High-Mobility InSe Transistors: The Role of Surface Oxides

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ABSTRACT: In search of high-performance field-effect transistors (FETs) made of atomic thin semiconductors, indium selenide (InSe) has held great promise because of its high intrinsic mobility and moderate electronic band gap (1.26 eV). Yet the performance of InSe FETs is decisively determined by the surface oxidation of InSe taking place spontaneously in ambient conditions, setting up a mobility ceiling and causing an uncontrollable current hysteresis. Encapsulation by hexagonal boron nitride (h-BN) has been currently used to cope with this deterioration. Here, we provide insights into the role of surface oxides played in device performance and introduce a dry-oxidation process that forms a dense capping layer on top, where InSe FETs exhibit a record-high two-probe mobility of 423 cm²/V·s at room temperature and 1006 cm²/V·s at liquid nitrogen temperature without the use of h-BN encapsulation or high-κ dielectric screening. Ultrahigh on/off current ratio of >10⁸ and current density of 365 μA/μm can be readily achieved without elaborate engineering of drain/source contacts or gating technique. Thickness-dependent device properties are also studied, with optimized performance shown in FETs comprising of 13 nm thick InSe. The high performance of InSe FETs with ultrathin dry oxide is attributed to the effective unpinning of the Fermi level at the metal contacts, resulting in a low Schottky barrier height of 40 meV in an optimized channel thickness.

KEYWORDS: indium selenide, native capping layer, oxidation, high-mobility transistors, hysteresis

Semiconducting layered materials show great promise as field-effect transistors (FETs) with device properties remarkably exceeding those of silicon when the vertical dimension is shrunken down to an atomic scale. Recent progress in such two-dimensional (2D) layered materials has facilitated the elucidation of their exotic electron transport properties and the developments of electronic applications. Along with the developments come advancements in our understanding of how metal contacts affect the 2D device performance. MoS₂ and WSe₂ are the two most representative materials, widely researched for the n- and p-type channels in the 2D FETs, respectively. A number of techniques have been employed to lower the contact barrier as well as to enhance the carrier mobility. For example, insertion of an oxide layer between the contact metal and 2D semiconductor can effectively unpin the Fermi level at the metal-2D semiconductor interface; deposition of a high-κ dielectric atop of the conduction channel can boost carrier mobility due to dielectric screening. Despite these efforts, a room-temperature mobility ceiling of ~300 cm²/V·s has been recently reported for FETs comprised of few-layer transition-metal dichalcogenides (TMDs). Search of other high-mobility 2D semiconductors has, therefore, been an imminent need to meet the development request of the semiconductor industry. Black phosphorus appears as an alternative in this regard, which exhibits a mobility as high as 1000 cm²/V·s in 10 nm-thick multilayers. Unfortunately, the chemical instability and the lack of suitable growth technology make it impractical for device applications.

Indium selenide, which possesses a low effective electron mass of 0.14m₀ (MoS₂: 0.45m₀; Si: 0.19m₀) and a moderate band gap of 1.26 eV, has recently attracted increasing attention. An intrinsic mobility (measured using four-probe method) of six-layer InSe encapsulated by hexagonal boron nitride of 560 cm²/V·s has been recently reported for FETs comprised of few-layer transition-metal dichalcogenides (TMDs). Search of other high-mobility 2D semiconductors has, therefore, been an imminent need to meet the development request of the semiconductor industry. Black phosphorus appears as an alternative in this regard, which exhibits a mobility as high as 1000 cm²/V·s in 10 nm-thick multilayers. Unfortunately, the chemical instability and the lack of suitable growth technology make it impractical for device applications.

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The thick and bumpy oxide layer is produced after the air oxidation. The height profile of a scan line marked by the red dashed line is displayed. (f) Schematic image of surface oxidation in air. A thick and bumpy oxide layer is produced after the air oxidation.

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**RESULTS AND DISCUSSION**

Figure 1 shows the characterizations of InSe flakes oxidized in air. The optical images of as-exfoliated and air-oxidized InSe flakes are shown in Figure 1a,b, respectively. To monitor the evolution of oxidation process, time-dependent Raman spectra were taken for a 6 nm- (Figure 1c) and a 3 nm-thick (Figure 1d) InSe flake. The Raman spectrum of the as-exfoliated 6 nm-thick flake peaks at 115, 178, 199, and 228 cm⁻¹, corresponding to the vibration mode of \( A_1'(\Gamma)^1 \), \( E'(\Gamma)^1 \), \( \Gamma^2 \), and \( \Gamma^3 \) (TO, \( A_2' \) (\( \Gamma^1 \)) – TO, \( A_2' \) (\( \Gamma^1 \)) – TO, \( A_2' \) (\( \Gamma^1 \)) – TO, respectively. Aging in air causes a slow but spontaneous oxidation. A noticeable broad band progressively developed between 150–250 cm⁻¹, and the four InSe characteristic modes vanish after a week of natural oxidation. The appearance of broad band between 150–250 cm⁻¹ indicates the formation of amorphous \( \text{In}_2\text{Se}_3 \) in the oxidation process, implying that the oxidation begins with the reaction: \[ 12\text{InSe} + 3\text{O}_2 \rightarrow 4\text{In}_2\text{Se}_3 + 2\text{In}_2\text{O}_3 \]
Owing to the unique crystalline structure of InSe, where half of the Se atoms are sitting on top, substitution of Se by O occurs in the early stage of the reaction at room temperature. The resulting oxide shares similar Raman characteristics with InSe, as compared to the spectra of as-exfoliated and few-hour oxidized InSe in Figure 1c. The oxidation proceeds with a transformation to In$_2$Se$_3$ whose amorphous nature features in the development of a Raman broad band between 150−250 cm$^{-1}$. Further reaction with oxygen eventually turns the intermediate state of amorphous In$_2$Se$_3$ into a stable In$_2$O$_3$:24

$$2\text{In}_2\text{Se}_3 + 3\text{O}_2 \rightarrow 2\text{In}_2\text{O}_3 + 6\text{Se}$$

At this state, the broad band vanishes, and the spectrum becomes featureless in the range of 50−400 cm$^{-1}$ after an exposure to the air for 4 weeks (Figure 1c). In addition, the InSe flakes covered with In$_2$O$_3$ exhibit an appreciable reduction of optical contrast because of the change of the total reflective index (Figure 1b).

Of particular importance here is the quality of the oxide formed in air, since it is the most relevant in the context of contact barrier in a transistor operation. Figure 1e shows the surface morphology of the air oxide inspected by AFM. Oxidation in air causes an uneven surface. Numbers of spotty bumps in nanometer scale can be seen, indicating a loose structure of the oxide formed in air, resembling the air oxide of black phosphorus.25 The root-mean-square roughness of the bumps is $\sim0.9$ nm. The loose and bumpy nature of the air oxide allows oxygen and water molecules progressively to penetrate deep into the inner layers, forming oxides as the source of degradation. Notably, the extent of surface oxidation strongly correlates with the thickness. As the number of layers increases, the band gap gradually decreases, and the CBM potential with respect to the redox potential of O$_2$/O$_2^−$ is lowered.26,27 When the CBM lies below the redox potential of O$_2$/O$_2^−$, photogenerated electron transfer from the conduction band to O$_2$ molecules will become prohibited, suppressing the production of reactive O$_2^−$. As a result, the oxidation is progressively decreased with the increase of thickness. Such bandgap-dependent generation of excitons, along with the CBM-dependent charge transfer, makes the thinner flakes produce more O$_2^−$ than the thicker ones and eventually accelerates the degradation of thinner flakes. Figure 1cd compares the evolution of Raman spectra upon oxidation for a 6 nm- and a 3 nm-thick flakes, respectively. In the same oxidation period, the characteristic InSe peaks can be explicitly identified for the 6 nm flake, whereas only a broad band is observed between 150 and 250 cm$^{-1}$ for the 3 nm-thick flake, indicative of a favored oxidation in thinner layers.

When the water molecules come into play in the oxidation process, the most significant impact is on the structural distortion and disintegration, as observed in the bumpy surface of the resulting oxides (Figure 1f). To avoid the inner layers from being further oxidized, forming a dense and robust cap

Figure 2. Oxidation of InSe in pure oxygen. (a) Optical image of InSe flakes oxidized in pure oxygen for 1 day. (b) Optical image of the same flakes after aging in air for 4 weeks. Time-dependent Raman spectra of the (c) 6 nm- and (d) 3 nm-thick InSe flakes marked in (a). (e) AFM image of InSe surface after oxidation in air. The height profile of a scan line marked by the red dashed line is displayed. (f) Schematic image of surface oxidation in air. A thin and smooth oxide layer is produced after the dry oxidation.
oxide is of particular importance. Moreover, a high-quality oxide can also serve as a tunnel barrier for Fermi level unpinning in the contact with metals. Here, we introduce a dry oxidation process. The as-exfoliated InSe flakes are oxidized in a sealed chamber filled with 30 Torr of ultrapure oxygen for 24 h, forming a dense oxide layer that can keep the inner InSe layers intact as exposed to the air. Figure 2c,d shows the evolution of Raman spectra for 6 nm- and 3 nm-thick InSe flakes capped with such dry oxide, respectively. For the 6 nm-thick flake, no discernible broad band between 150 and 250 cm\(^{-1}\) was found right after the oxidation in pure oxygen. The InSe covered with dry oxide shares the same Raman characteristic peaks with the pristine one, indicating the formation of nonstoichiometric thin oxide InSe\(_{1-x}\)O\(_x\). No amorphous In\(_2\)Se\(_3\) was formed until aging in air for more than a week, in sharp contrast to the cases of air oxidation. Although the development of amorphous In\(_2\)Se\(_3\) remains observable in the dry oxide after a long time of aging in air, the process has been greatly retarded. For the 3 nm-thick flake with dry oxide, the oxidation properties are similar to the air oxidation. Degradation of inner fresh layers takes place rapidly as the 3 nm-thick flake with dry oxide is exposed to the air, in accord with the picture of bandgap-dependent photoassisted oxidation reaction discussed above.

The most intuitive change of the InSe flakes oxidized in pure oxygen is perhaps the surface morphology, as shown in the AFM image in Figure 2e. The dry oxide exhibits an atomically smooth surface with a root-mean-square roughness of <0.16 nm, even after aging in air for a week. No bumps or bubbles are found, indicating that the oxide is chemically stable and does not react with moist water.

The chemical composition of the dry oxide is analyzed by XPS. Three types of samples are compared: as-exfoliated, dry-oxidized, and air-oxidized InSe. Figure 3a shows the O 1s spectra of these samples. A small but discernible peak at 531.6 eV can be found for the as-exfoliated InSe due to the unavoidable rapid oxidation in the process of sample loading.
the XPS chamber. For the air-oxidized InSe, the intensity of the O 1s peak is strong, instead. The oxidation can go deep into the inner layers, leading to an intense O 1s peak. For the dry-oxidized InSe, the O 1s peak is weak, as is the case of the as-exfoliated. Again, it implies the formation of a thin and nonstoichiometric indium selenide oxide, as conjectured from the Raman results. It should be noted that such a thin layer grown in pure oxygen can be readily removed using a short treatment of argon plasma (3 s) equipped within the XPS (Supporting Information Figure S1).

Figure 3b compares the Se 3d core levels of the three different cases, which are normalized with the intensity of the Si 3p. The doublet peaks at 54.7 and 55.5 eV correspond to 3d_{5/2} and 3d_{3/2} lines of the as-exfoliated InSe, respectively. Two remarkable features can be seen in the Se 3d spectra of the oxidized InSe: (1) the Se 3d peak intensity is significantly reduced for the air-oxidized InSe, indicative of a thick oxide layer on top. In contrast, the Se 3d peak intensity of the dry-oxidized InSe is only slightly reduced, in support of the formation of thin oxide InSe_{1-x}O_{x}. (2) Broadening of the full width at half-maximum (fwhm) and a remarkable downshift of the Se 3d binding energy by 1.2 eV are observed for the air-oxidized InSe, indicative of a complex oxide form involved.23,24,28

Figure 3c shows the In 3d core level spectra of the three different samples normalized with the intensity of the as-exfoliated InSe. The peaks at 445.3 and 452.8 eV correspond to 3d_{5/2} and 3d_{3/2} lines of the as-exfoliated InSe, respectively. The binding energy downshifts as InSe surface is covered with oxide. This downshift is attributed to electron transfer from the unoxidized inner layers to the surface oxide. This p-type doping of the InSe underneath the oxide is consistent with the transport measurements, as will be discussed later. The fwhm of the dry-oxidized InSe (0.93 eV) is found to be close to that of the as-exfoliated (0.92 eV). For the air-oxidized InSe, the fwhm of the In 3d peak increases to 1.15 eV. The peak can be deconvoluted into three subpeaks, corresponding, respectively, to InSe, In_{2}Se_{3}, and In_{2}O_{3} (Figure S2).28

To characterize the transport properties of InSe capped with air or dry oxide, back-gated FETs were made using different thickness of InSe flakes on SiO_{2}/Si, referred to as air or dry FETs hereafter. To ensure clean contacts and to avoid damages by the direct writing of electron beam in lithography (Figure S3), a shadow mask was used to define the drain and source contacts with Ti(20 nm)/Au(50 nm). It should be noted that the irradiation of electron beam on the contact regions causes a negative impact on the FET performance (Supporting Information). Prior to the deposition of metal contacts, the two types of samples were oxidized either in air or in pure oxygen (dry) for the same period of time (24 h). A vacuum annealing at 110 °C was applied before the electrical measurements were carried out in a probe station with a base pressure of 10^{-4} Torr. Figure 4a,b shows, respectively, the output characteristics at different gate voltages V_{gs} for the air and the dry FETs with the same channel thickness of 13 nm. The most striking difference between the air and dry FETs is the current density in the I−V_{ds} curves under the same biasing conditions. For the dry FET, the current normalized by the channel width can reach 300 μA/μm at V_{ds} = 5 V and V_{gs} = 80 V. For the air FET, in contrast, the I−V_{ds} curves saturate at small V_{ds}, limiting the current density at high bias. Remarkable changes have also been found in the conductivity vs gate voltage shown in Figure 4c,d. The two-terminal conductivity is defined as \( \sigma = I/V_{ds} \times L/W \), where L and W are the length and the width of the channel, respectively. For the air FET, in addition to the relatively lower \( \sigma \) at different \( V_{gs} \), the threshold voltage...
V_th is shifted to a large positive V_g as compared to that in the dry FET. The appreciable V_th shift in the air FET is attributed to strong p-doping caused by the cap oxides which involve InSe and In_{2}O_{3}. This strong electron transfer from the unoxidized InSe layers to the cap oxides is also supported by the remarkable downshift of the Se 3d and In 3d binding energies shown in Figure 3b,c. It is interesting to note that with increasing electron density in the InSe channels by an electrostatic gating, the dry FET displays a crossover from an insulating regime, where the conductivity increases with increasing temperature, to a metallic regime, where the conductivity increases with decreasing temperature. This metal–insulator transition occurs at a conductivity of \( \sim 1-3 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1} \) at room temperature, as shown in Figure 4f. The high on/off ratio in Figure 4f has been referred partly to water molecules embedded in the oxides and partly to the electric dipoles formed at the interface between the InSe and the oxides.

The air FET also features a remarkable current hysteresis as gate sweeps, in particular at high temperatures (inset to Figure 4c). This hysteresis in the \( I-V_g \) curves is also observed for the dry FET, but largely reduced (inset to Figure 4d). Though the underlying mechanism of the hysteretic behavior in air FETs has yet been systematically explored, plausible origins can be referred partly to water molecules embedded in the oxides and partly to the electric dipoles formed at the interface between the InSe and the oxides.

We extract the field-effect mobility by using the equation:

\[
\mu = \frac{L}{W} \frac{dG}{dV_g}
\]

where \( C_g \) stands for the back-gate capacitance, \( L \) and \( W \) are the length and width of the channel, and \( G = I/V_g \) is the conductance of the channel. Figure 4e shows the two-probe mobility as a function of temperature for the air and dry FETs with the same thickness of 13 nm. The transport results shown above demonstrate the advantages of native dry oxide as a superior contact interfacial layer for InSe FETs. The surface oxide plays two important roles in the device performance of InSe FETs: (1) it acts as a robust capping layer to protect the inner layer from being further oxidized; and (2) it also acts as a tunnel barrier which can effectively unpin the Fermi level. For a thin InSe flake, the oxide layer is thick. The thick tunnel barrier is not in favor of electron transport, and as a result, we see a lower current density and electron mobility for thin InSe. As the thickness of InSe increases, the oxide thickness decreases accordingly, giving rise to an appreciable increase of tunnel current and electron mobility. To better understand the Schottky barrier formed between the dry oxide and metal, we analyze the two-probe \( I-V \) curves as a function of temperature for the 13 nm-thick InSe dry FET. Electrical transport across a Schottky contact into the InSe channel can be described by the 2D thermionic emission equation:

\[
I = A_{2D} W T^{3/2} \exp \left( -\frac{q\varphi_s}{k_B T} \right) \left[ 1 - \exp \left( -\frac{-qV_th}{k_B T} \right) \right]
\]

where \( A_{2D} \) is the Richardson’s constant for 2D materials, \( W \) is the width of metal contact, \( q \) is the electronic charge, \( k_B \) is the Boltzmann constant, \( T \) is the temperature, and \( \varphi_s \) is the Schottky barrier height. The current as a function of gate bias shown in Figure 4b is used to extract the Schottky barrier height. We plot the current in logarithmic scale for various gate
voltage in Figure 6a. The slope of the Arrhenius plot, which is proportional to $\varphi_{th}$, decreases with increasing the gate voltage.

![Figure 6a](image)

**Figure 6.** Extraction of Schottky barrier height. (a) Linear fit of Arrhenius plot, ln($I$/$T^{3/2}$) vs 1000/T, from the $I$–$V_{gs}$ shown in Figure 4d. (b) Extracted effective barrier height $\varphi_{th}$ as a function of $V_{gs}$ for an InSe dry FET with channel thickness of 13 nm. Inset shows the band diagram at flat band conditions.

At $V_{gs} < 4$ V, the effective barrier height linearly responds to the gate voltage, indicating that the current flow is predominantly by the thermionic emission. Until $V_{gs}$ equals the flat-band voltage $V_{fb}$ (= 4 V), the tunneling current sets in. From Figure 6b, a Schottky barrier height of $\varphi_{th} = 40$ meV is extracted. This low barrier height is consistent with the highest mobility obtained for the 13 nm-thick InSe dry FETs and highlights the important role of dry oxide on the Fermi level unpinning.

**EXPERIMENTAL SECTION**

**Growth of InSe Single Crystals.** Single crystals of InSe with different area sizes and thicknesses were grown by chemical vapor transport method using ICl$_3$ as a transport agent. The powdered compounds of the crystals were prepared from the elements (In: 99.9999% and Se: 99.9999% in purity) by reaction at about 600 °C for 2 days in evacuated quartz ampules. To improve stoichiometry, selenium with extra 1 mol % was added to the stoichiometric mixture of the constituent elements. About 10 g of the synthesized elements together with an appropriate amount of transport agent (10 mg/cm$^3$ ICl$_3$) were introduced into a quartz ampule (22 mm OD, 17 mm ID, 20 cm in length), which was then cooled with liquid nitrogen, evacuated to 10$^{-5}$ Torr, and sealed. A growth temperature of 600 °C (heating zone) -> 500 °C (growth zone) with a gradient of ~5 °C/cm was set for crystal growth. The reaction was maintained for 288 h to produce large single crystals. At the end of the growth process, the synthetic InSe microplates essentially show a hexagonal shape with the areas from tens to hundreds $\mu$m$^2$. Some of them are even as large as several mm$^2$. The as-grown InSe plates reveal a black and shiny surface. The thickness of the crystal ranges from tens to hundreds $\mu$m. X-ray diffraction measurements indicated a two-layer hexagonal (2H) phase for the as-grown InSe. The weak van der Waals bonding between the layers means that the microplate can easily be separated to thin out (to ~ nm) from the c plane by using Scotch tape with mechanical exfoliation.

**Device Fabrication.** InSe flakes were exfoliated on silicon substrates with 290 nm thermal oxides in a glovebox filled with nitrogen. For the dry-oxidation devices, the flakes were directly sent into a sealed chamber filled with 30 Torr of ultrapure oxygen (99.999%) at 25 °C for 1 day. After 1 day oxidation, the flakes were taken out from the chamber. The source–drain electrodes were fabricated by thermally evaporated 20/50 nm Ti/Au electrodes through a homemade shadow mask. For air-oxidation devices, the same processes were used except for placing as-exfoliated flakes in air (25 °C and 50% humidity) for 1 day.

**Measurements and Characterizations.** XPS spectra were obtained using a VG Scientific ESCALAB 250 system. X-ray source was generated from the Al target (1486.8 eV) with a pass energy of 20 eV, and the takeoff angle for the collection of photoelectrons was 90° from the surface normal. The InSe flakes were exfoliated by a conventional copper tape and then exposed to air or 30 Torr of dry oxygen for 1 day for the air- or dry-oxidized samples, respectively. For Raman spectra, a high-resolution micro Raman spectrometer (LabRam 800, Horiba Jobin Yvon) equipped with a motorized sample stage was used to acquire the spectra. The excitation source is a 532 nm laser (2.33 eV) with a laser power below 0.1 mW to avoid laser-induced heating and damage. The laser spot size at focus was around 500 nm in diameter under a 100x objective lens. The morphology of the flakes was acquired by using the tapping mode of a commercial atomic force microscopy system (Innova, Vecco Inc.). The gate-dependent conductivities of the films were measured with a probe station under a vacuum condition of 10$^{-4}$ Torr.

**CONCLUSIONS**

In summary, native oxidation of InSe under ambient conditions forms a rough and thick oxide layer on top, which presents the root cause of device deterioration. A dry-oxidation process is introduced to form an nonstoichiometric In$_{x}$Se$_{1-x}$O$_{x}$ oxide which is smooth, dense, and self-limiting. InSe flakes with air and dry oxide were characterized by Raman spectroscopy and XPS. Both the air and dry oxidation processes are found to be thickness dependent. The dry oxide layer can effectively retard further oxidation of the inner unoxidized InSe layers as exposed to the air. InSe FETs with high-quality dry oxide show a remarkable two-probe mobility and on/off current ratio on a conventional SiO$_2$/Si substrate even without the use of h-BN encapsulation, high-$\kappa$ dielectric screening, or elaborate contact engineering. Both hysteresis and fast current saturation effects are substantially alleviated with this ultrathin capping layer, resulting in a current density as high as 365 $\mu$A/µm in a 5 $\mu$m-long device. Surface oxidation causes electron transfer from the InSe layers to the oxide. The resulting Debye length thus plays a crucial role in the performance of InSe FETs with different channel thickness. Controlling the surface oxidation of InSe pushes the development of high-performance InSe FETs with vertical miniaturization down to an atomic scale.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsnano.7b03531.
Details of removing ultrathin InSe$_x$,O$_y$ with argon plasma, oxidation products of InSe, dry-FET fabricated by conventional e-beam lithography, metal–insulator transition (MIT) of InSe, device with as-exfoliated InSe, stability of dry-FET, light-assisted oxidation process of InSe, and dry-FET with ultrathin InSe (4 nm) (PDF)

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Notes

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