Electrical characterization of two-dimensional materials and their heterostructures

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Abstract. Two-dimensional (2D) materials have gained enormous attention in recent years owing to their huge potential in future electronics and optics. On the one hand, conventional 2D materials like graphene, MoS₂, h-BN are being intensively studied, on the other hand, search for novel 2D materials is at a rapid pace. In this study, we have investigated electrical properties of 2D nanosheets of ultrathin Indium Selenide (InSe), a member of the III-VI chalcogenides’ family. The InSe layers were prepared via micromechanical cleavage of its bulk crystal and were integrated into a field-effect transistor (FET) device as the transport channel. On characterizing the InSe-based FETs, InSe showed n-type conductance with the mobility of 2.1x10⁻⁴ cm²V⁻¹s⁻¹.

1. Introduction
The electronic industry is teeming with new devices. An ever-increasing challenge to beat Moore’s law has led to the development of devices which are as small and compact as possible. The transistors today are already reaching nanometers (nm) of channel length [1]. However, as the devices are approaching sub-10 nm dimensions, it’s becoming increasingly difficult to follow Moore’s law. Silicon (Si), the dominating material in all electronic devices so far, is predicted to fail the sub-10 nm technology transistors because of severe short channel effects [2]. With this respect, two-dimensional (2D) materials have gained enormous attention in recent years owing to their intrinsic properties which compensate for the drawbacks of Si at such small dimensions [3-5]. Due to quantum confinement, they exhibit properties different from their bulk phases and thus, offer an opportunity to carry forward further miniaturization of electronic devices.

2D materials are theoretically known since decades [6-7], however, their first experimental proof came only in 2004, when Graphene was isolated from its bulk form Graphite [8]. Since then the field of 2D materials is booming, leading to more than 600 2D materials currently known [9-10]. The family of 2D materials consists of conductors, insulators and semiconductors, which are being intensively researched for their applications in the fields of electronics, optics and sensing [9-11].

Semiconducting 2D materials having a finite band gap, excellent transport properties and mechanical flexibility are of particular interest for the electronic industry. Transition metal-dichalcogenides (TMDCs) like MoS₂, WSe₂ are one of the most prominent classes of 2D materials, showing excellent semiconducting properties and thus, being researched world-wide [12-14]. Another class of semiconducting 2D materials rapidly gaining interest is the family of III-VI chalcogenides. These are layered materials with the formula–
MX (M=Ga, In; X=S, Se, Te), arranged in X-M-M-X tetra-atomic thick layers \[^5\].

Their thermal stability and absence of dangling bonds make them highly suitable for nanoelectronic and photonic applications \[^15\]. Though many theoretical investigations have been done for these materials, very limited experimental knowledge is available regarding their performance in nano-electronics \[^9\, 16-17\].

Within the III-VI chalcogenide family, we have focused our study primarily on Indium Selenide (InSe). One of the major reasons is its relatively high stability as compared to other members of the family, which gave us the chance to characterize it over a length of time. Moreover, InSe has lighter electron effective mass \(m^* = 0.143 m_o\) and shows higher mobility \(\mu\) of \(~ 10^3 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}\) as compared to MoS\(_2\) \(m^* = 0.45 m_o\), \(\mu = 50–200 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}\) \[^15\] and thus, can be used for fast, high performance electronics where MoS\(_2\) has been proved undesirable. In this study, we have investigated electrical properties of atomically thin layers of InSe in order to understand the underlying transport phenomena.

InSe consists of vertically stacked 2D sheets of Se-In-In-Se and has a honeycomb lattice as shown in figure 1. The atoms in the same layer are held by covalent bonds, whereas inter-layer bonding is via Van-der-Waals forces. In bulk form, InSe has a direct band gap of 1.3 eV at room temperature \[^5\].

![Figure 1](image)

2. Experimental Section

In order to understand the electrical properties of 2D InSe layers, we fabricated InSe-based field-effect transistors (FETs) in back gate configuration, where the transport channel was atomically thin layer of InSe.

2.1. Micromechanical cleavage of InSe ultrathin layers

Atomically thin layers of InSe were obtained via micromechanical cleavage of its bulk crystal. The InSe bulk crystal (2H-InSe) used for this study was bought commercially from 2D Semiconductors with a purity of 99.995\%. The bulk crystal was repeatedly peeled off with the help of a Scotch-tape to obtain few and single layers of InSe, which were deposited directly on highly p-doped Silicon (Si) substrates with 283 nm of SiO\(_2\) layer serving as back gate for the fabricated devices. The identification of single and few layers of InSe was done using the techniques of optical microscopy and atomic force microscopy (AFM) which have been discussed in later sections.

2.2. Device fabrication

On top of the deposited InSe layers, metal electrodes (source and drain) were defined using high-precision electron-beam lithography (EBL). The electrodes were fabricated by depositing a Ti (5 nm)/Au (100 nm) layer by electron-beam evaporation, followed by standard lift-off process in acetone.

2.3. Device characterization

The optical images of the InSe nanoflakes were taken with an optical microscope (OLYMPUS BX51). The thickness and roughness of the flakes were determined by tapping mode AFM in air using a Bruker Multimode 8 scanning probe microscope. The electrical measurements of the fabricated devices were carried out in back gate configuration in a semiautomatic Süss Microtec Probe System PA200 combined with a semiconductor characterization system (4200-SCS, Keithley Instruments). To maintain same environmental conditions during the electrical measurements of the devices, the temperature of the wafer chuck of the probe system was maintained at 25.0±0.5 \(^\circ\text{C}\) by using a
precision temperature control system. In addition to this, an air dehumidifier system was also used in order to maintain low humidity inside the probe system down to a dew point of -40 °C.

3. Results and Discussion
Figure 2(a) shows InSe layers deposited on Si/SiO₂ (283 nm) substrate via standard scotch tape technique as discussed above. In this method, different thicknesses ranging from single to bulk layers are deposited. However, one of the simplest and fastest techniques to distinguish between different layer thicknesses is optical imaging. Different layer thicknesses of InSe produce different color contrasts with respect to the interference of the underlying SiO₂ layer on exposing them to the visible light. In figure 2(a) the change in color of the flakes with changing thicknesses can be easily seen. Thinner layers give a low contrast, which improves with increasing thickness or number of layers. Using AFM we confirmed the thicknesses of the selected layers as shown in figure 2(b).

![Figure 2](image1.png)

**Figure 2.** (a) Optical image and (b) AFM of InSe flakes on Si/SiO₂ substrate; the number of layers—1 L, 2 L... are labelled as measured under AFM.

In our study, we have focused on electrical properties of ultrathin layers of InSe i.e. single or few layer structures. The optical image of one of our fabricated devices from InSe bilayer structure is shown in figure 3(a) and schematic of bottom-gate FET device on ultrathin InSe layer in figure 3(b).

![Figure 3](image2.png)

**Figure 3.** (a) InSe layer (dashed) 2.3 nm thick (as determined by AFM) contacted with Ti/Au metal electrodes, (b) schematic of bottom-gate FET, InSe serving as the transport channel.

The channel length i.e. distance between the contact pads for this device is approx. 2 μm. We measured the electrical characteristics of the FET by applying a drain-source voltage (V_{ds}) and a back gate voltage (V_{bg}) as shown in the schematic in figure 3(b). The measured output characteristic (Figure 4(a)) shows that Au metal did not form ohmic contacts to the InSe layer. The non-linearity in the negative bias and no current flow in the positive bias shows formation of a Schottky contact. The transfer characteristics (Figure 4(b)) shows semiconducting n-type behavior with mobility (μ) of 2.1×10⁴ cm²V⁻¹s⁻¹, estimated using the expression of field-effect mobility:

\[
\mu = \frac{dI_{ds}/dV_{bg}}{L/W} \times \frac{1}{C_{ox}V_{ds}}
\]

where I_{ds} is the drain-source current, V_{bg} is the back gate voltage, L and W are length and width of the channel respectively, C_{ox} is the capacitance per unit area of the SiO₂ layer and V_{ds} is the drain-source voltage. It should be noted that our values for the field-effect mobility are underestimated due to the...
formation of Schottky contacts and indulgence of very high contact resistance in our two-probe measurements.

Figure 4. I–V curves for InSe bilayer FET (a) Ids–Vds curve for Vbg sweep from 0 to 30 V, (b) Ids–Vbg curve for Vds sweep from -1 to 1V.

4. Conclusion
In this work, we have investigated ultrathin layers of InSe, a non-conventional 2D material which still lacks in-depth experimental studies. The layers showed good electrical properties w.r.t. transistor performance and an intrinsic n-type behavior. However, the mobility obtained was very low as compared to theoretical estimations, which was attributed to high contact resistance due to formation of Schottky contact between Au and InSe layer or to the degradation of InSe surface in the ambience.

Acknowledgement
We thank Mr. Holger Lange for assistance with the probe system and Mr. Vinayakrishna Joshi for AFM measurements. This work was kindly supported by the Initiative and Networking Fund of the Helmholtz Association of German Research Centers through the International Helmholtz Research School for Nanoelectronic Networks, IHRS NANONET (VH-KO-606).

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