that the concentration of the In$_2$Se$_3$ dispersion was monitored by optical absorbance in order to assemble high-quality thin films. The single-layer, assembled In$_2$Se$_3$ thin films were dense when the characteristic absorbance at 450 nm was about 0.6 after the In$_2$Se$_3$ inks were diluted 500 times.

2.4. Fabrication of In$_2$Se$_3$ FETs

The channels of the FETs were fabricated by nanofiber masks. Aligned polyurethane nanofibers, whose diameters were maintained at ~500 nm, were printed on the substrates covered by In$_2$Se$_3$ single flakes or thin films. Subsequently, metal coatings (5 nm/45 nm Cr/Au) with a $200 \times 200 \mu$m metal mask were deposited by thermal evaporation to create source and drain electrodes. Finally, the SiO$_2$/Si substrates were immersed in the DMF solvent for 30 min and sonicated for 5 min to remove the polyurethane fiber, and the channel of the FET devices could be successfully prepared. In order to improve the contact between the electrode and the In$_2$Se$_3$, the devices were annealed at 200 °C in vacuum for 2 h before the test.

3. Results

3.1. Synthesis and Characterizations of In$_2$Se$_3$ Nanosheets

In$_2$Se$_3$ nanosheets were synthesized by the electrochemical intercalation of quaternary ammonium ions, as shown in Figure 1a. The electrochemical intercalation was performed in a 5 mg·mL$^{-1}$ tetraheptylammonium bromide (THAB) acetonitrile solution, with the bulk In$_2$Se$_3$ and the carbon rod serving as cathode and anode, respectively. Driven by external voltage, the positively charged THA$^+$ ions were inserted into the bulk In$_2$Se$_3$ and became fluffy and fell off from the cathode. The THAB-intercalated In$_2$Se$_3$ were collected and sonicated in a 0.2 M polyvinyl pyrrolidone (PVP) solution (PVP: molecular weight of about 10,000) for 30 min to form a brown dispersion of In$_2$Se$_3$ nanosheets (Figure 1b). There is a new XRD peak at a diffraction angle of about 6 in the THAB-intercalated In$_2$Se$_3$, indicating that the interlayer spacing of the THAB-intercalated In$_2$Se$_3$ increased from 9.7 Å to 17 Å and further proved the successful insertion of the THA$^+$ ions (Figure 1c). PVP acts as a surfactant to stabilize the In$_2$Se$_3$ nanosheet solution and prevent the agglomeration and sedimentation. The excessive PVP was removed by repeatedly washing with N,N-Dimethylformamide (DMF). The final In$_2$Se$_3$ dispersion was centrifuged at 1000 rpm for 3 min to sort the nanosheets. The sediments containing unexfoliated crystallites were discarded. The supernatant was concentrated in DMF for characterization and thin-film assembly. AFM showed that the exfoliated In$_2$Se$_3$ nanosheets had micron-level lateral dimensions (Figure 1d). A total of 90% of the In$_2$Se$_3$ nanosheets have thicknesses of 2.2 nm, further confirming the few-layer nature of the In$_2$Se$_3$ nanosheets and the uniformity of the thicknesses (Figure 1e). The lamellar structure of the In$_2$Se$_3$ nanosheet was verified by a transmission electron microscopy (TEM) image (Figure 2a), and the selected area electron diffraction (SAED) patterns indicated the single crystalline characteristics of the In$_2$Se$_3$ nanosheet (Figure 2b). The In 3d and Se 3d binding energy peaks of the electrochemically intercalated In$_2$Se$_3$ shifted to higher values as compared with the bulk In$_2$Se$_3$ due to the n-type doping induced by the insertion of the THA$^+$ ions (Figure 2c,d).
Figure 1. Synthesis and characterizations of In$_2$Se$_3$ nanosheets. (a) Schematic of the electrochemical exfoliation of the layered In$_2$Se$_3$ into nanosheets. (b) Photograph of In$_2$Se$_3$ dispersion in DMF. (c) XRD patterns of the THAB-intercalated bulk In$_2$Se$_3$ compared with commercial In$_2$Se$_3$. (d) Atomic force microscopy (AFM) image of In$_2$Se$_3$ nanosheets. (e) Thickness distribution of In$_2$Se$_3$ nanosheets.

Figure 2. Characterizations of In$_2$Se$_3$ nanosheets. (a) TEM image of In$_2$Se$_3$ nanosheets. (b) Selected-area electron diffraction image of an In$_2$Se$_3$ nanosheet. (c) Comparison of In 3d binding energies of electrochemically intercalated In$_2$Se$_3$ nanosheets and bulk In$_2$Se$_3$. (d) Comparison of Se 3d binding energies of electrochemically intercalated In$_2$Se$_3$ nanosheets and bulk In$_2$Se$_3$. 
3.2. LbL Assembled In$_2$Se$_3$ Thin Films

Well-ordered and uniform 2D semiconductor thin films are of vital importance to device performance. We chose LbL assembly to fabricate In$_2$Se$_3$ thin films by sequentially adsorbing the PDDA solution and the In$_2$Se$_3$ dispersion on SiO$_2$/Si substrates through electrostatic interactions (Figure 3a). The zeta potential of the In$_2$Se$_3$ dispersion was found to be $-18.1\,\text{mV}$ (Figure 3b). The intercalation of tetraheptylammonium ions led to the injection of electrons into the In$_2$Se$_3$ crystal structures and the slightly negatively charged In$_2$Se$_3$ nanosheets [22]. Before LbL assembly, the SiO$_2$/Si substrates were pre–cleaned with acetone as well as ethanol and isopropyl alcohol and then treated with oxygen plasma at 100 W for 5 min to produce a superwetting surface. The substrates were alternatively immersed in the PDDA solution (0.1 wt %) and the In$_2$Se$_3$ dispersion, with rinsing by ultrapure water and drying by air gun after each adsorption. The Raman characteristic peak originating from the A$_1$(LO + TO) of the In$_2$Se$_3$ thin film were consistent with the bulk In$_2$Se$_3$, while the A$_1$(LO) phonon mode of the In$_2$Se$_3$ thin film exhibited a small shift toward lower wavenumbers arising from the smaller vibration coherence length along the c-axis as a result of the weak van der Waals interaction (Figure 3c). The optical microscope image of the LbL-assembled In$_2$Se$_3$ thin film revealed that the nanosheets in a wide range of films were evenly stacked and assembled into homogeneous thin films (Figure 3d). From the local AFM and SEM image of the LbL-assembled In$_2$Se$_3$ thin film, we can deduce that the adjacent nanosheets were assembled on the substrate through broad-area, plane-to-plane Van der Waals contacts (Figures 3e and 4). The TEM images of the LbL-assembled In$_2$Se$_3$ thin film show that the adjacent nanosheets are stacked tightly together with mixed crystalline lattices on the boundaries and further demonstrate decent interfaces (Figure 5). The number of in-plane grain boundaries in the LbL-assembled 2D semiconductor thin films were greatly reduced and will significantly improve charge transport performance.

Figure 3. LbL assembly of In$_2$Se$_3$ thin films. (a) Schematic of the LbL assembly process. (b) Zeta potential of In$_2$Se$_3$ nanosheets dispersed in deionized water. (c) Raman spectra of In$_2$Se$_3$ thin films (purple) compared with bulk In$_2$Se$_3$ (red). (d) Optical microscope image of LbL-assembled In$_2$Se$_3$ thin films. (e) AFM image of LbL-assembled In$_2$Se$_3$ thin films.
Figure 4. SEM image of LbL-assembled In$_2$Se$_3$ thin films at different magnifications. (a) SEM image of LbL-assembled In$_2$Se$_3$ thin films with 2 µm scale bar. (b) SEM image of LbL-assembled In$_2$Se$_3$ thin films with 1 µm scale bar.

Figure 5. TEM image of LbL-assembled In$_2$Se$_3$ thin films. (a) Low-resolution TEM image of the LbL-assembled In$_2$Se$_3$ thin films. (b) High-resolution TEM image of the contact region between lateral nanosheets (The red dotted line is the edge of a nanosheet. The right shows the lattice of a single nanosheet, and the left shows the lattice of stacked nanosheets).

3.3. Performance of FETs from Electrochemically Exfoliated In$_2$Se$_3$ Nanosheets

To investigate the electric properties of solution-processed In$_2$Se$_3$, we further prepared In$_2$Se$_3$ single-flake and In$_2$Se$_3$ thin-film FETs on a 300 nm SiO$_2$/Si substrate. The channels of the FETs were fabricated by nanofiber masks (Figure 6) [23]. First, we used the diluted and concentrated In$_2$Se$_3$ dispersion to adsorb the sparse In$_2$Se$_3$ nanosheets and dense In$_2$Se$_3$ thin films on a pre-treated SiO$_2$/Si substrate by LbL assembly. Then, polyurethane nanofibers were printed and metal electrodes were deposited in order to fabricate FET devices. Figure 7a shows the scanning electron microscope (SEM) image of the In$_2$Se$_3$ single-flake FET with a length of 506 nm and an average width of 600 nm. The In$_2$Se$_3$ single-flake is perfectly flat on the channel to ensure good contact between the electrode and the In$_2$Se$_3$ nanosheet. The $I_{sd}$–$V_{sd}$ output characteristics of the In$_2$Se$_3$ single-flake FET showed a linear trend, indicating ohmic contacts between the In$_2$Se$_3$ single-flake and electrode (Figure 7b). The forward and reverse $I_{sd}$–$V_g$ transfer characteristics of the In$_2$Se$_3$ single-flake FET showed a typical n-type behavior with an on/off ratio of $1.5 \times 10^3$ at $V_{sd} = 1$ V (Figure 7c). The electron mobility of individual In$_2$Se$_3$ nanosheets can be calculated to be $12.8 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ from the linear–regime transfer characteristics using the following equation:

$$\mu = \frac{\text{d}I_{sd}}{\text{d}V_g} \times \frac{L}{WC_s V_{sd}}$$
where $L$ and $W$ are the channel length and width, and $C_s$ is the areal capacitance of 300 nm SiO$_2$/Si. The channel length and width of the In$_2$Se$_3$ thin film FET device are 549 nm and 200 µm, respectively (Figure 7d). The $I_{sd}-V_{sd}$ output characteristics of the In$_2$Se$_3$ thin film FET exhibited non-linear dependence on $V_{sd}$ due to the pinch-off effect of the FET channel (Figure 7e). The electron mobility of In$_2$Se$_3$ thin films reached 0.2 cm$^2$ V$^{-1}$ s$^{-1}$, with an on/off ratio of $7 \times 10^4$ at $V_{sd} = 1$ V (Figure 7f). The carrier mobility of the In$_2$Se$_3$ single flake is much higher than that of the In$_2$Se$_3$ thin film due to the sheet-to-sheet contact resistance, and the device performance may average out in the percolating thin films. The observed clockwise hysteresis in the transfer characteristics of the In$_2$Se$_3$ single-flake and thin-film FETs was attributed to charge trapping and detrapping at the interface between the In$_2$Se$_3$ and the SiO$_2$ (Figure 7c,f) [24]. To further understand the relative effects of PDDA, the spin-coated In$_2$Se$_3$ thin-film FETs were fabricated. The doping of the PDDA caused the positive shift of the threshold voltage and the lower maximum on-current as compared with the spin-coated In$_2$Se$_3$ thin-film FET (Figure 8a,b).

![SEM image of the channel fabricated by nanofiber masks.](image)

The performance of electrochemically exfoliated In$_2$Se$_3$ single-flake and thin-film FETs were comparable to those made from other methods in terms of mobilities and on/off ratios (Table 1) [25–29]. A comprehensive comparison is provided in Table 1. The electrochemically exfoliated In$_2$Se$_3$ single-flake FET performance was superior to some mechanically exfoliated In$_2$Se$_3$ flake FETs [27]. The electron mobilities and on/off ratios of the LbL-assembled In$_2$Se$_3$ thin film was close to those of the spin-coated In$_2$Se$_3$ thin films and CVD-grown In$_2$Se$_3$ thin films [12,28]. The outstanding device performances of both single flakes and thin films are attributed to the high-quality nanosheets with uniform sizes and thicknesses prepared by electrochemical intercalation.
Figure 7. Transistor performance of In$_2$Se$_3$. (a) SEM image of the In$_2$Se$_3$ single-flake FET with a single In$_2$Se$_3$ nanosheet as the FET channel. (b) $I_{sd}$–$V_{sd}$ output characteristics of the In$_2$Se$_3$ single-flake FET. (c) $I_{sd}$–$V_{ds}$ transfer characteristics of the In$_2$Se$_3$ single-flake FET. (d) SEM image of the In$_2$Se$_3$ thin-film FET channel. (e) $I_{sd}$–$V_{sd}$ output characteristics of the In$_2$Se$_3$ thin-film FET. (f) $I_{sd}$–$V_{ds}$ transfer characteristics of the In$_2$Se$_3$ thin-film FET.

Figure 8. (a) $I_{sd}$–$V_{sd}$ transfer characteristics of the LbL-assembled and spin-coated In$_2$Se$_3$ thin film FETs on a linear scale. (b) $I_{sd}$–$V_{sd}$ transfer characteristics of the LbL-assembled and spin-coated In$_2$Se$_3$ thin film FETs on a logarithmic scale.

Table 1. Comparison of In$_2$Se$_3$ FET performance with different preparation methods.

| Preparation Methods         | Mobility (cm$^2$ V$^{-1}$ s$^{-1}$) | On/Off Ratio | Reference |
|-----------------------------|-------------------------------------|--------------|-----------|
| Mechanically exfoliated In$_2$Se$_3$ flake | 30                                   | $10^5$       | [25]      |
| Mechanically exfoliated In$_2$Se$_3$ flake | 1.26                                 | 500          | [27]      |
| CVD-grown In$_2$Se$_3$ thin film | $2.51 \times 10^{-3}$                | 12           | [26]      |
| CVD-grown In$_2$Se$_3$ thin film | 1                                    | $10^3$       | [28]      |
Table 1. Cont.

| Preparation Methods                  | Mobility (cm² V⁻¹ s⁻¹) | On/Off Ratio | Reference |
|--------------------------------------|------------------------|--------------|-----------|
| PVD-grown p type In₂Se₃ flake        | 2.5                    | 10³          | [29]      |
| Spin-coated In₂Se₃ thin film         | 0.2                    | 10⁵          | [12]      |
| Electrochemically exfoliated In₂Se₃ flake | 12.8                  | 1.5 × 10³    | This work |
| LbL-assembled In₂Se₃ thin film        | 0.4                    | 7 × 10⁴      |           |

4. Conclusions

In conclusion, we prepared high-quality In₂Se₃ nanosheets through an electrochemical intercalation approach. Homogeneous In₂Se₃ thin films were assembled by the alternate adsorption of PDDA and a nanosheet solution, driven by electrostatic attraction. FETs from solution-processed In₂Se₃ single flakes and thin films showed satisfying performance and were comparable to those from CVD-grown In₂Se₃ thin films, mechanically exfoliated In₂Se₃ flakes, and spin-coated In₂Se₃ thin films. LbL-assembled 2D semiconductor thin films are promising candidates for emerging large-area, flexible, and wearable electronic applications.

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